

Short Communication

FeMoO₄ Nanoprism Supported on Nickel Foam as Electro-catalyst for Oxygen Evolution Reaction

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Developing electrocatalyst with high efficiency and low cost for oxygen evolution reaction (OER) is of paramount importance to explore sustainable energy technologies. In this work, bimetallic FeMoO₄ nanoprism supported on nickel foam is synthesized through a simple one-step hydrothermal method. The optimal molar ratio of molybdenum to iron (1:1) of FeMoO₄ is investigated for OER. Benefiting from unique nanoprism structure, fast transmission of electrons or ions, rich exposure of surface active sites, and excellent conductivity enhanced by the substrate of nickel foam (NF), FeMoO₄-1/NF electrode exhibits excellent electrocatalytic performance with an overpotential of 284 mV to reach 100 mA cm⁻² in 1.0 M KOH. This work provides a promising strategy for water oxidation in alkaline solution.

Keywords: FeMoO₄; bimetallic; nanoprism; nickel foam; oxygen evolution reaction

1. INTRODUCTION

Over the past decade years, people have been looking for clean energy to replace fossil fuels [1-4]. Hydrogen is demonstrated as an ideal choice owing the green, renewable, friendly nature [5-8]. Water electrolysis technology provides an effective way for hydrogen generation. However, oxygen evolution reaction (OER) as the anode reaction of water electrolysis is a multi-step electron transfer process, which has a slow reaction rate and high overpotential [9-12]. Therefore, exploring a kind of highly efficient electrocatalyst toward OER is of great importance. RuO₂ and IrO₂ are considered to be the best electrocatalysts for oxygen evolution reaction. While the limited reserves and expensive cost makes them not suitable to large-scale production for electrocatalytic reactions. Thus, it is significant to explore highly active and non-precious electrocatalysts [13-19].

With the valence electron structures of $3d^{6-8}4s^2$, the transition-metal (Fe, Co, Ni) oxides, hydroxides, and their alloys all exhibit excellent OER activity [20-25]. Also, people have found that metal molybdates ($M\text{MoO}_4$), as a family of functional materials, exhibit outstanding catalytic property in many fields, such as lithium-ion batteries (LIBs), supercapacitors and electrocatalysis due to the high oxidation state of molybdenum [26-28]. For instance, Zhang et al. reported that FeMoO_4 nanorods synthesized by one-step solvothermal method can be used as an electrocatalyst material with excellent properties [30]. Furthermore, some of these catalysts have to be immobilized on electrode surfaces using a polymer binder such as Nafion or PTFE, which are not conducive to active sites exposure, thereby increasing resistance and decreasing the catalytic activity. Therefore, the supported electrocatalysts may be the promising choice for high conductivity and dispersion [31-35].

In this work, nickel foam with a unique three-dimensional structure is used as the substrate to support FeMoO_4 nanoprism with rich defects and high intrinsic activity by a facile one-step hydrothermal method. Meanwhile, we explore the optimal ratio of molybdenum to iron to achieve the best OER performance. The results show that the FeMoO_4 nanoprism with the molar ratio of 1:1 ($n_{\text{Mo}}:n_{\text{Fe}}$) exhibits the best OER performance compared with those of contrastive catalysts, which only demands an overpotential of 284 mV to reach a current density of 100 mA cm^{-2} in 1.0 M KOH.

2. EXPERIMENTAL SECTION

2.1 Chemicals and materials

All reagents are of analytical grade and were used without further purification. Nickel foam (NF) was purchased from Kunshan Kuangxun Electrical Co., Ltd. Sodium molybdate dihydrate ($\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$) and Iron (II) chloride tetrahydrate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$) were purchased from Sinopharm Chemical Reagent Co., Ltd. Deionized water ($18.2 \text{ M}\Omega \text{ cm}$ at $25 \text{ }^\circ\text{C}$) was used for experiments.

2.2 Preparation of NF based electrocatalysts

The preparation of FeMoO_4 -1 is as follows. Prior to use, the nickel foam (NF, $1 \times 2 \text{ cm}$) was ultrasonically cleaned in dilute sulfuric acid, acetone and then ethanol for 30 min respectively. 1.0008 g of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ and 0.8710 g of $\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$ were dissolved in 45 ml of water under magnetic stirring for 15 min to form uniform solution. Then, the brown solution obtained above was transferred into a 100 mL Teflon-lined stainless steel autoclave. The washed NF was immersed into the above solution. Then, the autoclave was heated at $100 \text{ }^\circ\text{C}$ for 24 h. After cooling to $25 \text{ }^\circ\text{C}$ naturally, the NF was taken out and washed with water and ethanol several times and then dried at $60 \text{ }^\circ\text{C}$ for later use.

The same procedures were applied to obtain FeMoO_4 -2 and FeMoO_4 -3 by changing the amount of $\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$ to 0.4355 g and 1.3065 g, respectively.

The preparation of FeO_x is similar to that of FeMoO_4 -1 except that 1.0008 g of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ and 0.8710 g of $\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$ are replaced by 2.0016 g of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$.

The preparation of MoO_x is similar to that of FeO_x except that 2.0016 g of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ is replaced by 1.7418 of $\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$.

2.3 Characterization

The crystal information was examined with X-ray diffraction (XRD) on X'Pert PRO MPD using Cu K α with 2θ range from 10° to 90° . X-ray photoelectron spectra (XPS) measurements were conducted to study the chemical states of the constituent elements via a VG ESCALABMK II photoelectron spectrometer with Al K α X-ray radiation as excitation source. Scanning electron microscopy (SEM) images (Hitachi S-4800) and high-resolution transmission electron microscopy (HRTEM) images (FEI Tecnai G², 200 kV) were conducted to reveal the microscopic morphology of the samples.

2.4 Electrochemical measurements

The electrocatalytic performances were investigated on a Gamry Reference 600 electrochemical workstation with a three-electrode system in 1.0 M KOH, using the NF based electrodes as the working electrode, platinum plate as the counter electrode, and saturated calomel electrode (SCE) as the reference electrode. The electrolyte of 1.0 M KOH was protected by O₂ during the process. All linear sweep voltammetry (LSV) polarization curves were recorded at a scan rate of 5 mV s⁻¹. Electrochemical impedance spectroscopy (EIS) data is performed with frequency from 10⁵ Hz to 0.1 Hz at an AC voltage of 0.4 V (vs. SCE). The electrochemical capacitance (C_{dl}) is determined by cyclic voltammograms (CVs) at increasing scan rates from 40 to 120 mV·s⁻¹. The iR correction to data with the series resistance (R_s) is performed by $\eta_{\text{corr}} = \eta - iR_s$. The potential conversion from SCE to RHE is based on the following equation: $E_{\text{RHE}} = E_{\text{SCE}} + 0.245 + 0.059 \times \text{pH}$.

3. RESULTS AND DISCUSSION

The XRD pattern of FeMoO₄ in Figure 1a shows a set of diffraction peaks at 13.0° , 23.0° , 26.2° , 32.5° , and 39.7° , corresponding to the (110), (021), (220), (022), and (330) planes of FeMoO₄ (PDF No. 00-022-0628) except for the peaks at 44.8° , 52.2° and 76.8° belonging to the NF substrate (PDF No. 00-003-1051). The FeMoO₄-2 and FeMoO₄-3 exhibit similar XRD patterns (Figure 1b), indicating that they are the same kind of FeMoO₄ only in different molar ratios. But the peak intensity of FeMoO₄-2 and FeMoO₄-3 are weaker than FeMoO₄-1, indicating FeMoO₄ prepared with the ratio of 1:1 (n_{Mo}:n_{Fe}) has higher purity and higher crystallinity than the molar ratio of 1:2 and 3:2. Therefore, we speculate the best ratio of Mo to Fe is 1:1.

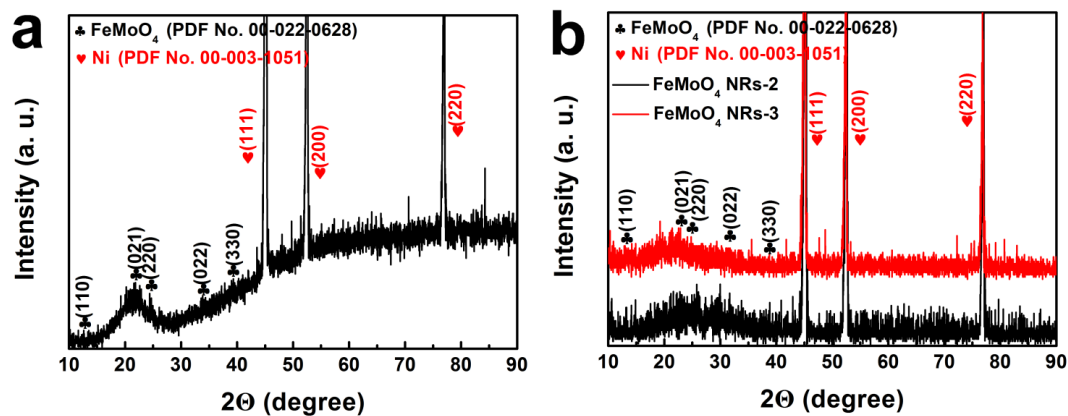


Figure 1. XRD patterns of (a) FeMoO₄-1, (b) FeMoO₄-2 and FeMoO₄-3.

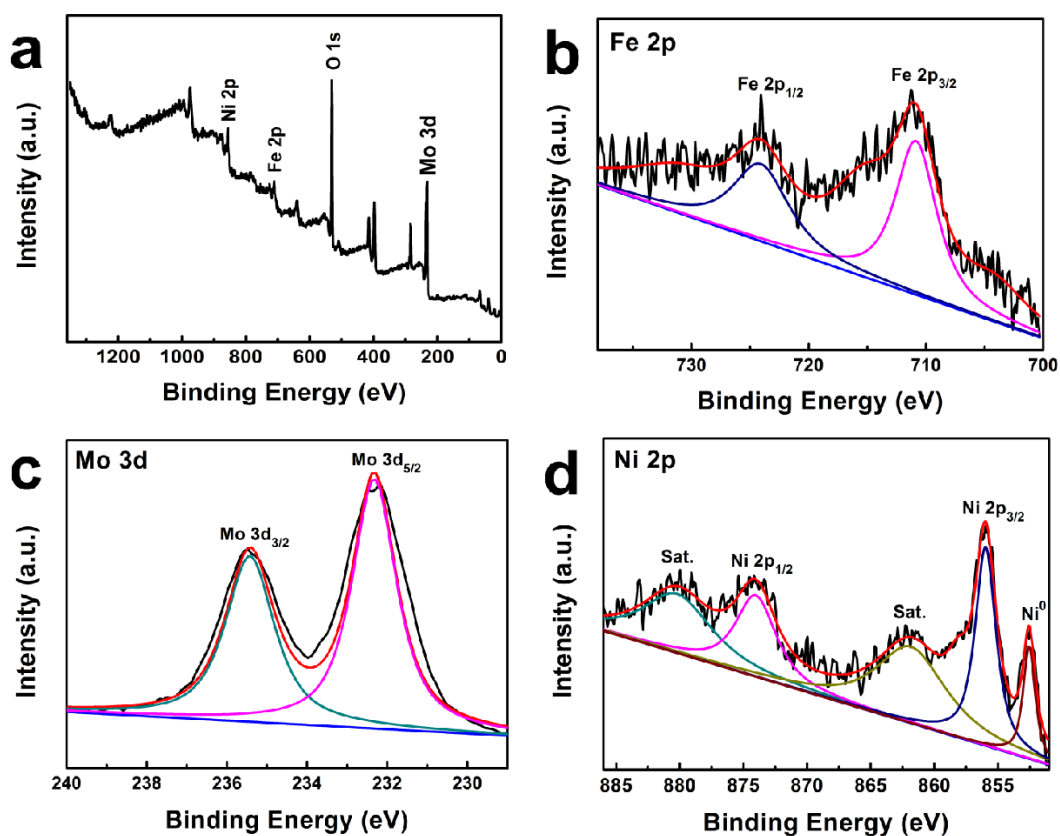


Figure 2. (a) Full XPS survey spectra of FeMoO₄-1, and XPS spectra of FeMoO₄-1 in the (b) Fe 2p; (c) Mo 3d and (d) Ni 2p regions

XPS measurement is further conducted to investigate the composition and valence state of FeMoO₄. FeMoO₄-1 was selected as the aimed electrocatalyst since it shows the best catalytic performances among all the prepared electrocatalysts (see detailed discussion below). As shown in Figure 2a, it shows that FeMoO₄ is composed of Fe, Mo, Ni and O as the major elements. From Figure 2b, the Fe 2p spectrum shows two peaks at 710.8 eV and 724.1 eV, which correspond to Fe 2p_{3/2} and Fe 2p_{1/2}, respectively [36]. As shown in Figure 2c, two peaks located at 232.3 eV and 235.4 eV can be

assigned to Mo 3d_{5/2} and Mo 3d_{3/2}, respectively, indicating the presence of high oxidation state of molybdenum, which is responsible for high catalytic activity [37]. Moreover, the XPS spectrum of Ni 2p (Figure 2d) show that binding energy peaks at 855.9 and 874.0 eV and associated satellite peaks at 861.8 and 880.2 eV, correspond to Ni 2p_{3/2} and Ni 2p_{1/2}, respectively. Moreover, the peak located at 852.5 eV belongs to Ni⁰. It is worth to note that element Ni derived from nickel foam can improve the conductivity and accelerate the diffusion of electrolytes and gas release.

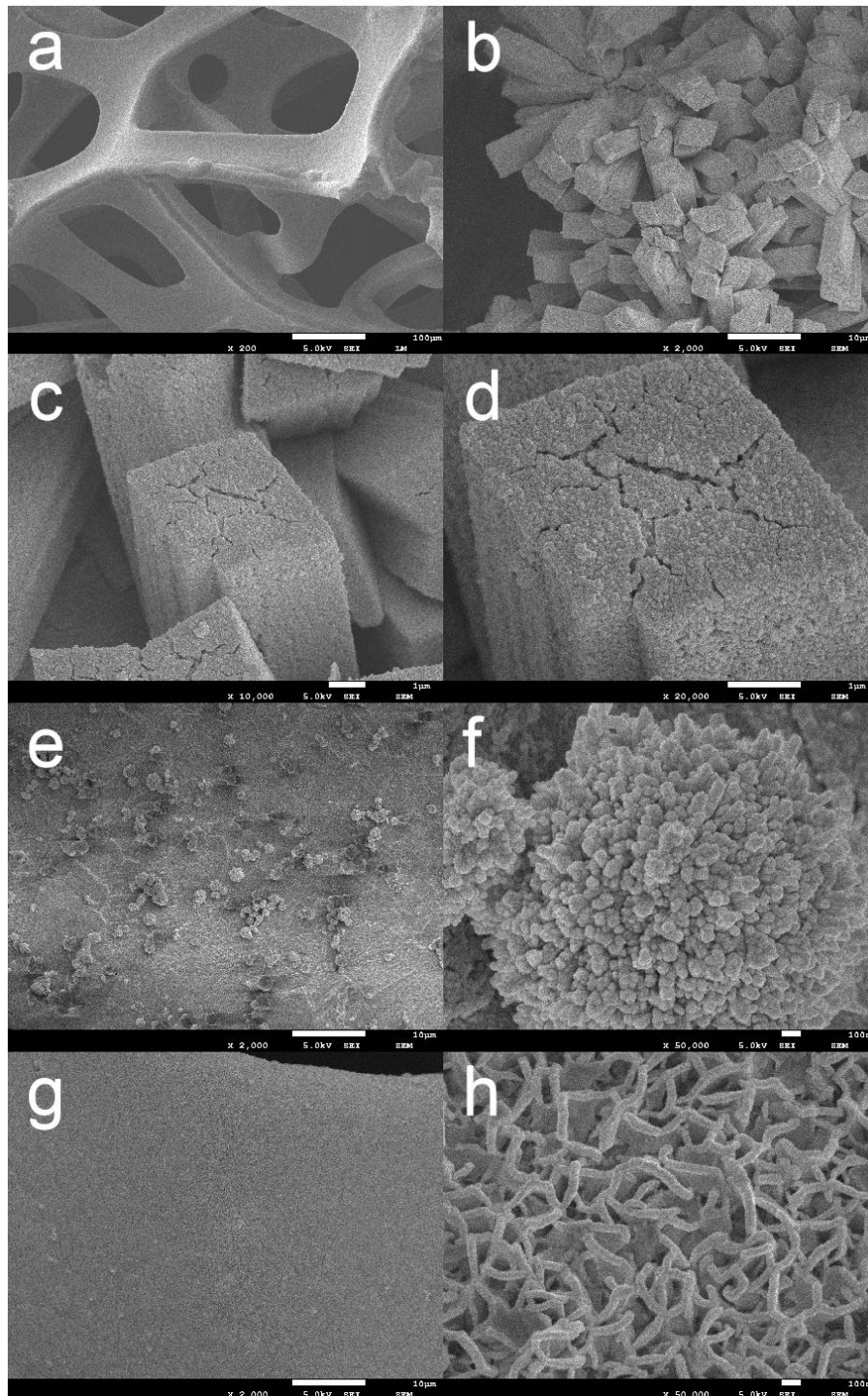


Figure 3. SEM images of (a) blank NF (b-d) FeMoO₄-1/NF (e, f) FeO_x/NF and (g, h) MoO_x/NF

The morphologies of blank NF, FeMoO₄-1, FeO_x and MoO_x are characterized by SEM (Fig 3). As shown in Figure 3a, blank NF with three-dimensional porous structure can expose active species and improve the conductivity. The SEM images of FeMoO₄-1 in Figure 3b-d reveal that a large number of uniform nanoprisms with an average length of 10 μm and diameter side length of 2.5 μm are formed. This unique structure provides excellent mass transfer channels for electrochemical catalytic processes and accelerate the transfer of electrons or ions. Figure 3e-f show many small balls are formed on the surface of FeO_x/NF, which are composed of sea urchin-like particles after being enlarged. Figure 3g-h show that uniform film composed of many vertical nanosheets with an average length of 200 nm is formed on the surface of MoO_x/NF.

HRTEM images of FeMoO₄-1 in Figure 4a-b also present prism-like morphology consistent with SEM results. As shown in Figure 4c, the edge of the prism is covered with rough film, which belongs to FeMoO₄. The loose structure facilitates the exposure of the active sites, thereby improving the catalytic performance of oxygen evolution reaction. In Figure 4d, the marked lattice fringe with d-spacing of 0.34 nm agrees well with the (220) plane of FeMoO₄ nanoprism. All of these characterizations demonstrate the successful preparation of FeMoO₄/NF nanoprism.

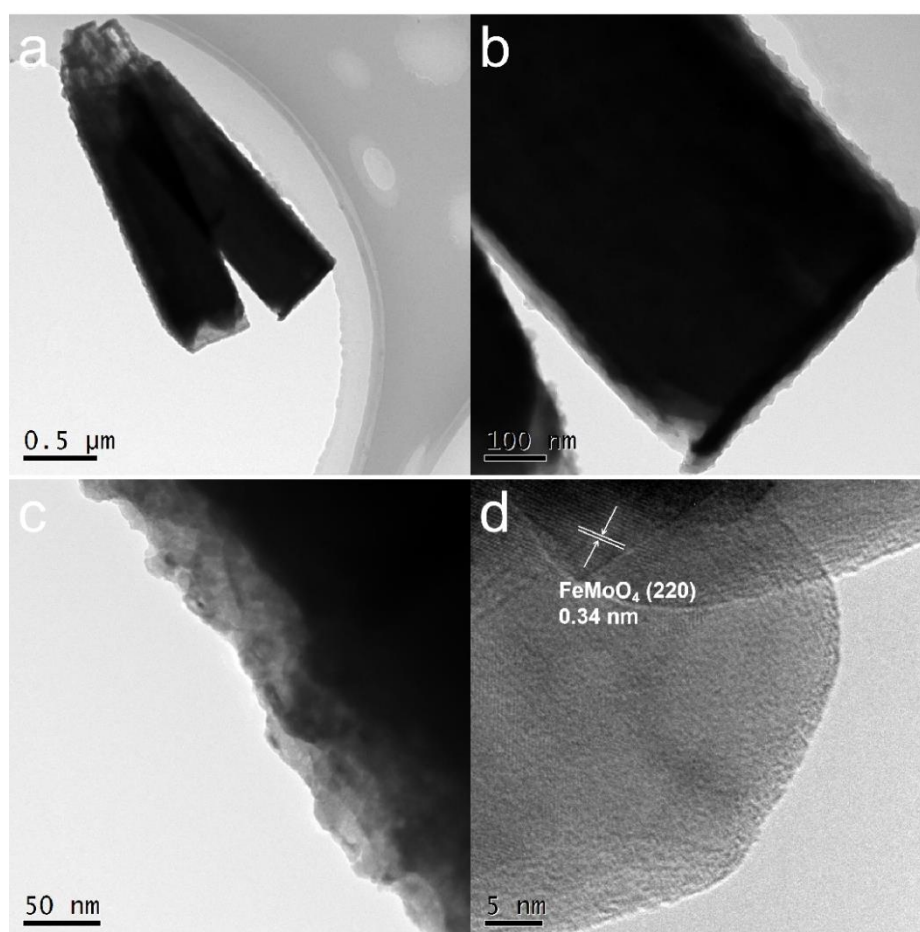


Figure 4. HRTEM images of (a-d) FeMoO₄-1/NF

The electrocatalytic activities of blank NF, FeMoO₄-1, FeMoO₄-2, FeMoO₄-3, FeO_x and MoO_x toward OER are investigated in 1.0 M KOH. LSV curves are first discussed in Figure 5a. Among the three FeMoO₄/NF electrodes of different ratios of Mo to Fe, the FeMoO₄-1 exhibits the highest OER activity with a lowest overpotential of 284 mV to reach 100 mA cm⁻² as compared to 292 and 286 mV achieved by the FeMoO₄-2 and FeMoO₄-3 electrodes, respectively. The results demonstrate the advantage of nanoprism structure. It is worth to note that FeMoO₄-1 is also superior to other reported non-noble-metal electrocatalysts to reach 100 mA cm⁻² in 1.0 M KOH, including FeSe₂/NF (308 mV) [37] and Ni₃S₂/NF (450 mV) [40]. Table 1 gives a more detailed comparison. In addition, Tafel plots in Figure 5b are obtained by Tafel equation ($\eta = b \log j + a$) to evaluate OER kinetics. Again, the FeMoO₄-1 electrode achieves the lowest Tafel plot of 62.66 mV dec⁻¹, with the FeMoO₄-3 electrode (72.36 mV dec⁻¹) coming next, followed by the FeMoO₄-2 electrode (85.87 mV dec⁻¹), indicating that the FeMoO₄-1 electrode has a fast kinetics for OER in 1.0 M KOH. Electrochemical impedance spectroscopy (EIS) was used to evaluate the charge transfer resistance of the sample electrode during OER process. As displayed in Figure 5c, the resulting Nyquist plots indicate that the FeMoO₄-1 electrode with the smallest semicircle has the smallest charge transfer resistance than FeMoO₄-2, FeMoO₄-3, FeO_x and MoO_x. Furthermore, electrochemically active surface area (ECSA) is another important factor influencing the catalytic performance. The double layer capacitances (C_{dl}) are applied to estimate ECSAs of catalysts using CVs at 40, 60, 80, 100 and 120 mV s⁻¹. As displayed in Figure 5d, FeMoO₄-1/NF possesses the largest C_{dl} value (12.05 mF cm⁻²), consistent with the conclusion drawn from the overpotential, Tafel slope and Nyquist plots.

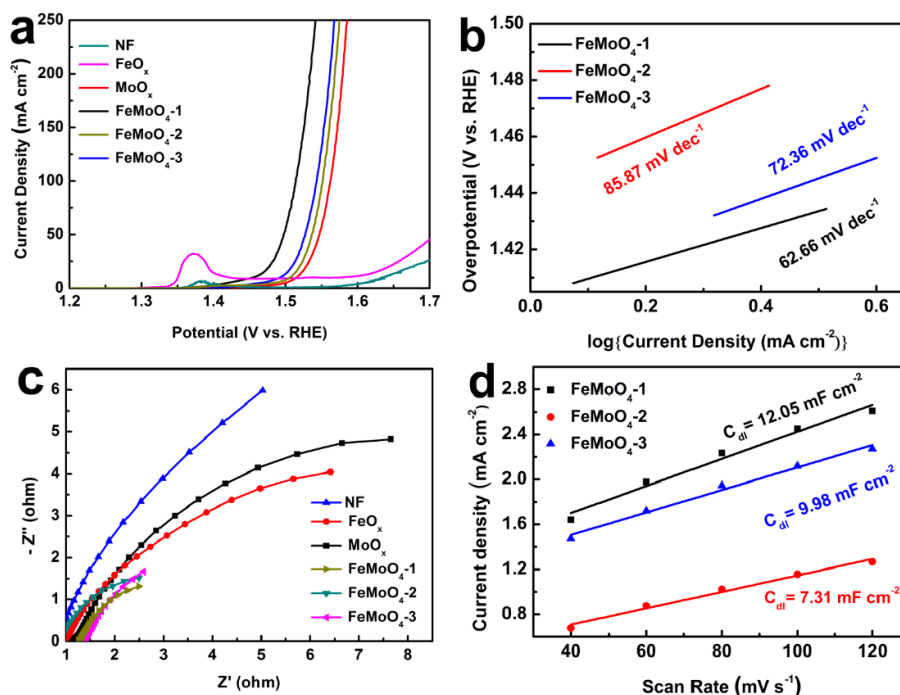


Figure 5. (a) LSV curves of blank NF, FeO_x/NF, MoO_x/NF, FeMoO₄-1/NF, FeMoO₄-2/NF and FeMoO₄-3/NF recorded in 1.0 M KOH at a scan rate of 5 mV s⁻¹ and corresponding (b) Tafel plots and (c) Nyquist plots. (d) Plots of current density difference against scan rate for calculation of double layer capacitance (C_{dl}).

Table 1. Comparison of OER performance with other non-noble-metal electrocatalysts in 1.0 M KOH.

Catalyst	J (mA cm ⁻²)	Voltage (mV)	Ref.
FeMoO ₄ -1/NF	100	284	This work
NiCo ₂ O ₄ @CoMoO ₄ /NF-7	20	265	38
FeSe ₂ /NF	100	308	39
FeOOH(Se)/IF	100	364	40
FeB ₂	100	410	41
Ni ₃ S ₂ /NF	100	450	42
CoFe/NF	10	220	43
NiCo ₂ O ₄	10	290	44
CuCo ₂ O ₄ /NrGO	10	360	45
β-NiMoO ₄	10	300	46
CoMoO ₄	10	343	47

4. CONCLUSIONS

In summary, highly efficient electrocatalysts have been developed by in-situ growth of FeMoO₄ nanoprism on nickel foam through a simple one-step hydrothermal method. The electrochemical measurements suggest that the FeMoO₄-1/NF electrode is a promising oxygen evolution reaction catalyst in alkaline solution. Compared with single metal oxides, the FeMoO₄-1/NF with a molar ratio of 1:1 (n_{Mo}:n_{Fe}) demonstrates the enhanced activity for OER with a small overpotential of 284 mV to reach 100 mA cm⁻². Our work provides the promising way for designing bimetallic material as excellent OER electrocatalysts.

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