

## Magnetic fluid-assisted CoFeNi/NF bifunctional electrocatalyst for high-efficiency water splitting in alkaline medium

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**ABSTRACT** Developing efficient bifunctional electrocatalysts is vital for scalable hydrogen production via overall water splitting. Here, a magnetic-fluid-assisted CoFeNi–MF/NF catalyst was synthesized through a two-step hydrothermal process by integrating Fe<sub>3</sub>O<sub>4</sub>-based magnetic fluid into vertically aligned CoFeNi nanosheets on nickel foam. Structural analyses confirmed the formation of an FCC CoFeNi alloy coupled with spinel Fe<sub>3</sub>O<sub>4</sub>, creating an interconnected architecture with abundant active sites. Magnetic-fluid incorporation enhanced charge transfer, oxygen content, and electron/spin polarization, enabling faster catalytic kinetics. In 1.0 M KOH, CoFeNi–MF/NF delivered low overpotentials of 169 mV for HER and 199 mV for OER at 10 mA·cm<sup>−2</sup>, along with Tafel slopes of 133.09 and 91.41 mV·dec<sup>−1</sup>. The catalyst also showed low charge-transfer resistance and enlarged electrochemically active surface area. Durable operation at 100 mA·cm<sup>−2</sup> for 24 h further confirmed its stability. This work presents a promising strategy for designing magnetic-responsive, non-noble-metal electrocatalysts for alkaline water splitting.

**KEYWORDS** bifunctional electrocatalyst, alkaline water splitting, HER, OER

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### 1. Introduction

In recent years, environmental issues, particularly the greenhouse effect resulting from the combustion of fossil fuels, have garnered significant attention due to the intensification of ecological preservation [1]. Consequently, the global energy paradigm is shifting from non-renewable sources to clean and renewable energy types, including solar, wind, hydrogen, geothermal, and others. Among these, hydrogen energy stands out as a sustainable and environmentally safe energy carrier [2]. Fuel cells are one of the most efficient ways to utilize hydrogen, converting chemical energy directly into electricity with high efficiency and zero pollutant emissions throughout the process [3]. However, the high production cost of hydrogen remains one of the main barriers to the practical application of fuel cells [4, 5]. Therefore, identifying cost-effective and large-scale hydrogen production methods is crucial. Water electrolysis is considered as one of the most efficient solutions for hydrogen production, given the abundance of water resources on Earth [6, 7]. Furthermore, electricity generated by solar panels and wind turbines can be used to drive water electrolysis, thereby reducing the consumption of fossil fuels and minimizing CO<sub>2</sub> emissions [8–10]. Despite these advantages, additional voltage is required for water electrolysis, which must exceed the thermodynamic standard potential (1.23 V) due to interfacial resistance, concentration polarization, and other factors [11, 12]. Hence, the development and fabrication of efficient electrocatalysts to minimize this extra voltage have attracted considerable interest. Currently, alkaline water electrolysis has been commercialized and widely adopted due to several advantages. First, alkaline conditions allow operation at lower overpotentials at high current densities, thereby reducing energy consumption and extending the service life of electrolysis equipment [13]. Second, in contrast to acidic environments, alkaline conditions offer broader catalyst material selection, including many non-noble transition metals and their oxides, which lowers the cost of catalyst usage [14]. Third, electrolyzers designed for alkaline water splitting are simpler and more economical because they do not require expensive components like proton exchange membranes [15]. Nevertheless, recent attention is turning to neutral media for water electrolysis due to corrosion issues associated with alkaline conditions. Neutral electrolytes are attractive for their ease of preparation, lower hazards, and environmental compatibility [16]. However, challenges remain in this area, including low mass transfer rates,

reduced current densities, high overpotentials, limited durability and stability, and an insufficient understanding of reaction mechanisms and kinetics in neutral media [17]. In summary, developing more efficient, durable, and cost-effective electrocatalysts is of critical importance for advancing hydrogen production through water electrolysis, particularly under alkaline and neutral conditions [18]. In this context, modern research has demonstrated that incorporating magnetic fluid (MF) materials into electrocatalysts can significantly enhance their electrochemical activity [19–21]. Especially when MF, rich in ferromagnetic particles, is combined with CoFeNi-based catalysts, remarkable improvements in HER and OER performance have been observed. This is attributed to enhanced electron transport and mass transfer facilitated by the magnetic field [22, 23]. Magnetic fluids containing nanoparticles such as  $\text{Fe}_3\text{O}_4$  can align under an external magnetic field, contributing to the formation of new active catalytic centers on the electrode surface [24, 25]. Additionally, the synergistic interaction of Co, Fe, and Ni elements with the magnetic field improves electron density and reaction kinetics at the electrode/electrolyte interface [26, 27]. This reduces overpotential, improves Tafel slopes, and lowers energy consumption.

Several studies have shown that liquid-magnetic CoFeNi composites exhibit high current density, low onset potentials, and long-term durability under alkaline and neutral conditions [28–30]. Optimal distribution of magnetic particles activates the catalytic surface, accelerates hydrogen and oxygen evolution, and facilitates the detachment of gas bubbles from the electrode [31]. Furthermore, liquid magnetic electrocatalysts can be synthesized via simple hydrothermal or chemical routes, reducing production costs and increasing practical feasibility [32, 33]. CoFeNi-based nanocomposites demonstrate high activity for HER and OER due to synergistic effects among d-orbitals [34]. For example, the NFC@CNSs-700 catalyst developed by Yaseen et al. incorporates  $\text{CoFe}_2\text{O}_4$  and  $\text{FeNi}_3$  particles within a carbon nanosheet structure, achieving excellent stability and a Tafel slope of  $56.36 \text{ mV} \cdot \text{dec}^{-1}$  for OER and a capacitance of  $19.44 \text{ mF cm}^{-2}$  for HER [35]. Similarly, Liu et al. designed CoFeNi-LDH and CoP/FeNi<sub>2</sub>P heterojunctions tested at ampere-level current densities in 1.0 M KOH and alkaline seawater, demonstrating over 100 hours of stable operation [36]. These findings highlight the influence of magnetic field-assisted catalysis on electron configuration, spin polarization, and local magnetic heating, all of which improve HER/OER efficiency. Structural optimization, as seen in NiCoFe-Se@LDH/NF and FeNiMo-OOH catalysts, further enhances performance, demonstrating strong electron interactions and excellent stability [37].

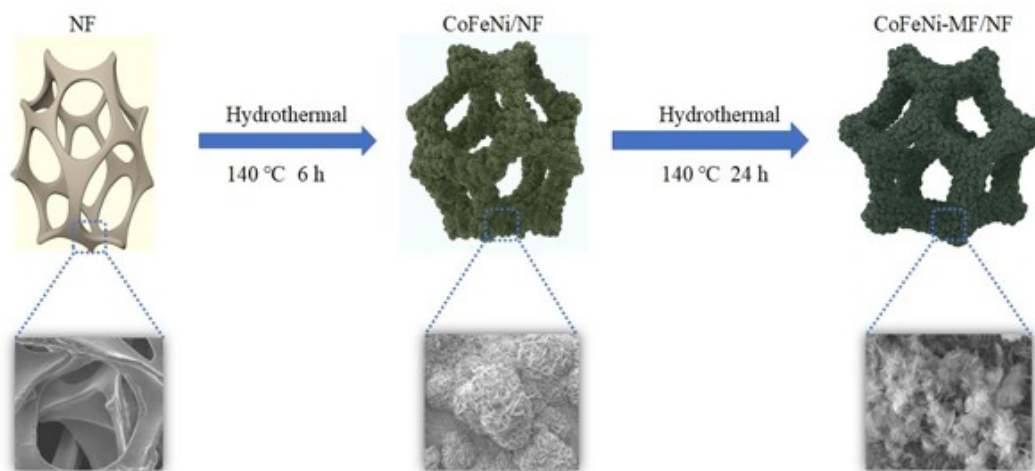


FIG. 1. Schematic illustration of the fabrication process for CoFeNi-MF/NF

In recent years, the development of highly active and durable bifunctional electrocatalysts for water splitting has attracted significant attention as a sustainable strategy for hydrogen production. Among transition metal-based systems, CoFeNi alloys have emerged as promising candidates due to their synergistic electronic interactions and cost-effectiveness. However, achieving efficient charge and mass transfer while maintaining long-term electrochemical stability remains a major challenge. To address this, a novel strategy was implemented in this study, where  $\text{Fe}_3\text{O}_4$ -based magnetic fluid (MF) was integrated into a CoFeNi/NF structure through a two-step hydrothermal synthesis. This hybrid approach facilitates enhanced electronic conductivity and accelerates charge transport, particularly under the influence of a magnetic field, leading to improved catalytic activity. The resulting CoFeNi-MF/NF nanocomposite demonstrates a dual-phase structure composed of FCC-type CoFeNi alloy and spinel-type  $\text{Fe}_3\text{O}_4$ . This architecture significantly increases the density of electrochemically active sites and exposes more surface area for reaction. As a result, the electrocatalyst exhibits outstanding performance toward both hydrogen evolution reaction (HER) and oxygen evolution reaction (OER). Specifically, the overpotentials at a current density of  $10 \text{ mA} \cdot \text{cm}^{-2}$  were found to be 169 mV for HER and 241 mV for OER, indicating highly efficient reaction kinetics. Corresponding Tafel slopes of  $86 \text{ mV} \cdot \text{dec}^{-1}$  (HER) and  $74 \text{ mV} \cdot \text{dec}^{-1}$  (OER) further confirm the rapid reaction pathways. Electrochemical double-layer capacitance ( $C_{dl}$ ) measurements suggest a notably enhanced

electrochemically active surface area (ECSA) in the CoFeNi–MF/NF system compared to the pristine CoFeNi/NF. In addition, the catalyst maintains excellent stability, showing negligible degradation after long-term chronopotentiometry and cyclic voltammetry tests. These results demonstrate the potential of CoFeNi–MF/NF as a robust and efficient bifunctional electrocatalyst for overall water splitting, offering a scalable pathway toward clean hydrogen energy technologies.

## 2. Materials and methods

### 2.1. Materials and chemicals

The following chemicals were used in this study: Iron(III) nitrate nonahydrate  $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ , Sigma-Aldrich, 99 %), Nickel(II) nitrate hexahydrate  $(\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O})$ , Alfa Aesar, 98 %), Cobalt(II) nitrate hexahydrate  $(\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O})$ , Sigma-Aldrich, 98 %), Ammonium fluoride  $(\text{NH}_4\text{F})$ , Merck, 99.5 %), Urea  $(\text{CO}(\text{NH}_2)_2)$ , Sigma-Aldrich, 99 %), Iron(III) chloride hexahydrate  $(\text{FeCl}_3 \cdot 6\text{H}_2\text{O})$ , Sigma-Aldrich, 98 %), Polyethylene glycol (PEG, Mw = 4000, Alfa Aesar, 99 %), Hydrazine hydrate  $(\text{N}_2\text{H}_4 \cdot \text{H}_2\text{O})$ , Sigma-Aldrich, 98 %), Ethanol  $(\text{C}_2\text{H}_5\text{OH})$ , Merck, 99.9 %), and deionized water (prepared in the lab). Nickel foam (NF,  $3 \times 3 \text{ cm}^2$ , 0.3 mm thick, 95 %) from Delving Metal Technology Co., Ltd. (China) was used as the substrate. All reagents were used as received without further purification.

### 2.2. Synthesis of CoFeNi/NF

CoFeNi/NF was synthesized using a hydrothermal method. Specifically, 0.45 mmol  $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ , 0.9 mmol  $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ , 0.45 mmol  $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ , 3 mmol  $\text{NH}_4\text{F}$ , and 6 mmol urea were dissolved in 80 mL deionized water and stirred for 30 minutes. The resulting solution and a  $3 \times 3 \text{ cm}^2$  piece of nickel foam were transferred to a 100 mL Teflon-lined autoclave. The hydrothermal reaction was conducted at 140 °C for 6 hours. After cooling to room temperature, the sample was washed multiple times with ethanol and distilled water, dried at 70 °C for 4 hours, and then annealed at 350 °C.

### 2.3. Synthesis of magnetic fluid (MF)

The magnetic fluid was prepared hydrothermally. 0.28 mmol  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$  and 0.3 mmol PEG were dissolved in 30 mL deionized water, followed by dropwise addition of 3 mL hydrazine hydrate  $(\text{N}_2\text{H}_4)$ . A reddish precipitate formed immediately. The mixture was transferred to a Teflon-lined autoclave and heated at 140 °C for 4 hours. After cooling, the product was magnetically separated and washed several times with ethanol and water.

### 2.4. Integration of MF with CoFeNi/NF

10 mg of MF was dispersed in a 1:1 mixture of ethanol and deionized water (50 mL total) using ultrasonication for 30 minutes. The dispersion and a pre-prepared CoFeNi/NF electrode were placed in a 100 mL Teflon-lined autoclave and subjected to a second hydrothermal treatment at 140 °C for 24 hours. The resulting sample was washed with distilled water and ethanol, dried at 70 °C for 4 hours, and annealed at 400 °C for 2 hours.

### 2.5. Physical characterization

X-ray diffraction (XRD, PANalytical X'Pert PRO) and scanning electron microscopy (SEM, JEOL JSM-IT510) were employed to analyze the structural and morphological properties. XRD measurements were performed using  $\text{Cu-K}\alpha$  radiation ( $\lambda = 1.5406 \text{ \AA}$ ). SEM images were used to evaluate the uniformity and morphology of the nanostructured surfaces.

### 2.6. Electrochemical measurements

Electrochemical tests were conducted using a Corrtest CS310X (Wuhan, China) workstation in a standard three-electrode setup. A Pt electrode served as the counter electrode, Hg/HgO as the reference electrode, and the synthesized electrocatalysts as the working electrodes. All tests were performed in 1.0 M KOH electrolyte.

Electrode potentials were converted to the reversible hydrogen electrode (RHE) scale using the formula:

$$E_{\text{RHE}} = E_{\text{Hg/HgO}} + 0.098V + 0.059 \times \text{pH}.$$

Overpotentials ( $\eta$ ) were calculated using:

$$\eta = E_{\text{RHE}} - 1.23V.$$

Linear sweep voltammetry (LSV) measurements were recorded at a scan rate of  $20 \text{ mV} \cdot \text{s}^{-1}$  with iR compensation. Cyclic voltammetry (CV) was carried out at scan rates ranging from  $1.0$  to  $5.0 \text{ mV} \cdot \text{s}^{-1}$ . Electrochemical impedance spectroscopy (EIS) was performed over the frequency range 0.01 to 100 Hz. In order to approximate the empirical data of EIS, Randleselectrical circuit was used.

### 3. Results and discussion

The surface morphology of the pristine Ni foam (NF) substrate, the CoFeNi/NF, and CoFeNi-MF/NF nanocomposites synthesized upon it was thoroughly examined using scanning electron microscopy (SEM) to understand the structural evolution and its implications for electrocatalytic performance. Fig. 2(a–c) illustrates the top-view SEM images of these samples. As shown in Fig. 2(a), the pristine NF substrate exhibits a smooth, open, and interconnected 3D porous structure composed of metallic ligaments. This architecture not only ensures low internal resistance and high electrical conductivity but also facilitates easy electrolyte access and gas diffusion during electrochemical reactions. Additionally, the NF substrate possesses excellent mechanical stability, making it a widely employed support material for direct catalyst deposition in water electrolysis systems. Upon hydrothermal synthesis of the CoFeNi nanocomposite onto the NF substrate, significant morphological changes occur. In Fig. 2(b), it is evident that vertically aligned, sharp-edged nanosheets are densely and uniformly grown on the surface of the Ni foam. These nanosheet structures create a hierarchical morphology characterized by a large specific surface area, which is known to be beneficial for maximizing the density of catalytically active sites. The ordered and homogeneous coverage of the nanosheets also indicates strong interaction and good adhesion between the CoFeNi precursor and the NF framework. Moreover, such nanosheet arrays can promote efficient ion and electron transport pathways due to the reduced diffusion length, ultimately contributing to improved electrocatalytic kinetics. The morphology undergoes further transformation after the introduction of magnetic fluid (MF) into the CoFeNi/NF system. As depicted in Fig. 2(c), the surface of the CoFeNi-MF/NF sample becomes more compact and fully covered with overlapping, plate-like structures. These layers are believed to form due to the effective interaction between the MF (rich in  $\text{Fe}_3\text{O}_4$  nanoparticles) and the preformed CoFeNi nanosheets, resulting in a secondary surface modification. The presence of MF induces additional texturing and increases the surface roughness, which can further enhance the number of exposed catalytic sites. Simultaneously, the coating with MF may improve the overall electrical conductivity by contributing conductive and magnetic nanostructures, potentially facilitating charge carrier transport within the catalyst film. This multilayered and interconnected architecture plays a crucial role in electrochemical performance. Firstly, the high-aspect-ratio nanosheets ensure a large electrochemical surface area (ECSA), which is directly correlated with the number of redox-active sites available for the hydrogen evolution reaction (HER) and oxygen evolution reaction (OER). Secondly, the hierarchical structure, including both nanosheet growth and MF coating, facilitates rapid mass and charge transport. The open macroporous network of the NF backbone promotes fast gas bubble detachment and minimizes mass transport limitations. Meanwhile, the nanosheet/MF composite layer ensures short diffusion distances for electrolyte ions and efficient electron transport to the active sites. Furthermore, the intimate contact between the catalyst layer and the NF scaffold reduces interfacial resistance and improves long-term operational stability. The morphological characteristics observed via SEM are consistent with the desired properties of an efficient bifunctional electrocatalyst. The synergistic combination of the NF substrate's porous conductivity, the CoFeNi nanosheets' high surface area, and the MF-derived modification layer results in a structurally robust and electrochemically favorable electrode. These features are expected to enhance not only the intrinsic catalytic activity of the material but also its stability under prolonged electrolysis conditions in an alkaline environment.

To evaluate the elemental composition and spatial distribution of the constituent elements in the synthesized electrocatalysts, energy-dispersive X-ray spectroscopy (EDS) analysis was conducted, including both elemental mapping and point spectrum measurements (Fig. 3(a–g)). This characterization is crucial for confirming the successful incorporation and uniform dispersion of the key metallic components (Ni, Fe, and Co), as well as oxygen, which collectively contribute to the electrochemical performance of the material. For the CoFeNi/NF sample, the EDS spectrum (Fig. 3(f)) reveals the presence of nickel (Ni), iron (Fe), cobalt (Co), and oxygen (O) with corresponding weight percentages of 63.2, 8.6, 8.6, and 19.6 %, respectively. The high content of Ni reflects its predominance as both a substrate and active phase component, while Fe and Co are successfully integrated in comparable proportions, forming a homogeneous trimetallic oxide system. The oxygen content confirms the partial oxidation of the metallic components, resulting in the formation of mixed metal oxides (MMOs), which are well-known for their enhanced electrocatalytic activity in both the hydrogen evolution reaction (HER) and oxygen evolution reaction (OER) processes. In contrast, the CoFeNi-MF/NF sample exhibits a notable shift in elemental composition, with Ni reduced to 50.6 %, Fe increased to 11.9 %, Co slightly decreased to 6.3 %, and O significantly increased to 31.2 % (Fig. 2(g)). This variation is attributed to the introduction of the magnetic fluid (MF), which primarily contains  $\text{Fe}_3\text{O}_4$  nanoparticles. The elevated Fe content indicates the successful incorporation of MF-derived iron oxide, while the sharp increase in oxygen content is indicative not only of  $\text{Fe}_3\text{O}_4$  inclusion but also of additional surface oxidation processes occurring during or after synthesis. Such oxidation may result in the formation of amorphous or hydrated oxide phases, which can further enhance electrocatalytic activity by increasing surface defect density and active site exposure. The elemental mapping images (Fig. 3(a–d)) for the CoFeNi-MF/NF sample corroborate the uniform distribution of Ni, Fe, Co, and O across the catalyst surface. Homogeneous dispersion of these elements is critical to ensure consistent catalytic behavior across the electrode, preventing localized performance degradation or instability. This uniformity also suggests that the synthesis route employed facilitates even nucleation and growth of the active components across the porous substrate, a key factor in achieving high and reproducible electrochemical efficiency.

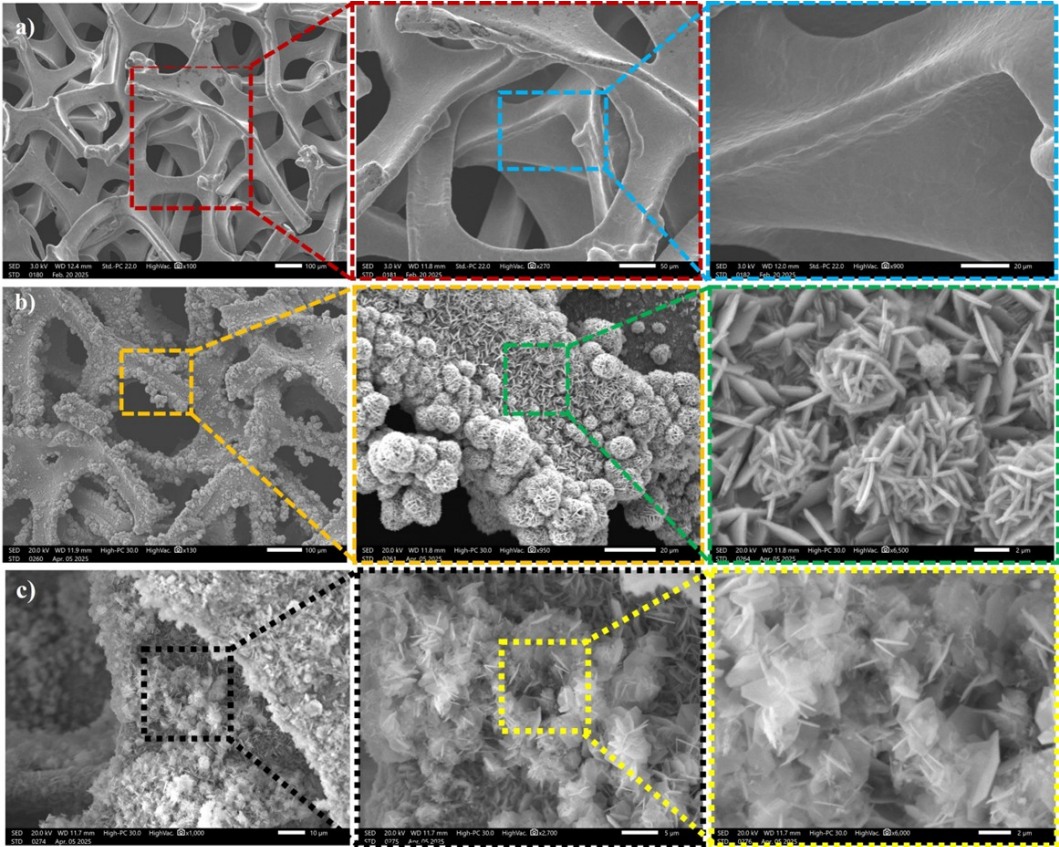


FIG. 2. SEM images of a) NF, b) CoFeNi/NF, c) CoFeNi–MF/NF

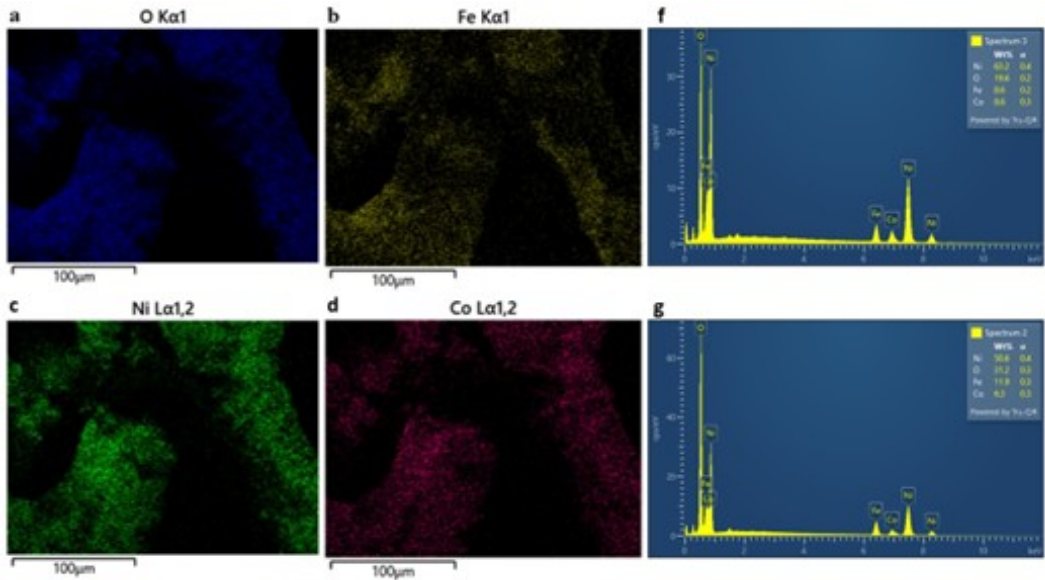


FIG. 3. a-d) EDS elemental mapping of f) EDS spectrum of CoFeNi/NF and g) EDS spectrum of CoFeNi–MF/NF

The increased Fe content in the CoFeNi–MF/NF confirms successful MF integration and magnetic nanoparticle loading. Crystallinity and phase identification were examined via XRD patterns (Fig. 4(a)). For CoFeNi/NF, three main peaks were observed at  $2\theta \approx 44.5^\circ$ ,  $51.8^\circ$ , and  $76.4^\circ$ , corresponding to the (111), (200), and (220) planes of the face-centered cubic (FCC) structure, characteristic of Ni–Co–Fe alloys. Upon addition of MF, additional weak diffraction peaks appeared near  $2\theta \approx 30.1^\circ$ ,  $31.5^\circ$ , and  $39.2^\circ$ , which can be indexed to (220), (100), and (111) planes of  $\text{Fe}_3\text{O}_4$  (magnetite). Although the characteristic peaks are weak, likely due to the relatively low content or amorphous nature of MF nanoparticles, their presence supports the partial incorporation of  $\text{Fe}_3\text{O}_4$  into the composite structure. The average crystallite size was estimated using the Scherrer formula, where the broadening of the FCC peaks indicated size variation. For CoFeNi/NF, the crystallite size was calculated to be approximately 18.2 nm, whereas for CoFeNi–MF/NF, it slightly decreased to 16.3 nm, suggesting that MF addition leads to finer nanostructures. This reduction enhances the surface-to-volume ratio, which is beneficial for electrocatalytic activity. In summary, the combined SEM, EDS, and XRD results confirm the successful formation of a uniform, high-surface-area, and crystalline CoFeNi–MF/NF electrode. The structural features – nanosheet morphology, MF coating, and increased oxygen content – collectively enhance active site density, electrical conductivity, and catalytic efficiency for HER and OER processes.

The chemical structure and bonding environment of the CoFeNi/NF electrocatalyst were further investigated through Fourier-transform infrared (FTIR) spectroscopy (Fig. 4(b)). The FTIR spectrum exhibits several characteristic vibrational bands corresponding to oxygen-containing surface groups, residual solvent species, and metal-oxygen lattice modes, confirming the successful formation of a multimetal oxide/hydroxide network on the substrate. A broad absorption band centered at  $3378\text{ cm}^{-1}$  is attributed to the stretching vibrations of surface hydroxyl groups (O–H). Such a broad O–H mode is typical for metal hydroxides and physically adsorbed water molecules, indicating that the catalyst surface is rich in hydroxyl terminations. In alkaline electrocatalysis, the presence of abundant O–H groups plays an essential role in accelerating water dissociation during the Volmer step of HER and contributes to the formation of MOOH intermediates during OER, thereby enhancing both catalytic pathways. A weaker band observed near  $2357\text{ cm}^{-1}$  corresponds to physically adsorbed  $\text{CO}_2$  species, which are commonly detected on metal oxide surfaces exposed to air. The small vibrational feature around  $2181\text{ cm}^{-1}$  is associated with  $\text{C}\equiv\text{N}$  or  $\text{–CN}$  stretching, likely originating from residual urea-derived intermediates from the hydrothermal synthesis. The presence of such organic residuals is minimal and does not affect the structural or catalytic properties of the final material. The band located at  $1635\text{ cm}^{-1}$  is assigned to the bending vibration of molecular water (H–O–H), confirming the presence of adsorbed water molecules trapped within the porous nanosheet framework. The peak at  $1356\text{ cm}^{-1}$  corresponds to C–O stretching vibrations, which originate from surface carbonate or adsorbed organic groups, often introduced during washing or drying. Most importantly, the low-wavenumber region ( $500\text{--}800\text{ cm}^{-1}$ ) provides direct structural evidence for the formation of mixed metal-oxygen frameworks. A pronounced absorption peak at  $693\text{ cm}^{-1}$  is characteristic of Co–O stretching vibrations, confirming the presence of cobalt oxide/hydroxide phases such as  $\text{Co}(\text{OH})_2$  or  $\text{CoOOH}$ -related species. The absorption at  $603\text{ cm}^{-1}$  corresponds to the Fe–O vibrational mode, demonstrating the incorporation of iron oxides/hydroxides such as  $\text{FeOOH}$  or  $\text{Fe}_2\text{O}_3$  into the catalyst lattice. Additionally, the peak at  $545\text{ cm}^{-1}$  is assigned to the Ni–O stretching mode, confirming the presence of nickel-based oxide/hydroxide species (likely  $\text{Ni}(\text{OH})_2$  or  $\text{NiOOH}$ ). The simultaneous appearance of Co–O, Fe–O, and Ni–O bands demonstrates the formation of a trimetallic oxide/hydroxide network, which aligns closely with the XPS findings showing multivalent  $\text{Co}^{2+}/\text{Co}^{3+}$ ,  $\text{Fe}^{2+}/\text{Fe}^{3+}$ , and  $\text{Ni}^{2+}/\text{Ni}^{3+}$  species. The presence of multiple metal-oxygen vibrational bands suggests strong electronic interactions among the three metals, which can modulate the local electronic environment, enhance charge-transfer processes, and facilitate the formation of high-valence oxyhydroxide intermediates under OER conditions. Collectively, the FTIR analysis supports the successful synthesis of a multimetal oxide/hydroxide framework on the NF substrate, characterized by abundant hydroxyl groups, adsorbed water, and well-defined M–O lattice modes for Co, Fe, and Ni. These structural features are directly relevant to the electrocatalytic performance of the material. The hydroxyl-rich surface promotes water adsorption and dissociation during HER, whereas the presence of strong M–O stretching vibrations confirms the existence of catalytically active Co–O, Fe–O, and Ni–O units that undergo in situ transformation into high-valent MOOH phases during OER. These structural characteristics synergistically contribute to the experimentally observed low overpotentials and efficient bifunctional activity of the CoFeNi/NF catalyst.

The surface chemical composition and electronic structure of the CoFeNi/NF electrocatalyst were comprehensively investigated through X-ray photoelectron spectroscopy (XPS). The wide-scan spectrum confirms the presence of Co, Fe, Ni, O, and C elements, indicating the successful formation of a trimetallic hydroxide/oxide layer on the nickel foam substrate. Distinct Co 2p, Fe 2p, and Ni 2p signatures demonstrate that all three metals exist in mixed-valence states, which is a crucial electronic feature governing the bifunctional electrocatalytic behavior of multimetallic catalysts in alkaline media. The Co 2p high-resolution region reveals two main spin–orbit doublets centered at 780.6 eV (Co  $2p_{3/2}$ ) and 796.1 eV (Co  $2p_{1/2}$ ), accompanied by satellite peaks located near 786.1 and 802.3 eV. Deconvolution identifies the coexistence of  $\text{Co}^{2+}$  (780.4 eV) and  $\text{Co}^{3+}$  (781.6 eV) components, along with a minor metallic  $\text{Co}^0$  (778.2 eV) contribution. The mixed  $\text{Co}^{2+}/\text{Co}^{3+}$  states suggest that the catalyst surface is partially oxidized, enabling rapid  $\text{Co}^{2+} \leftrightarrow \text{Co}^{3+}$  redox cycling during the oxygen evolution reaction (OER). The presence of  $\text{Co}^{3+}$  is strongly associated with the formation of  $\text{CoOOH}$  intermediates, known to serve as highly active OER centers, whereas  $\text{Co}^{2+}$  species contribute to hydrogen adsorption/desorption during the hydrogen evolution reaction (HER). The metallic  $\text{Co}^0$  fraction further enhances electrical conductivity, thereby

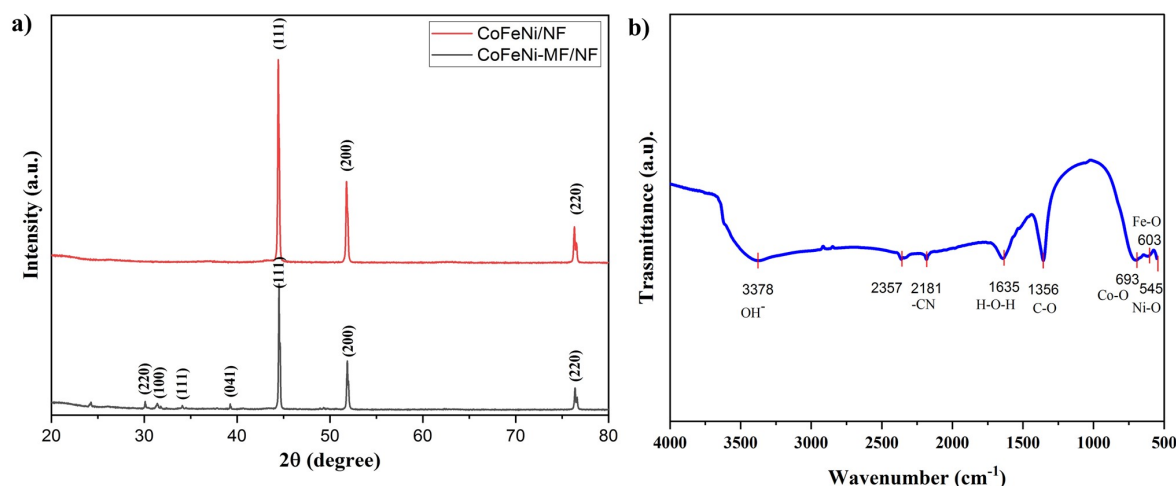


FIG. 4. a) XRD analysis of synthesized CoFeNi/NF and CoFeNi-MF/NF b) FT-IR spectra of CoFeNi MF/NF

promoting charge mobility across the electrode-electrolyte interface. The Fe 2p spectrum displays characteristic peaks at 710.8 eV (Fe 2p<sub>3/2</sub>) and 724.9 eV (Fe 2p<sub>1/2</sub>), with satellite features positioned at 717.2 eV, indicating the presence of oxidized iron species. Peak deconvolution identifies Fe<sup>2+</sup> (710.4 eV), Fe<sup>3+</sup> (712.0 eV), and a weak Fe<sup>0</sup> (707.3 eV) component. The Fe<sup>2+</sup>/Fe<sup>3+</sup> redox couple plays a significant role in tuning the electronic configuration of the adjacent Co and Ni sites. Numerous studies have shown that Fe incorporation into Co- or Ni-based systems facilitates lattice oxygen activation and enhances OER kinetics through the formation of FeOOH motifs. The strong intensity of Fe<sup>3+</sup> indicates that the catalyst surface possesses high-valence iron sites capable of participating in proton-electron-coupled steps that are essential for O–O bond formation. The Ni 2p spectrum shows two main peaks at 855.8 eV (Ni 2p<sub>3/2</sub>) and 873.3 eV (Ni 2p<sub>1/2</sub>), along with pronounced satellite features located near 861.4 and 879.2 eV. Deconvolution confirms the presence of Ni<sup>2+</sup> (855.4 eV) and Ni<sup>3+</sup> (856.5 eV) species. The existence of Ni<sup>3+</sup> indicates that the catalyst surface readily transforms into NiOOH under anodic polarization, which is recognized as a primary active phase for OER. The Ni<sup>2+</sup>/Ni<sup>3+</sup> couple is also indicative of facile electronic reconstruction, enabling fast electron exchange during reaction cycles. The significant proportion of Ni<sup>3+</sup> species correlates well with the experimentally observed low OER overpotential and favorable Tafel slope, demonstrating that Ni plays a key role in accelerating reaction kinetics. The O 1s spectrum consists of two major components: lattice oxygen (M–O) at 529.7 – 530.1 eV and hydroxyl/adsorbed oxygen species (M–OH, O<sub>ads</sub>) at 531.4 – 531.9 eV. The dominant M–OH contribution implies that the catalyst surface is rich in hydroxyl terminations – an essential characteristic for alkaline electrocatalysis. During HER, surface hydroxyl groups facilitate rapid water dissociation (Volmer step), lowering the activation barrier for H<sup>\*</sup> formation. During OER, these hydroxyl groups participate in generating MOOH intermediates, which function as catalytically active sites for O–O coupling. The C 1s peak at 284.6 eV (C–C/C–H) and 286.2 eV (C–O) originates from surface adventitious carbon and the underlying substrate. Overall, the XPS results demonstrate that the CoFeNi/NF catalyst possesses a highly modulated electronic environment characterized by mixed-valence Co<sup>2+</sup>/Co<sup>3+</sup>, Fe<sup>2+</sup>/Fe<sup>3+</sup>, and Ni<sup>2+</sup>/Ni<sup>3+</sup> species, combined with partially metallic Co<sup>0</sup>, Fe<sup>0</sup>, and Ni<sup>0</sup> domains. This multivalence is a hallmark of highly active multimetal catalysts, as it enhances the density of redox-active centers, facilitates rapid electron transfer, and enables dynamic surface reconstruction under electrochemical conditions. The strong electronic coupling among Co, Fe, and Ni likely alters the local density of states near the Fermi level, optimizing adsorption energies for reaction intermediates and reducing overall reaction barriers. These electronic features are fully consistent with the catalyst's superior HER and OER behavior. In alkaline HER, the presence of metallic sites (Co<sup>0</sup>/Ni<sup>0</sup>), moderate-strength hydrogen-binding centers (Co<sup>2+</sup>, Ni<sup>2+</sup>), and hydroxyl-rich surfaces work synergistically to improve water dissociation and hydrogen release kinetics, resulting in a low HER overpotential. In OER, the abundance of high-valence Co<sup>3+</sup>, Fe<sup>3+</sup>, and Ni<sup>3+</sup> species strongly favors the formation of active oxyhydroxide intermediates (CoOOH, FeOOH, NiOOH), which accelerate the proton-electron transfer steps necessary for oxygen evolution. The observed catalytic performance therefore directly originates from the unique electronic structure revealed by XPS, confirming that the hydrothermal synthesis strategy successfully produces a highly active, electronically optimized trimetallic electrocatalyst.

### 3.1. Electrochemical measurement for OER

To determine the electrochemical activity of the synthesized electrocatalyst samples in 1.0 M KOH alkaline electrolyte, a conventional three-electrode setup was used. LSV polarization curves were collected to assess catalytic activity and oxidation-reduction/catalytic indicators for HER and OER. The results for OER showed that the CoFeNi-MF/NF catalyst achieved maximum activity. For comparison, CoFeNi/NF, MF/NF, and NF were analyzed under the same conditions. As shown in Fig. 6(a), CoFeNi-MF/NF exhibited higher OER efficiency compared to other catalysts. The OER

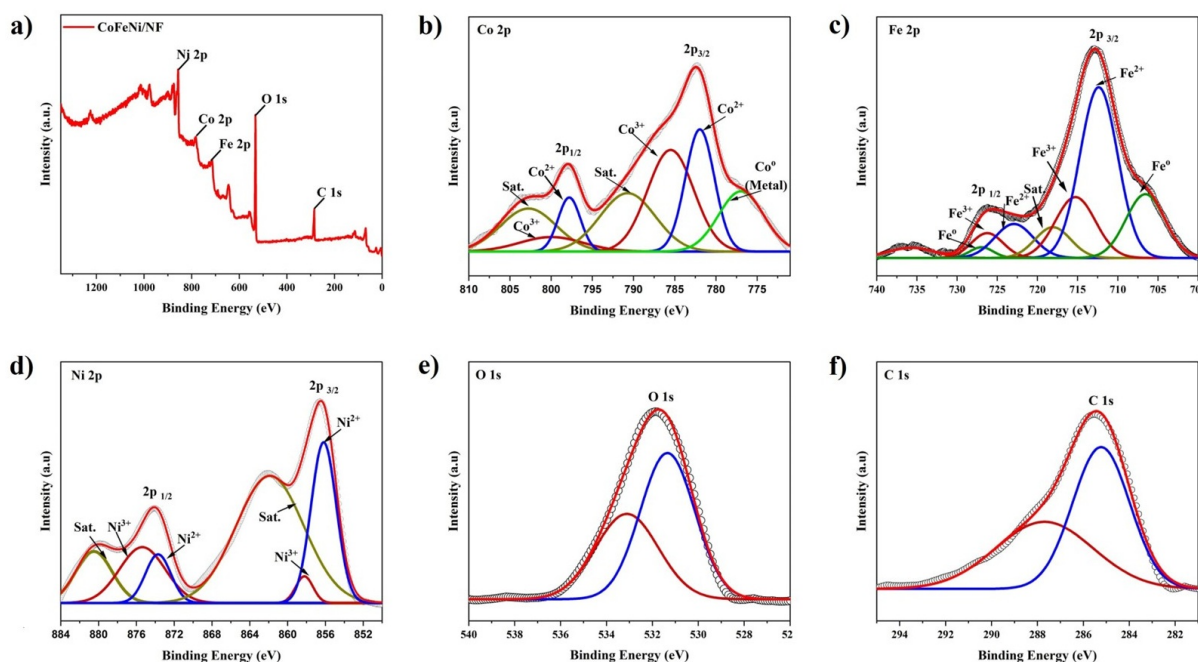


FIG. 5. X-ray photoelectron spectrum of CoFeNi-NF: a) survey spectrum, b) Co 2p, c) Fe 2p, d) Ni 2p, e) O 1s and f) C 1s

overpotential was only 199 mV at  $10 \text{ mA} \cdot \text{cm}^{-2}$ . However, CoFeNi/NF and MF/NF required higher overpotentials of 310 and 217 mV at a current density of  $10 \text{ mA} \cdot \text{cm}^{-2}$ , respectively. All catalysts contain NF, and to demonstrate the catalytic activity of NF, we compared the catalytic performance of bare NF. The results showed that NF had an overpotential of 470 mV at  $10 \text{ mA} \cdot \text{cm}^{-2}$ . This proves that adding MF during the synthesis process can improve the catalytic effect. LSV tests with different catalysts are shown in detail in Fig. 6(a), which confirms the superior performance of CoFeNi-MF/NF for OER among these catalysts. It was determined that the thermodynamic efficiency of the process for the CoFeNi-MF/NF electrode under standard conditions is 86 %, while for CoFeNi/NF, MF/NF, and NF electrodes it is 80, 85, and 72 %, respectively, which indicates the ratio of useful energy output to total energy input. This efficiency value reflects how effectively the system can convert the supplied energy under these controlled conditions into the required chemical or electrical energy. Achieving high thermodynamic efficiency is crucial as it directly affects the overall energy efficiency and stability of the system. Additionally, the kinetics and mechanism of the OER reaction were assessed by measuring the corresponding Tafel slope. It is known that OER exhibits slow kinetics and presents a barrier in the water splitting process due to its multi-electron pathway. The results showed that the Tafel slope of CoFeNi-MF/NF ( $91.41 \text{ mV} \cdot \text{dec}^{-1}$ ) is significantly lower than that of CoFeNi/NF ( $81.69 \text{ mV} \cdot \text{dec}^{-1}$ ), MF/NF ( $236.76 \text{ mV} \cdot \text{dec}^{-1}$ ), and NF ( $150.68 \text{ mV} \cdot \text{dec}^{-1}$ ), as seen in Fig. 6(b). This indicates that the reaction kinetics in CoFeNi-MF/NF are optimal. The low Tafel slope of the CoFeNi-MF/NF catalyst implies a rapid dynamic reaction rate, which significantly enhances the catalyst's efficiency. Fig. 6(c) presents the electrochemical impedance spectroscopy (EIS) results for CoFeNi-MF/NF, conducted in the frequency range of 0.01 – 100 Hz at 1.5 V versus RHE. The figure includes the impedance spectrum and its equivalent circuit. As evident from Fig. 6(c), the charge transfer resistance ( $R_{ct}$ ) of CoFeNi-MF/NF was determined to be  $1.0 \Omega$ . This low value indicates good electrical conductivity rapid surface charge transfer. The electrochemically active surface area (ECSA) is a key parameter of electrocatalysts, representing the portion of the electrode surface that actively participates in electrochemical reactions. Unlike the geometric surface area, which only considers the physical dimensions of the electrode, ECSA reflects the actual number of accessible catalytic sites available for charge transfer. Higher ECSA typically enhances electrocatalytic efficiency by increasing the number of available reaction sites for key processes such as OER and HER. Double-layer capacitance,  $C_{dl}$ , is a parameter representing the capacitance of the electrical double layer formed at the interface between the electrode surface and the electrolyte solution. When a potential is applied to the electrode, ions from the electrolyte are attracted to the surface, forming a double layer with a capacitance that depends on the electrode's surface area. To determine the electrochemical surface area (ECSA), the double-layer capacitance ( $C_{dl}$ ) is initially obtained from cyclic voltammetry (CV) measurements in the non-Faradaic region. This is achieved by plotting the average current density against the scan rate, where the resulting slope corresponds to the  $C_{dl}$  value. Fig. 6(d) shows the cyclic voltammetry (CV) curves for the CoFeNi-MF/NF electrode, recorded at different scan rates (1, 2, 3, 4, and 5 mV/s). These curves demonstrate the capacitive behavior of the electrode in the non-Faradaic region, confirming the formation of an electrochemical double layer. A gradual increase in current density with increasing scan rate indicates improved charge storage capabilities and surface area influence. The linear fit of  $\Delta i/2$  versus scan rate

to estimate  $C_{dl}$  yields a capacitance value of  $32.84 \text{ mF}\cdot\text{cm}^{-2}$  for OER, as shown in Fig. 6(e). This relatively high  $C_{dl}$  suggests that the CoFeNi-MF/NF composite possesses a large active surface area, which facilitates charge accumulation and enhances reaction kinetics during water oxidation. To better understand the origin of the enhanced OER activity, the electrochemically active surface area (ECSA) of the electrodes was evaluated based on the double-layer capacitance ( $C_{dl}$ ). In alkaline media, a specific capacitance ( $C_s$ ) value of  $0.04 \text{ mF cm}^{-2}$  was used to estimate the ECSA,

$$ECSA = \frac{C_{dl}}{C_s}.$$

As presented in Fig. 6(e), the  $C_{dl}$  values increase progressively from NF to CoFeNi-MF/NF, suggesting that the surface becomes more electrochemically accessible after each modification step. The calculated ECSA values are 280, 631, 631, and  $821 \text{ cm}^2$  for NF, MF/NF, CoFeNi/NF, and CoFeNi-MF/NF, respectively. Among them, CoFeNi-MF/NF shows the highest  $C_{dl}$  ( $32.84 \text{ mF cm}^{-2}$ ), indicating that more active sites are exposed to the electrolyte. This increase is likely related to the formation of a more open and rough nanosheet structure after the incorporation of magnetic fluid. A larger ECSA generally means that more reaction sites are available, which helps explain the improved OER performance. Therefore, the enhanced catalytic behavior of CoFeNi-MF/NF can be reasonably attributed, at least in part, to its increased electrochemically active surface area.

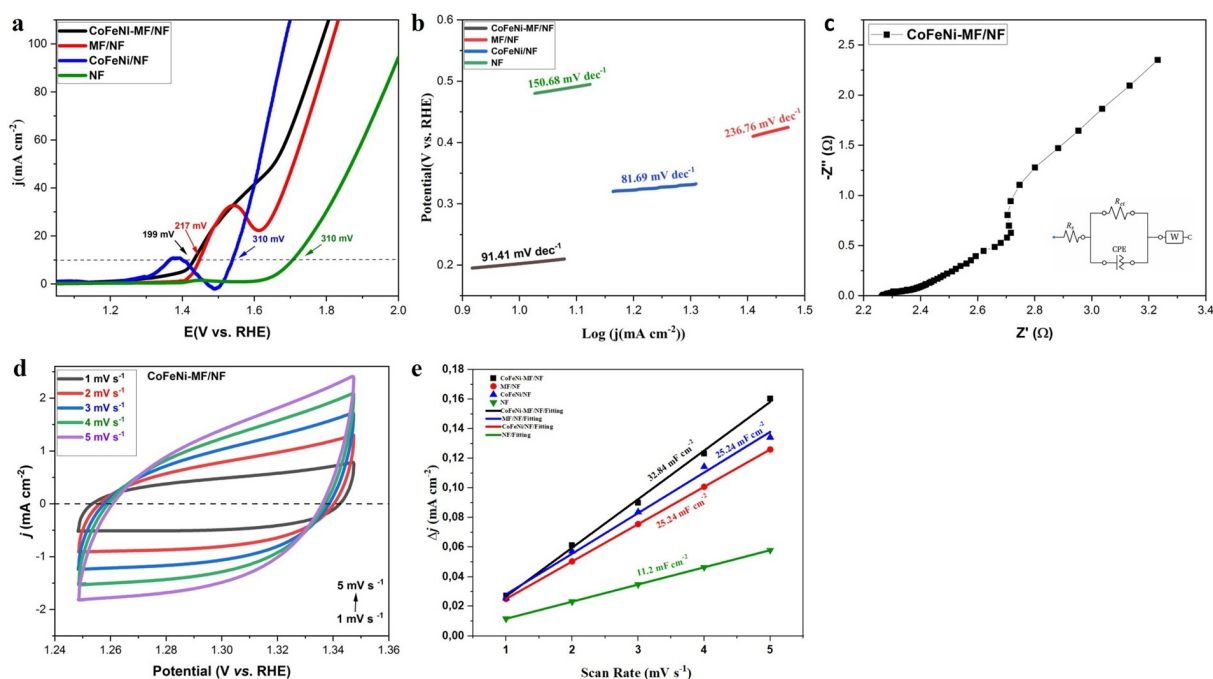


FIG. 6. OER performance under 1 M KOH: a) LSV polarization curves of asprepared catalysts for OER; b) Corresponding Tafel plots; c) Nyquist plot over the frequency range d) CV of CoFeNi-MF/NF catalyst at different scan rates; e) The differences in current density variation ( $\Delta j = j_a - j_c$ ) plotted against scan rate fitted to a linear regression for  $C_{dl}$  estimation

### 3.2. Electrochemical measurement for HER

The performance of the synthesized catalysts for HER was studied. Among these catalysts, CoFeNi-MF/NF in 1 M KOH medium requires the lowest overpotential of 169 mV to achieve a current density of  $10 \text{ mA}\cdot\text{cm}^{-2}$ , as shown in Fig. 7(a). Similarly, the HER performance of CoFeNi/NF, MF/NF, and NF under 1 M KOH was compared. CoFeNi/NF requires overpotentials of 284, 185, and 304 mV to drive a catalytic current density of  $10 \text{ mA}\cdot\text{cm}^{-2}$  for HER. This demonstrates a significant difference compared to CoFeNi-MF/NF, further highlighting the efficiency advantage of CoFeNi-MF/NF in electrocatalysis. Additionally, Fig. 7(b) shows the Tafel slope values of CoFeNi-MF/NF ( $133.09 \text{ mV}\cdot\text{dec}^{-1}$ ), CoFeNi/NF ( $240.08 \text{ mV}\cdot\text{dec}^{-1}$ ), MF/NF ( $174.26 \text{ mV}\cdot\text{dec}^{-1}$ ), and NF ( $169.54 \text{ mV}\cdot\text{dec}^{-1}$ ). The mechanisms of HER and OER reactions are believed to be based on several factors, such as Tafel slope, electrolyte, and others. Generally, the Volmer reaction involves electron adsorption and proton discharge in the first stage, followed by electrodesorption and the Heyrovsky reaction. The results showed that the CoFeNi-MF/NF catalyst exhibits a Tafel slope of  $133.09 \text{ mV}\cdot\text{dec}^{-1}$ , suggesting Volmer–Heyrovsky pathways with adsorption as the rate-limiting step.

The kinetics and charge transfer properties of the synthesized CoFeNi-MF/NF catalysts were studied using electrochemical impedance spectroscopy (EIS). In the low-frequency range, the small semicircle in the impedance data arises

from the faradaic resistance due to charge transfer at the electrode/electrolyte interface. The CoFeNi–MF/NF catalyst showed an  $R_{ct}$  value of  $7.0\ \Omega$ , as seen in Fig. 7(c). The study revealed that a low  $R_{ct}$  at the electrode/electrolyte interface can enhance the electron transport properties of nanostructured materials, thereby increasing catalytic efficiency. The  $C_{dl}$  method was used to calculate the ECSA value of the prepared catalysts. CV measurements were performed at various scan rates of  $1 - 5\ \text{mV}\cdot\text{s}^{-1}$  in the non-faradaic region in a  $1.0\ \text{M}\ \text{KOH}$  medium. As shown in Fig. 7(d–e), the CoFeNi–MF/NF catalyst exhibits a significant  $C_{dl}$  value of  $58.2\ \text{mF}\ \text{cm}^{-2}$ . Such a surface area provides more contact points between the electrolyte and the catalytic surface. A similar analysis was performed for the HER process to further clarify the relationship between the electrochemically active surface area and catalytic performance. The ECSA values were estimated from the  $C_{dl}$  using the same method described above. As shown in Fig. 7(e), the  $C_{dl}$  values differ significantly among the samples, indicating noticeable changes in the accessible surface area after each modification step. The corresponding ECSA values were calculated to be  $240\ \text{cm}^2$  for NF,  $585\ \text{cm}^2$  for MF/NF,  $1102\ \text{cm}^2$  for CoFeNi/NF, and  $1455\ \text{cm}^2$  for CoFeNi–MF/NF. Among all the electrodes, CoFeNi–MF/NF exhibits the highest  $C_{dl}$  value ( $58.2\ \text{mF}\ \text{cm}^{-2}$ ), which corresponds to the largest ECSA. This suggests that the combined introduction of CoFeNi and magnetic fluid leads to a substantial increase in the number of exposed active sites. As a result, faster reaction kinetics can be achieved, which is consistent with the improved HER activity observed in the polarization curves. Overall, the superior HER performance of CoFeNi–MF/NF can be attributed, at least in part, to its significantly enlarged active surface area, although the contribution of intrinsic catalytic activity should also be considered. Fig. 7(f) shows a chronoamperometric stability test of the CoFeNi–MF/NF electrode at  $100\ \text{mA}\cdot\text{cm}^{-2}$ , where the stability potential over 24 hours demonstrates robust structural integrity and excellent long-term durability, which are necessary for the practical application of electrocatalysts.

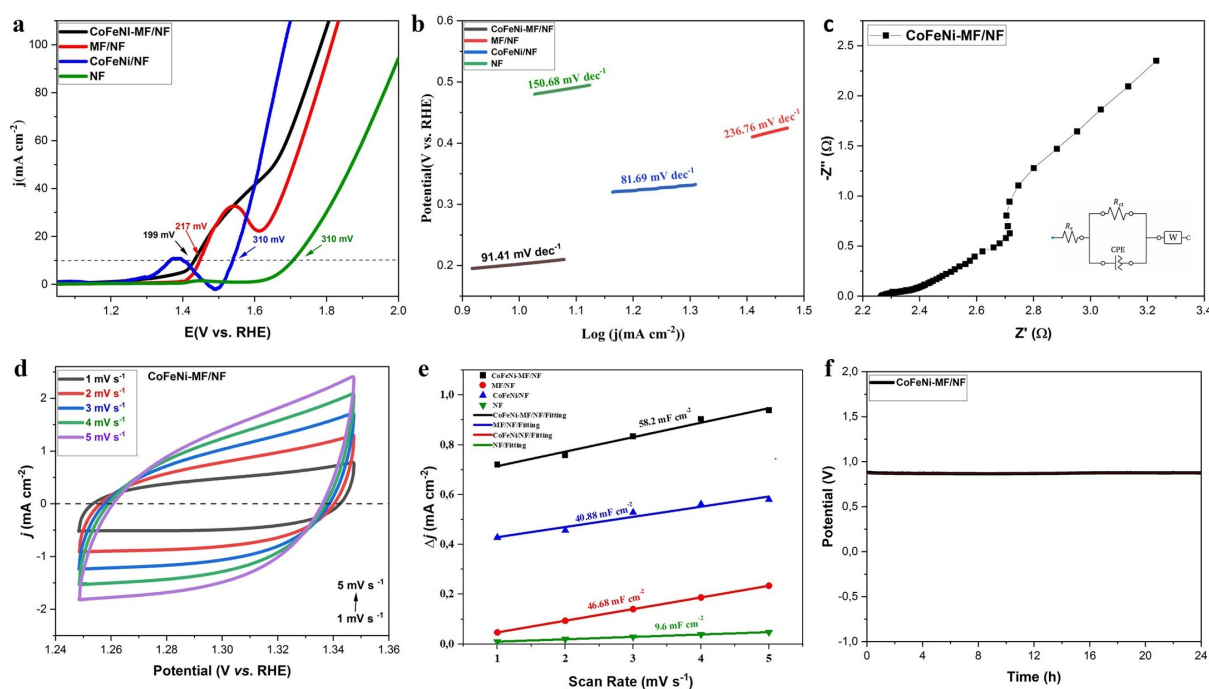


FIG. 7. HER performance under 1 M KOH: a) LSV polarization curves of asprepared catalysts for HER; b) Corresponding Tafel plots; c) Nyquist plot over the frequency range d) CV of CoFeNi–MF/NF catalyst at different scan rates; e) The differences in current density variation ( $\Delta j=j_a-j_c$ ) plotted against scan rate fitted to a linear regression for  $C_{dl}$  estimation; and f) Chronoamperometry test at a current density of  $100\ \text{mA}\cdot\text{cm}^{-2}$  for

The comparison of this research with other similar works is shown in the Table 1. The unique nanomassive morphology based on CoFeNi–MF/NF provided a significantly active surface for OER and HER, contributing to competitive overvoltage at  $10\ \text{mA}\cdot\text{cm}^{-2}$  and Tafel slopes. CoFeNi–MF/NF is a promising choice for water electrolysis.

#### 4. Conclusion

In this study, a highly efficient bifunctional electrocatalyst, CoFeNi–MF/NF, was rationally designed and synthesized through a two-step hydrothermal approach involving the integration of  $\text{Fe}_3\text{O}_4$ -based magnetic fluid into a CoFeNi nanosheet framework. Structural characterizations including XRD, SEM, EDS, FTIR, and XPS collectively confirm that the incorporation of MF introduces a dual-phase composition composed of FCC-type CoFeNi alloy and spinel  $\text{Fe}_3\text{O}_4$ , accompanied by the formation of vertically aligned nanosheets with significantly enhanced surface roughness

TABLE 1. Comparative analysis of this study with other similar works

| Electrocatalyst                                                 | Current density<br>(mA·cm <sup>-2</sup> ) | Overpotential |     | Tafel slopes mV·dec <sup>-1</sup> |       | Reference |
|-----------------------------------------------------------------|-------------------------------------------|---------------|-----|-----------------------------------|-------|-----------|
|                                                                 |                                           | HER           | OER | HER                               | OER   |           |
| Co <sub>0.45</sub> Fe <sub>0.45</sub> Ni <sub>0.9</sub> -MOF/NF | 50                                        | 225           | 244 | 91.6                              | 127   | [19]      |
| NFC@CNSs-700                                                    | 10                                        | 213           | 256 | 21.95                             | 61.29 | [24]      |
| CoFeNi-MF/NF                                                    | 10                                        | 169           | 199 | 91                                | 91.41 | This work |
| CoFeNi-/NF                                                      | 10                                        | 270           | 310 | 92                                | 81.69 | This work |

and defect density. The uniform dispersion of Co, Fe, Ni, and O, as revealed by elemental mapping, highlights the successful construction of a chemically and electronically interconnected multimetallic architecture. The introduction of Fe<sub>3</sub>O<sub>4</sub> nanoparticles via magnetic fluid not only increases the Fe and O content in the composite but also establishes stronger electronic coupling and magnetic-field-induced polarization effects among the Co-Fe-Ni centers. These synergistic interactions promote improved charge transfer, optimized adsorption/desorption energetics, and accelerated formation of active MOOH species under alkaline conditions. FTIR spectra further confirm the presence of abundant hydroxyl groups and well-defined M-O vibrational modes, while XPS analysis reveals the coexistence of mixed-valence states (Co<sup>2+</sup>/Co<sup>3+</sup>, Fe<sup>2+</sup>/Fe<sup>3+</sup>, Ni<sup>2+</sup>/Ni<sup>3+</sup>) and partially metallic domains, which collectively play a crucial role in enhancing reaction kinetics. Electrochemical measurements demonstrate that CoFeNi-MF/NF exhibits remarkable bifunctional activity toward both HER and OER in 1.0 M KOH. The catalyst requires low overpotentials of 169 mV (HER) and 199 mV (OER) at 10 mA·cm<sup>-2</sup>, significantly outperforming pristine CoFeNi/NF and MF/NF. Corresponding Tafel slopes of 133.09 mV·dec<sup>-1</sup> (HER) and 91.41 mV·dec<sup>-1</sup> (OER) further confirm rapid reaction kinetics facilitated by the magnetic-fluid-assisted architecture. The exceptionally low charge-transfer resistances – 7.0 Ω for HER and 1.0 Ω for OER-combined with the high double-layer capacitance values (58.2 mF cm<sup>-2</sup> for HER, 32.84 mF cm<sup>-2</sup> for OER) attest to the expanded electrochemically active surface area and superior intrinsic conductivity of the hybrid catalyst. The electrochemically active surface area (ECSA) analysis further confirms the superior catalytic performance of the CoFeNi-MF/NF electrode. For OER, the CoFeNi-MF/NF catalyst exhibits the highest ECSA value of 821 cm<sup>2</sup>, while for HER, it reaches 1455 cm<sup>2</sup>, both significantly higher than those of the reference samples. This increase in ECSA indicates a larger number of accessible active sites, which contributes to enhanced reaction kinetics in both HER and OER processes. These findings demonstrate that the improved electrocatalytic activity of CoFeNi-MF/NF is closely related to its enlarged active surface area. Long-term chronopotentiometry at 100 mA·cm<sup>-2</sup> for 24 hours demonstrates outstanding structural and electrochemical durability, validating the robustness of the CoFeNi-MF/NF system under practical operating conditions. These results collectively reveal that magnetic-fluid-induced modifications profoundly enhance both catalytic performance and stability by optimizing electron/spin transport, strengthening interfacial interactions, and enabling efficient gas evolution dynamics. Overall, the CoFeNi-MF/NF nanocomposite developed in this work represents a promising, cost-effective, and scalable electrocatalyst for overall water splitting. The proposed design strategy-combining trimetallic alloys with magnetic-fluid-assisted surface engineering-offers a versatile platform for future development of advanced magnetic-field-responsive catalysts for industrial hydrogen production under alkaline and potentially neutral media.

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*Conflicts of interest*

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

*Data availability*

Data associated with this study are available from the corresponding author upon request.