Research Article

Carbon Nanotubes and Copper Oxide as Electro-Catalysts for Rechargeable Zinc-Air Batteries

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Abstract

CNTs and CuO will be used as electrocatalysts for redox reactions in rechargeable zinc-air batteries as the aim of this research. A carbon felt substrate was used to coat the CuO/CNTs composite, and the cathode properties were examined. Analyzed using XRD, FE-SEM, EDS, FT-IR, CV, and LSV. The results indicate that CuO/CNTs exhibits a monoclinic and hexagonal crystal structure, along with the presence of aromatic structures within the CNTs. The retention of PVDF was confirmed, and the composite demonstrated uniform dispersion on the carbon felt. The elemental composition was found to be C (54.52%), F (37.35% Cu (4.95%) and O (3.18%). At an applied voltage of 2 V, the current density increased from 0.66 to 6.92 mA cm^{-2}, while at 2.40 V, the current density decreased from –0.34 to –3.67 mA cm^{-2}. After 50 cycles, the current density declined by 5− 10%, indicating moderate electrochemical stability. The findings suggest that CuO/CNTs is a promising bifunctional electrocatalyst for both OER and ORR. Additionally, this research can be further developed into educational materials to enhance the understanding of zinc-air battery technology.

Keywords: Carbon Nanotubes; Copper Oxide; Electro-catalysts; Zinc-Air Batteries

1. Introduction

Zinc-air batteries (ZABs) have garnered significant attention as promising alternatives for next-generation renewable energy storage due to their high theoretical energy density, cost-effectiveness, operational safety, and environmental sustainability, with the potential to exceed traditional lithium-ion batteries in long-term applications [1 – 4]. The core structure of ZABs consists of a zinc anode paired with an air cathode, operating through the oxygen reduction reaction (ORR) when discharging and the oxygen evolution reaction (OER) while charging. The electrochemical reversibility of these oxygen processes plays a crucial role in determining the overall efficiency, cyclability, and durability of zinc-air batteries [5 – 8]. Despite their considerable promise, a critical technological challenge remains the development of economically feasible bifunctional electrocatalysts that can effectively enable both the oxygen reduction reaction (ORR) and the oxygen evolution reaction (OER) with high efficiency and long-lasting stability [9 – 11].

Noble metal-based catalysts, such as platinum (Pt), ruthenium dioxide (RuO_2), and iridium dioxide (IrO_2), are recognised for their outstanding catalytic performance. However, they face challenges due to high costs, limited availability, and insufficient long-term durability [12 – 13]. This has led to the exploration of more abundant and economical materials, like carbon-based nanostructures, which offer large surface areas, excellent electrical conductivity, and chemical stability [14 – 17]. Carbon nanotubes (CNTs) have become a noteworthy scaffold material due to their remarkable conductivity, outstanding mechanical properties, and high aspect ratio, which enhance efficient electron transport in electrocatalytic reactions.

Copper oxide (CuO), recognised as a p-type transition metal oxide, has undergone extensive examination of its bifunctional catalytic characteristics attributed to its diverse oxidation states (Cu^{+}/Cu^{2+}). These properties significantly improve both the oxygen reduction reaction (ORR) and the oxygen evolution reaction (OER), showcasing rapid and efficient kinetics [18]. The combination of CuO with CNTs is expected to yield synergistic advantages. CuO provides multiple active sites for oxygen-related redox processes, while CNTs deliver a highly conductive and stable framework that enhances charge transport and maintains structural integrity. Despite these theoretical benefits, there is a scarcity of studies that have thoroughly investigated the synergistic effects of CuO/CNT composites as bifunctional electrocatalysts for rechargeable zinc-air batteries (RZABs).

This study aims to address the existing research gap by synthesising CuO/CNT composite catalysts through solution blending and evaluating their structural, morphological, and electrochemical properties in air electrodes for rechargeable zinc-air battery systems. The investigation emphasises the catalytic performance and practical synthesis techniques suitable.

2. Materials and Methods

2.1 Materials

Multi-walled Carbon Nanotubes (MWCNTs, 99.99%) with carbon tube lengths of 3 − 12 mm, outer tube diameters of 12 nm, and tube wall thicknesses of 4 nm were procured from NANO GENERATION Co., Ltd. Cupric oxide (Cu, 97.00%) and zinc acetate dihydrate ($Zn(CH_3COO)_2$, 99.50%) were acquired from LobaChemie Pvt. Ltd. Additional reagents, including potassium hydroxide (KOH, 85%), ethyl alcohol (99.99%), and polyvinylidene fluoride (PVDF), were sourced from RCL Labscan. All chemicals were of analytical grade and used as received without further purification.

2.2 Preparation of carbon felt electrode

The carbon felt electrode was prepared using a straightforward thermal treatment process. Initially, ethyl alcohol (99%) was mixed with carbon felt at a 4×4 cm^2 carbon felt substrate. The saturated carbon felt was then placed in an oven at 90˚C for 2.10 h. After the thermal treatment, the carbon felt was recovered, yielding approximately 1.148 g of electrode material.


Soaked in ethyl alcohol for 10 min.

An oven at 90˚C for 2.10 h.

Carbon felt 1.148 g

Fig. 1 Schematic Illustration of the carbon felt electrode preparation process via thermal treatment**.**

2.3 Preparation of CuO/CNTs catalyst slurry

The resulting slurry had a total solid content of approximately 118 mg mL^{-1}, with a weight ratio of CuO : CNT : PVDF = 4:1:10.

1) PVDF of 1 g was dissolved in 10 mL of NMP by magnetic stirring at 60˚C for 100 min at 300 rpm.

2) CNTs of 0.10 g were added to the PVDF solution and stirred at 60˚C for 40 min at 300 rpm.

3) CuO of 0.40 g was then incorporated into the mixture and stirred at 60˚C for 15 min at 300 rpm.

4) The CuO/CNTs composite was successfully collected and dried, yielding around 12.70 mL of catalyst slurry. The mixture underwent ultrasonication at 40 kHz for a duration of 15 min to improve the dispersion of CNTs. The CuO/CNT catalyst slurry was utilised promptly following its preparation, without any storage period.

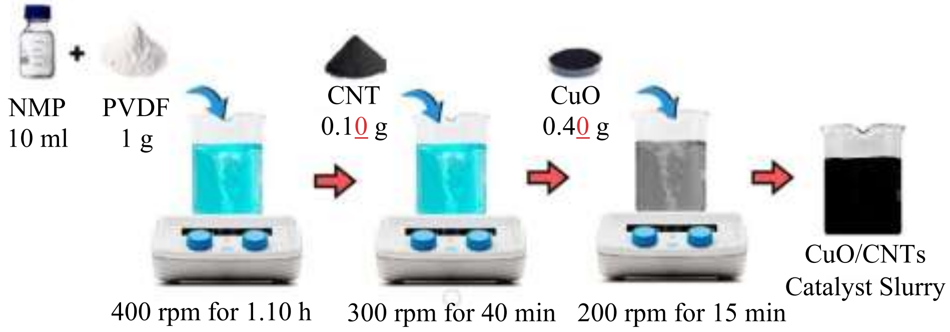

Fig. 2 Stepwise synthesis process of preparation of CuO/CNTs catalyst slurry.

2.4 Electrode assembly

The prepared CuO/CNTs catalyst slurry was applied to the carbon felt substrate using a doctor blade technique. The coating process was conducted with precise parameters temperature set at 0°C, blade speed at 3 mm s^{-1}, and coating length of 50 mm. This application procedure was repeated three times to ensure uniform catalyst distribution. Between each application, the coated electrode was dried in an oven at 90°C for 30 min. After the final coating and drying process, the mass of the finished electrode was recorded as 5.012 g.

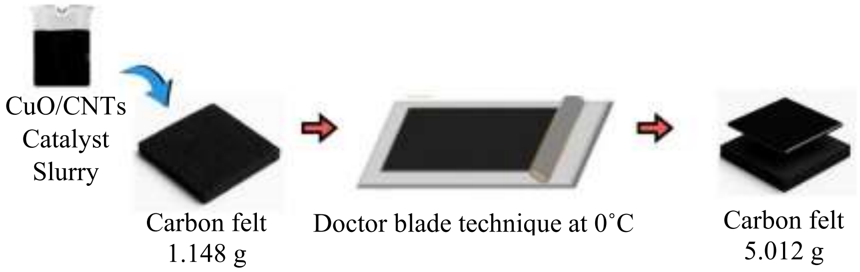

Fig. 3 Fabrication of CuO/CNTs-modified carbon felt electrode**.**

2.5 Preparation of electrolyte

The electrolyte solution was prepared by dissolving potassium hydroxide (KOH, 5.30 g, 4 Molar) and zinc acetate dihydrate ($Zn(CH_3COO)_2$, 0.44 g, 0.10 Molar) in 20 mL of DI water. The mixture was homogenized using a magnetic stirrer at 400 rpm for 10 min at ambient temperature, resulting in a clear electrolyte solution suitable for zinc-air battery testing.

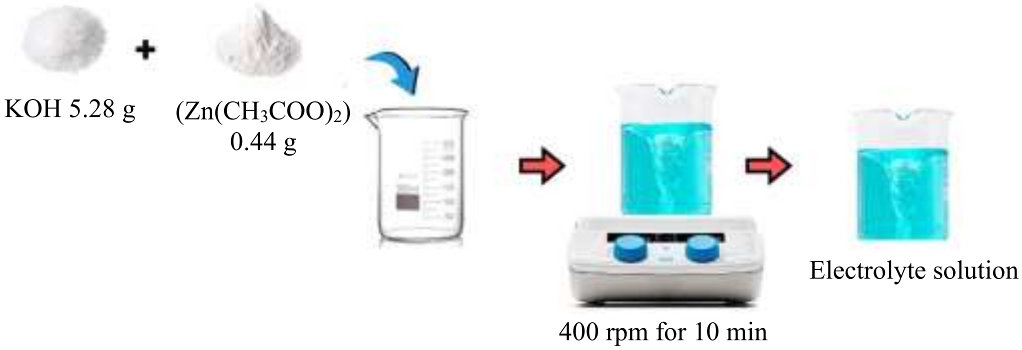

Fig. 4 Preparation of alkaline electrolyte solution.

2.6 Zinc-air battery module assembly

Utilized as the anode (negative electrode), where zinc undergoes oxidation, releasing electrons to generate electric current during the discharge process. Employed as the cathode (positive electrode) with the CuO/CNTs catalyst coating. This air electrode facilitates the oxygen reduction reaction by allowing atmospheric oxygen to enter and react at the catalyst sites. The prepared potassium hydroxide solution containing zinc acetate serves as the ionic conductor, facilitating ion transport between the zinc anode and carbon felt cathode during battery operation.

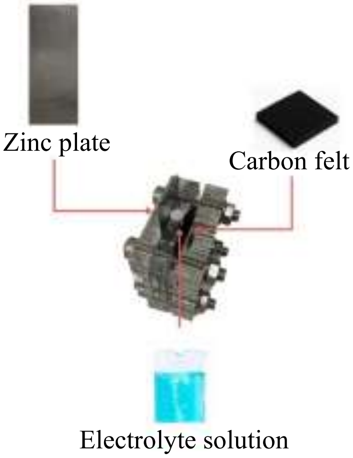

Fig. 5 Schematic assembly of zinc–air battery module.

3. Results and Discussions

The crystal structure and phase composition of the synthesized CuO/CNTs catalyst were analyzed using XRD. Fig. 6 presents the XRD patterns of the prepared materials. The diffraction peak at 2θ = 20.10° corresponds to the (110) plane, confirming the presence of PVDF as the binding material. The characteristic electrical conductivity enhancement from the incorporation of CNTs and CuO is evidenced by the diffraction peaks at 2θ = 32.50°, 35.50°, and 38.70° corresponding to the (002), (111), and (002) planes, respectively. The crystallite size of the CuO nanoparticles was recalculated using the Scherrer equation (1);

$$D = \frac{K\lambda}{\beta \cos\theta} \tag{1}$$

where D is the crystallite size, K is the scherrer constant (or shape factor), λ is the wavelength of the X-rays, β is the full-width at half-maximum (FWHM) of a diffraction peak, and θ is the Bragg angle. The based on the Lorentzian-fitted peak at 2θ = 38.95° with FWHM = 0.587°. The estimated average crystallite size was approximately 14.35 nm. Additional diffraction peaks observed at 2θ = 25.80° (002) and 78.30° (110) indicate the successful integration of CNTs in the composite structure.

The XRD analysis confirms the formation of a CuO/CNTs composite with well-defined crystalline structures of both components. The high crystallinity observed in the CuO phase suggests potential for enhanced catalytic activity, as crystalline structures typically provide more active sites for electrochemical reactions. Furthermore, the presence of clearly defined CNTs diffraction peaks indicates that the carbon nanotubes maintained their structural integrity during the composite preparation process, which is crucial for ensuring good electrical conductivity within the catalyst matrix.

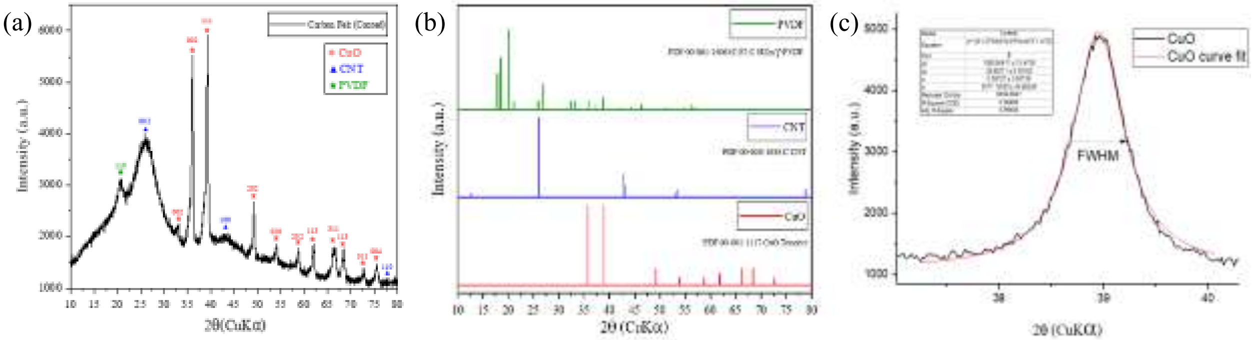

Fig. 6 XRD patterns of CuO/CNTs-modified carbon felt compared with standard diffraction profiles**.**

The surface morphology and elemental composition of the prepared materials were investigated using field emission scanning electron microscopy (FE-SEM) coupled with energy dispersive x-ray Spectroscopy (EDS). FE-SEM observations revealed distinct differences between pristine carbon felt and the CuO/CNTs-modified carbon felt.

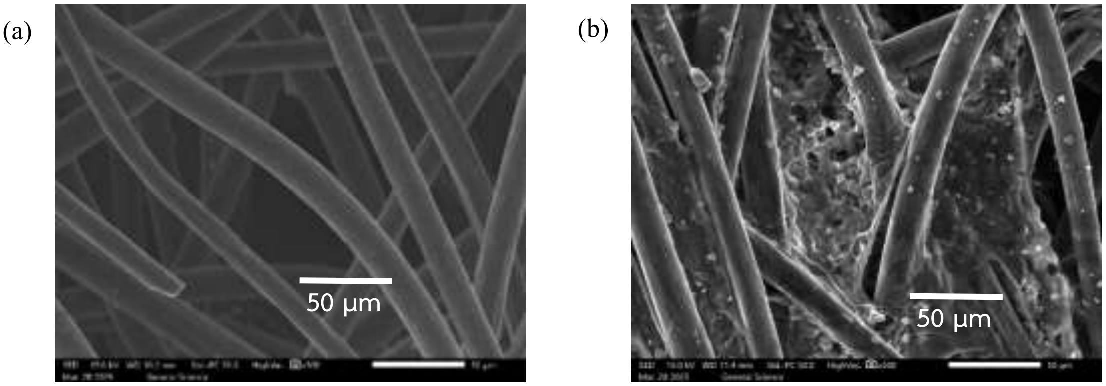

Fig. 7 FE-SEM top-view micrographs of (a) pristine carbon felt showing smooth and clean fiber surfaces, and (b) CuO/CNTs-modified carbon felt.

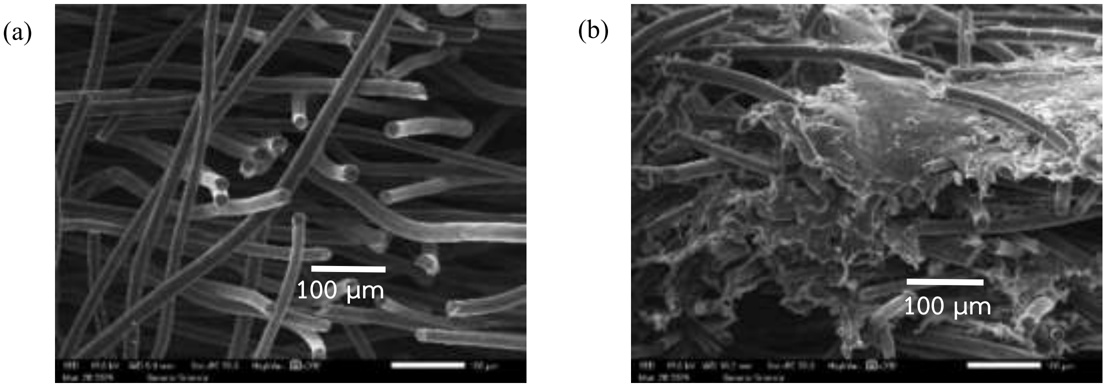

Fig. 8 FE-SEM cross section view of (a) pristine carbon felt showing smooth and clean fiber surfaces, and (b) CuO/CNTs-modified carbon felt.

Fig. 7 − 8 present FE-SEM images of the carbon felt substrates before and after catalyst coating at different magnifications. As shown in Fig. 7(a), the pristine carbon felt exhibits smooth fiber surfaces with uniform diameters and well-defined cylindrical structures. In contrast, evidenced by the presence of small particulate matter adhering to the carbon fibers.

Higher magnification images in Fig. 8 provide more detailed visualization of the morphological changes. Fig. 8(a) shows the pristine carbon felt fibers at magnifications of ×200, confirming their smooth and uniform structure. Comparatively, Fig. 8(b) illustrates the CuO/CNTs-modified carbon felt at corresponding magnifications, revealing a significant alteration in surface texture. The catalyst coating appears as irregular particulate deposits distributed across the carbon fiber surfaces, creating a roughened texture that potentially increases the electroactive surface area.

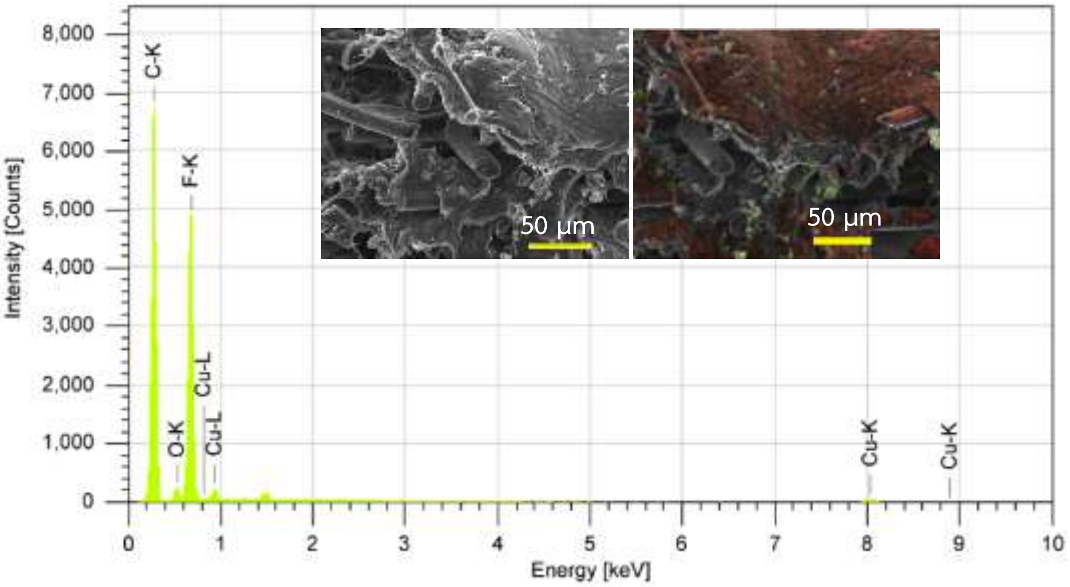

Fig. 9 Elemental composition of PVDF, CuO/CNTs.

The elemental composition of the CuO/CNTs composite was quantitatively determined using EDS analysis, as depicted in Fig. 9. The composite primarily comprises C (54.52%), O_2 (37.35%), Cu (4.95%), and F (3.18%). The high carbon content arises from both the carbon felt substrate and the embedded carbon nanotubes. The presence of copper and oxygen signals confirms the successful incorporation of CuO. Notably, the fluorine signal is attributed to the PVDF binder used during the catalyst preparation. Furthermore, the relatively high oxygen content may also result from the partial oxidation of the carbon substrate surface during thermal treatment, in addition to contributions from the PVDF. The relatively low copper content suggests that CuO is homogeneously and thinly distributed on the carbonaceous support, which is beneficial for maximizing catalytic utilization and ensuring uniform electrochemical activity across the surface.

Fourier transform infrared (FT-IR) spectroscopy was employed to identify the functional groups, organic compounds, and polymer-metal interactions present in the CuO/CNTs-modified carbon felt electrode. As illustrated in Fig. 10, the FT-IR spectrum reveals several characteristic absorption bands that confirm the successful incorporation of all components in the composite structure.

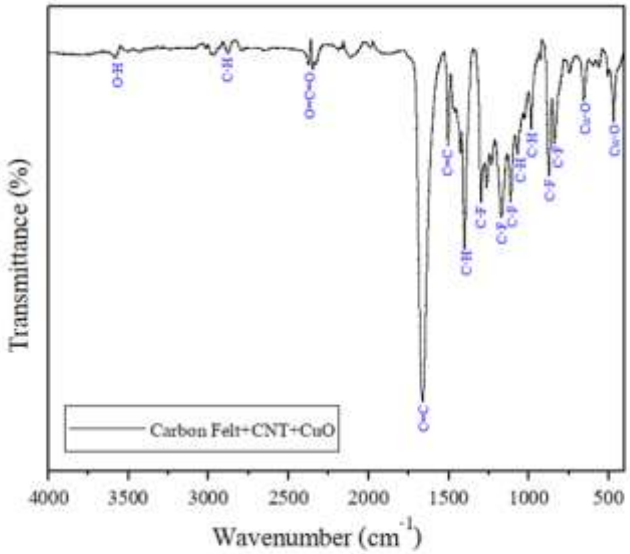

Fig. 10 Fourier transform infrared (FT-IR) spectroscopy of CuO/CNTs-modified carbon felt electrode.

The spectrum exhibits a prominent absorption band in the region of 1,600 − 1,650 cm^{-1}, attributed to C=C stretching vibrations, which indicates the graphitic structure inherent in both the carbon nanotubes and carbon felt substrate. The absorption band observed at 1,000 − 1,200 cm^{-1} corresponds to C-F stretching vibrations, confirming the presence of the PVDF binder used to integrate the catalyst components. Notably, the absorption band at 500 − 600 cm^{-1} is characteristic of Cu-O stretching vibrations, providing direct spectroscopic evidence for the successful deposition of CuO nanoparticles onto the carbon felt substrate.

The FT-IR analysis complements the XRD and FE-SEM, and EDS findings by providing molecular-level confirmation of the chemical bonds present in the composite. The identified functional groups play crucial roles in the electrochemical performance of the catalyst the graphitic C=C bonds facilitate electron transport, the C-F bonds from PVDF ensure mechanical stability of the catalyst layer, and the Cu-O bonds serve as active sites for the oxygen reduction and evolution reactions essential for zinc-air battery operation.

The electrochemical properties of the CuO/CNTs-modified carbon felt electrode were evaluated using cyclic voltammetry (CV) at a scan rate of 100 mV s^{-1}. Fig. 11 presents the CV curves recorded over 50 consecutive cycles, providing insights into the electrode's stability and electrochemical behavior.

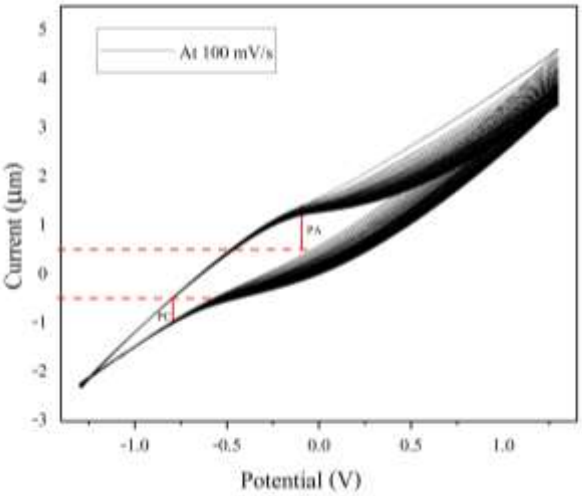

Fig. 11 Cyclic voltammetry (CV) of CuO/CNTs-modified carbon felt electrode.

The CV profiles demonstrate a consistent electrochemical response throughout the 50 cycles, with current variations limited to 5 − 10% between successive scans. This minimal variation indicates good electrochemical stability of the CuO/CNTs catalyst under repeated redox cycling, which is a crucial parameter for rechargeable zinc-air battery applications. The voltammogram exhibits characteristic features associated with the redox processes of copper species and oxygen reactions, with cathodic and anodic peaks corresponding to oxygen reduction and evolution reactions, respectively.

The quasi-rectangular shape of the CV curves suggests a capacitive behavior component in addition to the faradaic processes, which can be attributed to the high surface area provided by the carbon nanotubes and the porous structure of the carbon felt substrate. This combined faradaic-capacitive behavior is advantageous for energy storage applications as it enables both rapid charge transfer kinetics and substantial charge storage capacity.

The sustained electrochemical performance over multiple cycles can be attributed to several factors (1) the robust adhesion of CuO particles to the carbon felt substrate, as confirmed by FE-SEM analysis (2) the enhanced electrical conductivity provided by the CNTs network, facilitating efficient electron transport, has also been reported in previous studies on CuO/CNTs composites, where the incorporation of CNTs significantly improved the electrical conductivity of the hybrid catalyst [19]. And (3) the structural stability imparted by the PVDF binder, preventing catalyst Detachment during cycling. Collectively, these properties make the CuO/CNTs-modified carbon felt a promising bifunctional catalyst for rechargeable zinc-air battery applications. In this study, the electrode prepared via doctor blade maintained stable structure and electrochemical response after 50 CV cycles, confirming durability of the coating process. The consistent electrochemical responses confirm good mechanical stability of the doctor blade-coated electrodes. Although direct conductivity measurements of the CuO/CNTs composite were not performed in this study, previous studies have demonstrated that incorporation of CNTs into metal oxide composites significantly improves electrical conductivity due to the excellent charge transport properties of CNTs.

Linear Sweep Voltammetry (LSV) analysis was conducted to specifically evaluate the bifunctional catalytic activity of the CuO/CNTs composite for both oxygen reduction reaction (ORR) and oxygen evolution reaction (OER). As illustrated in Fig. 12, the composite demonstrates significant enhancement in both reactions compared to pristine carbon felt. For the ORR performance (Fig. 12(a)), the CuO/CNTs-modified electrode exhibits an onset potential at approximately –0.75 V (vs. Ag/AgCl), which is considerably more positive than that of pristine carbon felt (−1.00 V). More importantly, the CuO/CNTs catalyst significantly enhances the current density from −0.34 mA cm^{-2} (pristine carbon felt) to −3.67 mA cm^{-2} at a potential of 2.40 V, representing a nearly 11-fold increase in catalytic activity. In this work, impedance spectroscopy and discharge profiles were not measured due to laboratory constraints; however, the electrochemical performance of the CuO/CNTs electrode was evaluated using cyclic voltammetry (CV) and linear sweep voltammetry (LSV), which provided valuable insights into catalytic activity and stability. Future work will focus on including impedance and discharge characteristics for comprehensive benchmarking against literature reports**.**

Similarly, the OER performance (Fig. 12(b)) demonstrates remarkable improvement with the CuO/CNTs catalyst. At a potential of 2 V, the modified electrode generates a maximum current density of 6.92 mA cm^{-2}, which is approximately 10 times higher than the pristine carbon felt electrode (0.66 mA cm^{-2}). This substantial enhancement in OER activity is particularly significant for the charging process in rechargeable zinc-air batteries.

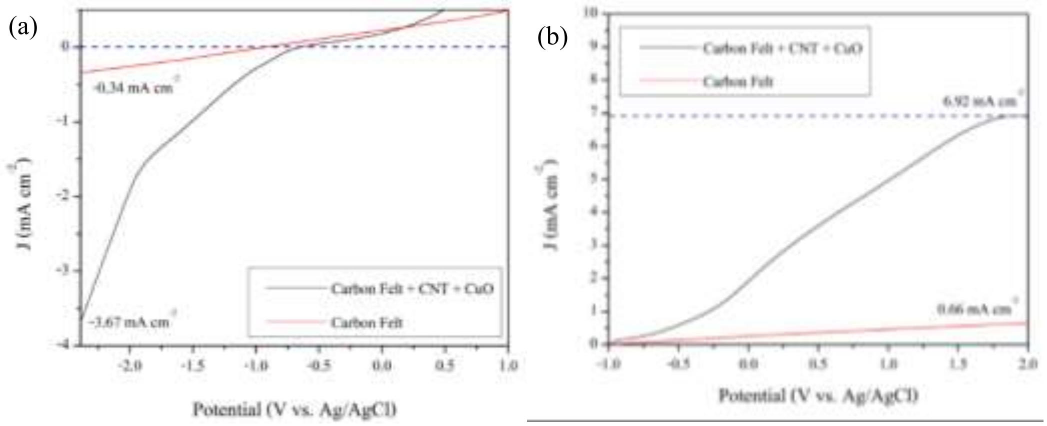

Fig. 12 Linear sweep voltammetry (LSV) curves of CuO/CNTs-modified and pristine carbon felt electrodes (a) LSV curve illustrating the oxygen reduction reaction (ORR) performance. (b) LSV curve for the oxygen evolution reaction (OER).

The impressive bifunctional catalytic performance can be attributed to several factors (1) the redox capabilities of copper species that facilitate electron transfer during oxygen reactions, (2) the high electrical conductivity provided by the carbon nanotubes that enhances charge transport, and (3) the increased electroactive surface area resulting from the nanostructured composite morphology as confirmed by SEM analysis. Additionally, the synergistic interaction between CuO and CNTs likely creates favorable local electronic environments that lower the activation barriers for both ORR and OER processes. Although electrochemical impedance spectroscopy (EIS) and discharge profile analysis (voltage–time and voltage–current density) are widely employed to further elucidate charge-transfer resistance and practical cell behavior, these measurements were not included in the present study due to instrumental limitations. Future work will incorporate detailed impedance spectra and galvanostatic discharge tests to provide a more comprehensive evaluation of the CuO/CNTs catalyst in zinc–air batteries.

4. Conclusion

The conceptual framework of this study highlights the rationale for integrating copper oxide (CuO) with carbon nanotubes (CNTs) as a bifunctional electrocatalyst in rechargeable zinc–air batteries. The combination is rooted in the complementary properties of both components. CuO provides abundant redox-active sites for oxygen reduction (ORR) and oxygen evolution (OER), while CNTs contribute a highly conductive network that enhances electron transport and structural stability. This synergy directly addresses two central challenges in zinc–air batteries—namely, the sluggish kinetics of oxygen electrocatalysis and the instability of electrode structures during repeated cycling.

Furthermore, the utilization of CuO represents a cost-effective alternative to noble metal catalysts such as RuO_2 and IrO_2, aligning with the broader objective of developing economically viable energy storage systems. When integrated with CNTs, the hybrid catalyst not only enhances the conductivity but also improves the durability of the electrode, mitigating degradation caused by repeated charge–discharge processes. This conceptual foundation is supported by recent reports in the literature, where the incorporation of carbon-based nanostructures with transition metal oxides has shown promising improvements in both activity and lifetime of electrocatalysts.

Finally, this work contributes to bridging the gap between fundamental electrocatalyst design and practical battery implementation. By demonstrating the effectiveness of CuO/CNTs within a zinc–air system, the study provides both mechanistic insight and applied significance, paving the way for scalable, low-cost, and durable energy storage solutions. This conceptual strengthening also positions the present work within the broader context of sustainable materials research and next-generation energy technologies.

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