

Electrocatalytic oxygen evolution by synergistically coupling layered double hydroxide Ni-based with MXene

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Abstract. Electrolysis presents a cost-effective and environmentally friendly approach for producing carbon-neutral hydrogen, addressing global environmental challenges. However, the slow kinetics of the oxygen evolution reaction (OER) pose a significant obstacle in many sustainable energy conversion processes. In this study, a novel two-dimensional (2D) $\text{Ti}_3\text{C}_2\text{T}_x$ MXene substrate was used to hybridize with Mo-doped NiCo phosphate nanostructures through a two-step hydrothermal process followed by phosphating calcination, resulting in a low-cost and efficient electrocatalyst. The porous, defect-rich $\text{Ti}_3\text{C}_2\text{T}_x$ flakes provide metal-like electrical conductivity and a large surface area, supporting the direct growth of active species and improving their catalytic performance for the OER. The synthesized $\text{Ni}_1\text{Co}_1\text{Mo}_{0.125}\text{Px}/\text{MXene}$ exhibited an overpotential of 314.6 mV at 10 mA cm^{-2} and a Tafel slope of 77 mV dec^{-1} , with an electrochemical double-layer capacitance (Cdl) of $7.1 \text{ } \mu\text{F cm}^{-2}$ corresponding to a large electrochemically active surface area (ECSA). Electrochemical impedance spectroscopy (EIS) confirmed a low charge-transfer resistance, while chronoamperometry (CA) stability tests demonstrated robust long-term durability under continuous operation. This unique nanostructure enhances electrocatalytic activity due to the synergistic effects between the metal components, offering improved performance for the oxygen evolution reaction.

Keywords: Oxygen evolution reaction, Water electrolysis, Hydrogen energy, MXene composites, Transition metal phosphides



1. Introduction

The heavy reliance on fossil fuels has led to significant environmental issues, underscoring the need for net-zero emissions via renewable energy and sustainable practices. Hydrogen, particularly through alkaline electrolysis, is a promising alternative, with the oxygen evolution reaction (OER) being a crucial yet slow step, requiring noble metal oxides like IrO_2 and RuO_2 [1], [2]. However, these materials are expensive and scarce, hindering commercialization. To address this, researchers are developing cost-effective, efficient OER catalysts using earth-abundant elements such as the transition metal phosphides (TMPs) group of Ni and Co phosphides [3]. TMPs are particularly promising due to their abundance, high activity, stability, and low cost [4], [5].

Recently, researchers have looked into two-dimensional (2D) nanomaterials, like MXene ($\text{Ti}_3\text{C}_2\text{T}_x$), which has shown potential in enhancing OER activity due to its high electrical conductivity, large surface area, and excellent stability [6], [7]. Combining MXene with metal nanoparticles like TMPs can also serve as excellent supports for stabilizing catalysts, which in turn increases the OER by improving the charge transference, electrolyte accessibility, and electrochemical active surface area (ECSA). Implementing MXene with TMPs catalysts appears to have received little attention, especially in the electrocatalytic activity of OER for alkaline water electrolysis [3], [4], [5], [8].

To enhance the electrocatalytic activities, incorporating Mo into NiCo-based catalysts on MXene substrates also offers several advantages. Mo doping can modulate the electronic structure by shifting the d-band center, thereby optimizing the adsorption energies of oxygenated intermediates during OER [9]. Furthermore, Mo enhances structural stability by preventing Ni/Co leaching under alkaline conditions [10]. When coupled with highly conductive MXenes, the synergistic effect of Ni, Co, Mo, and P provides a favorable environment for rapid charge transfer and the generation of abundant active sites [11]. This combination is expected to significantly boost OER performance.

In this work, hierarchical molybdenum-doped-NiCo phosphatized on $\text{Ti}_3\text{C}_2\text{T}_x$ MXene composite is developed by adjusting the ratio of Co and Mo precursor to the Ni precursor using a simple hydrothermal and phosphating route as depicted in Fig. 1. The OER activity was stimulated by the modulation of Mo doped with the molar ratio NiCoP_x electron structure through the interfacial interaction with MXene. The creation of effective and stable multicomponent catalysts can be guided by adjusting the interface interaction between the substrate and active components. The resulting $\text{NiCoMoP}_x/\text{MXene}$ was characterized for its physicochemical and electrochemical properties, highlighting its promising potential as an efficient electrocatalyst for sustainable energy applications, particularly in water electrolysis, hydrogen production, and other renewable energy conversion systems.

2. Experimental

2.1 Synthesis of $\text{Ti}_3\text{C}_2\text{T}_x$ MXene

The Ti_3AlC_2 MAX phase (purity >98%, particle size <40 μm) was purchased from Carbon-Ukraine. The 1 g of MAX phase (Ti_3AlC_2) was mixed with 9 M HCl and 1.62 g of LiF to selectively etch the Al layers and stirred at 35 °C for 24 hours, then washed, centrifuged, and dried overnight in a vacuum oven at 60 °C to obtain the MXene, $\text{Ti}_3\text{C}_2\text{T}_x$.

2.2 Synthesis of layered double hydroxide Ni-based with MXene

10 mg of synthesized $\text{Ti}_3\text{C}_2\text{T}_x$ MXene were mixed with an aqueous solution containing 50 mL of $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$, with a series of regulating molar ratios of Co and Mo precursor with the addition of 100 mM ascorbic acid, 100 mM urea, and 80 mM ammonium fluoride. The mixture was stirred for 2 h, then transferred to a Teflon-lined stainless-steel autoclave and heated at 140 °C for 12 h. After cooling, the product was rinsed with deionized water and dried overnight at 60 °C. The resulting $\text{NiCo}_2\text{O}_4\text{MoO}_4/\text{MXene}$ was placed in an alumina boat along with another boat containing 500 mg NaH_2PO_2 . Both boats were calcined at 300 °C for 1 h under Ar flow, yielding hierarchical $\text{NiCoMoP}/\text{MXene}$. The electrocatalysts designated as $\text{Ni}_1\text{Co}_1\text{Mo}_{0.125}\text{P}/\text{MXene}$, $\text{Ni}_1\text{Co}_{0.75}\text{Mo}_{0.1}\text{P}/\text{MXene}$, $\text{Ni}_1\text{Co}_{0.5}\text{Mo}_{0.1}\text{P}/\text{MXene}$, and $\text{Ni}_1\text{Co}_{0.4}\text{Mo}_{0.1}\text{P}/\text{MXene}$, with a schematic diagram of the process illustrated in Figure 1.

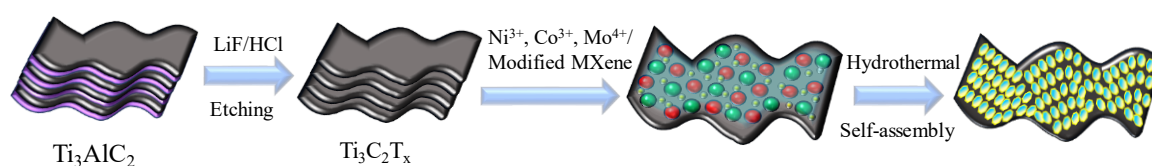


Figure 1. Schematic of the synthesis process of hierarchical $\text{NiCoMoPx}/\text{MXene}$ nanostructure.

2.3 Physicochemical characterization

Scanning electron microscopy (Hitachi SU8010, Japan) and transmission electron microscopy (TEM, FEI Talos L120C) operating at 120 kV were used to examine the surface morphology and nanostructure, respectively. The elemental and particle distribution was observed through mapping analysis using SEM (TESCAN VEGA3, Czech Republic, with AZtec analysis software, Oxford Instruments, UK). X-ray photoelectron spectroscopy (XPS, Kratos AXIS Ultra) was employed to analyse the surface chemical states.

2.4 Electrocatalytic activities of the oxygen evolution reaction (OER)

OER performance was assessed in 1.0 M KOH using a typical three-electrode setup consisting of glassy carbon electrode (GCE) as working electrode, a graphite rod as the counter electrode, and Ag/AgCl (3 M KCl) as the reference electrode. Linear sweep voltammetry (LSV) was measured at 5 mV/s with 85% iR correction. Tafel plots were derived from LSV curves using the equation: $\eta = b \log(j) + \alpha$ (η : overpotential, j : current density, b : Tafel value, α : exchange current density). The electrochemically active surface area (ECSA) was determined from the electrochemical double-layer capacitance (C_{dl}) from a cyclic voltammetry test, calculated by plotting the current density differences ($\Delta j = j_a - j_c$) against various scan rates (5, 25, 50, 75, and 100 mV s^{-1}) in the non-faradaic region, where the slope corresponds to $C_{dl}/2$. ECSA was then estimated as $\text{ECSA} = C_{dl}/C_s$, assuming a specific capacitance (C_s) of 0.04 mF cm^{-2} for a flat electrode in alkaline electrolyte. The turnover frequency (TOF) was calculated by the equation: $\text{TOF} = (J \times A) / (4 \times F \times m)$, where J represents the current density (A cm^{-2}) at an overpotential of 350 mV, A and m represent the area of the electrode and the number of moles of the active materials. All potentials were calibrated using the reversible hydrogen electrode (vs RHE): $E_{\text{RHE}} = E_{\text{Ag}/\text{AgCl}} + 0.059 \text{ pH} + 0.197$. 5 mg of each sample was prepared for catalyst ink in a mixture of 150 μL DI water, 150 μL isopropanol, and 50 μL Nafion solution.

3. Results and discussion

3.1 Physicochemical characterization of layered double hydroxide Ni-based with MXene

The crystal structures of exfoliated $\text{Ti}_3\text{C}_2\text{T}_x$ flakes, NiCoMoP/MXene composites, and the parent MAX phase Ti_3AlC_2 were analyzed by X-ray diffraction (XRD) (Figure 2). The Ti_3AlC_2 MAX phase exhibits characteristic peaks at the (002), (004), (101), (104), (105), (107), (109), and (110) planes (JCPDS No. 52-0875). The absence of several higher-order peaks in the 2θ range of $30\text{--}50^\circ$ and the reduction in intensity indicate the successful conversion of Ti_3AlC_2 into $\text{Ti}_3\text{C}_2\text{T}_x$ MXene via selective Al layer etching with HCl and LiF. The (002) peak of $\text{Ti}_3\text{C}_2\text{T}_x$ shifted from 9.5° to 6.5° is attributed to interlayer expansion induced by Al removal, surface termination groups, and Li^+ intercalation [11], with the shift reflecting increased interlayer spacing [12].

For the NiCoMoP/MXene composite, three distinct peaks at 44.52° , 51.96° , and 65.78° correspond to the (222), (200), and (211) planes of NiCo-LDH/NiCo phosphide (JCPDS No. 38-0715), confirming successful formation of NiCoMoP. The composite exhibits feature from both MXene and NiCo phases: the MXene (002) peak shifts slightly to lower angles, while NiCo-LDH/NiCoP peaks remain distinct, indicating strong interfacial coupling, Mo/P-induced lattice distortion, and intercalation of NiCoMoP nanoparticles between MXene layers. Compared to pristine NiCo-LDH, these modifications demonstrate partial integration of diffraction features, electronic redistribution, and surface reconstruction, which enhance electron transfer, structural stability, and catalytic activity during OER.

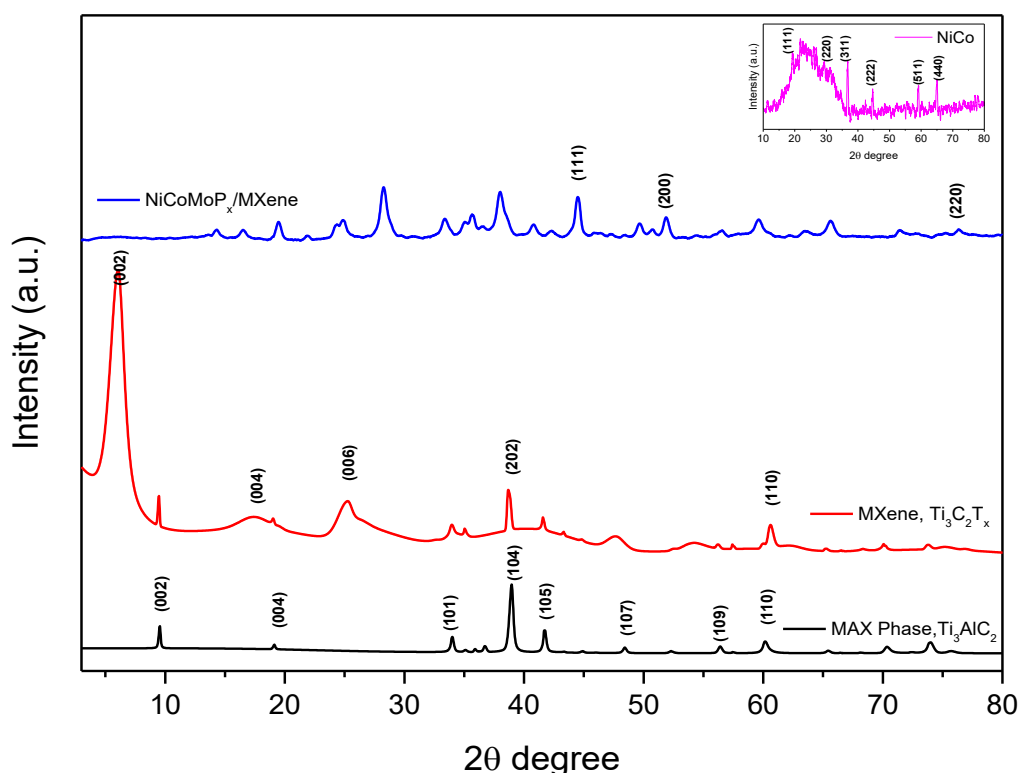


Figure 2. XRD patterns of the isolated $\text{Ti}_3\text{C}_2\text{T}_x$ flakes and NiCoMoP_x/MXene.

The morphology of the NiCoMoP/MXene and MXene was analysed using SEM and HRTEM, as depicted in Figure 3. Typically, the precursor Ti_3AlC_2 MAX phase was selectively etched by

using the HCl/LiF mixture to form isolated $\text{Ti}_3\text{C}_2\text{T}_x$ flakes, as shown in the SEM image in Figure 3a. The stacked layers of morphological features are clearly obtained when Al layers were successfully removed from the Ti_3AlC_2 MAX phase.

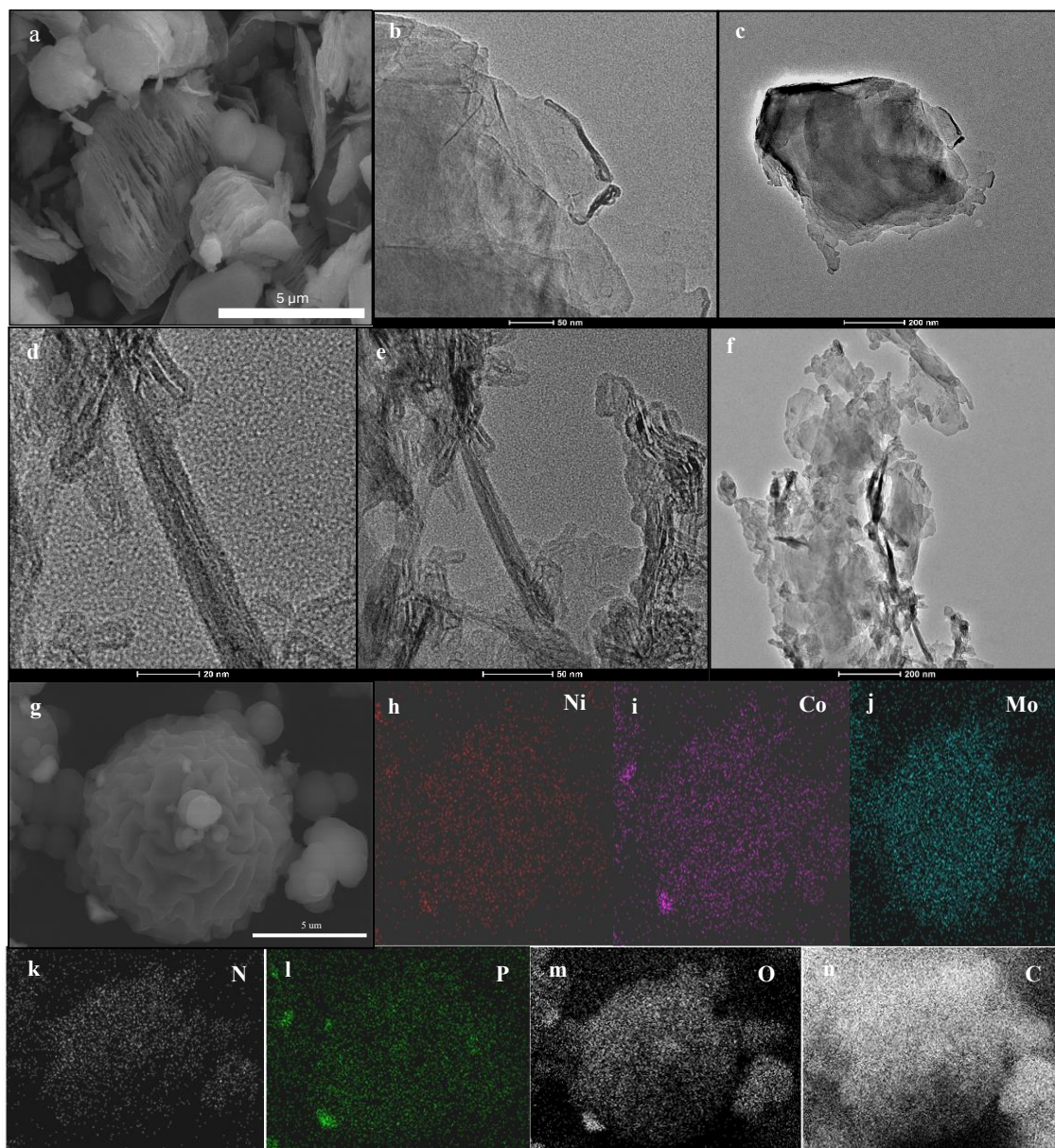


Figure 3. (a) SEM, (b) and (c) HRTEM images of a typical $\text{Ti}_3\text{C}_2\text{T}_x$ flakes structure, (d), (e), and (f) HRTEM image of NiCoMoP, and (g) SEM image of NiCoMoP with their mapping scan of elements (h), (i), (j), (k), (l), (m), and (n).

The high-resolution transmission electron microscopy (HRTEM), as shown in Figure 3b - c, further confirms the ultrathin nature of MXene nanoflakes. Following the in-situ growth of NiCoMoP on MXene, the structure is prone to aggregation and sized cluster production with having flower-like structure as portrayed in Figure 3h [9]. HRTEM images in Figure 3d-f were obtained to close up the morphology of NiCoMoP, which revealed a nanosheet-like structure that

was aggregated to form flower-like self-assembled structures, which is consistent with the SEM image. Furthermore, semi-quantitative mapping area analyses were conducted for a subset of areas, revealing the uniform distribution of Ni, Co, Mo, Ni, P, O, and C elements in NiCoMoP. This confirms the successful doping of the Mo element, and the heteroatom elements of P and N in developing a hybridize structure of NiCoMoP.

3.2 Electrocatalytic activities in oxygen evolution reaction (OER) of Ni-based with MXene

The OER activities of the prepared samples with manipulation of various molar ratios Ni-to-Co-to-Mo were denoted as $\text{Ni}_1\text{Co}_1\text{Mo}_{0.125}\text{P}/\text{MXene}$, $\text{Ni}_1\text{Co}_{0.75}\text{Mo}_{0.1}\text{P}/\text{MXene}$, $\text{Ni}_1\text{Co}_{0.5}\text{Mo}_{0.1}\text{P}/\text{MXene}$, and $\text{Ni}_1\text{Co}_{0.4}\text{Mo}_{0.1}\text{P}/\text{MXene}$ and displayed in Figure 4. The $\text{Ni}_1\text{Co}_1\text{Mo}_{0.125}\text{P}/\text{MXene}$ showed the highest current density produced with the lowest overpotential at η_{10} of about 314.6 mV, followed by 409.13 mV for $\text{Ni}_1\text{Co}_{0.4}\text{Mo}_{0.1}\text{P}/\text{MXene}$, and 419 mV for $\text{Ni}_1\text{Co}_{0.5}\text{Mo}_{0.1}\text{P}/\text{MXene}$. In contrast, the $\text{Ni}_1\text{Co}_{0.75}\text{Mo}_{0.1}\text{P}/\text{MXene}$ did not demonstrate satisfactory performance in OER.

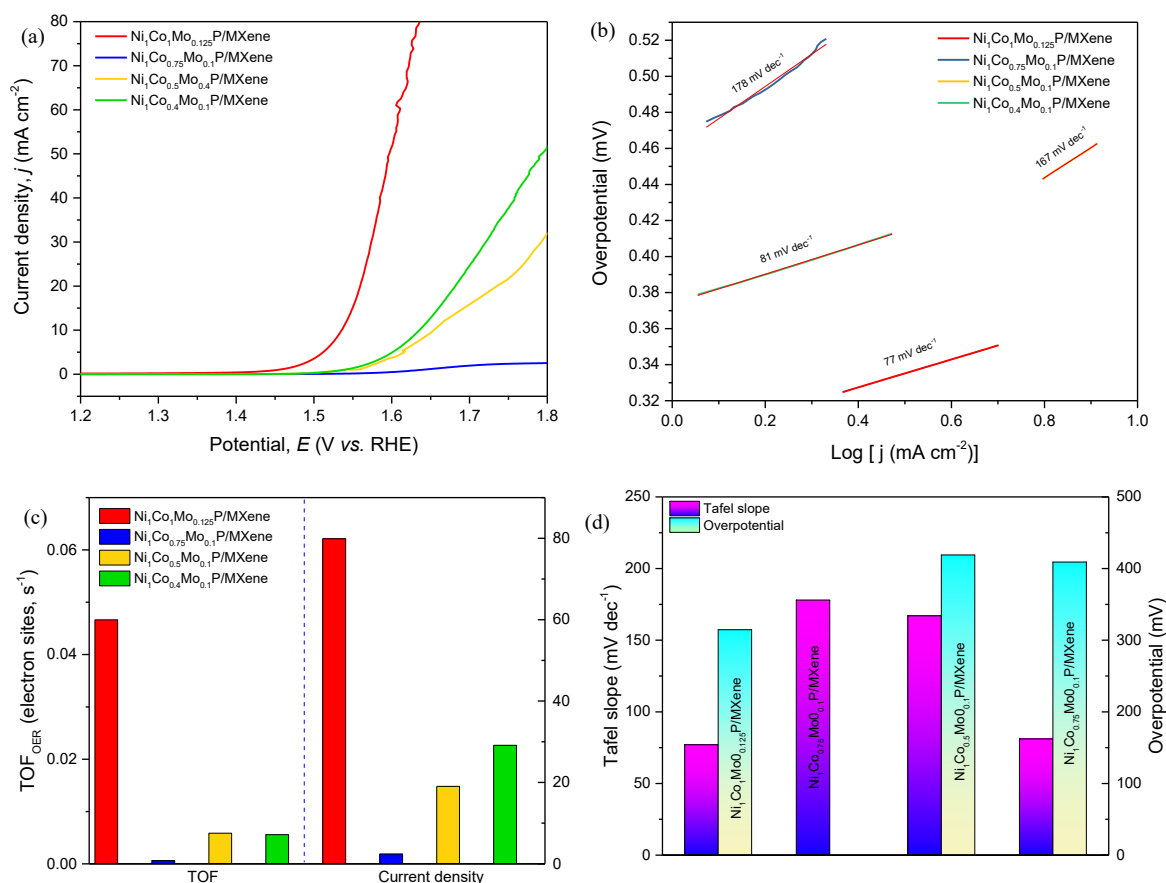


Figure 4. The electrocatalytic activities of OER in 1.0 mol/L KOH for electrocatalyst $\text{Ni}_1\text{Co}_1\text{Mo}_{0.125}\text{P}/\text{MXene}$, $\text{Ni}_1\text{Co}_{0.75}\text{Mo}_{0.1}\text{P}/\text{MXene}$, $\text{Ni}_1\text{Co}_{0.5}\text{Mo}_{0.1}\text{P}/\text{MXene}$, and $\text{Ni}_1\text{Co}_{0.4}\text{Mo}_{0.1}\text{P}/\text{MXene}$ at scan rate 5 mV s⁻¹ (a) LSV curve, (b) Tafel slope, (c) contrast histogram of TOF value and maximum current density, j (mA cm⁻²) produced, (d) comparison of Tafel slope with η_{10}

Additionally, the OER kinetics was assessed by fitting the Tafel plots of the electrocatalysts using the Tafel equation, $\eta = \alpha + b \log j$. Here, η (V) represents the overpotential, α (V) is the intercept, b (mV dec⁻¹) is the Tafel slope, and j (mA cm⁻²) is the current density. The $\text{Ni}_1\text{Co}_1\text{Mo}_{0.125}\text{P}/\text{MXene}$, $\text{Ni}_1\text{Co}_{0.75}\text{Mo}_{0.1}\text{P}/\text{MXene}$, $\text{Ni}_1\text{Co}_{0.5}\text{Mo}_{0.1}\text{P}/\text{MXene}$, and

Ni₁Co_{0.4}Mo_{0.1}P/MXene had Tafel slope values of 77, 178, 167, and 81 (mV dec⁻¹), respectively. The Ni₁Co₁Mo_{0.125}P/MXene showed the fastest response rate with the lowest Tafel slope value, a low exchange current density, α (1.497×10^{-4} mA cm⁻²), and a high turnover frequency (TOF) of 0.04664 s⁻¹ at $\eta = 400$ mV. The higher TOF of Ni₁Co₁Mo_{0.125}P/MXene comes from the combined effects of its molecule components. The presence of Mo changes the electronic environment, lowering the adsorption energy barrier of OER intermediates. Meanwhile, Ni and Co provide many redox active sites. Phosphorus aids rapid charge transfer stabilizes the local coordination environment, and improves electron conductivity. These effects result in higher turnover frequencies compared to undoped versions. The combined contributions of the elements in Ni₁Co₁Mo_{0.125}P/MXene lead to its increased TOF. Ni and Co offer plentiful redox-active sites, while Mo helps tune the electronic environment, reducing the adsorption energy barrier of OER intermediates. Phosphorus boosts electron conductivity and stabilizes the local coordination environment, which supports rapid charge transfer. These factors yield higher turnover frequencies than undoped counterparts. This shows that the open structure around the active site NiCoMo allows for efficient mass transport and rapid charge transfer across these active sites during diffusion, including both inward and outward diffusion of oxygen and OH⁻. These findings highlight that the combination of Ni, Co, Mo, and P in the MXene is essential in determining its electrocatalytic activity for the oxygen evolution reaction.

The OER kinetics is further supported by the electrochemical double-layer capacitance C_{dl} analysis as measured in Figure 5a. The Ni₁Co₁Mo_{0.125}P/MXene exhibited the largest slope among the tested samples, with the C_{dl} value being the highest at 18.2 μ F cm⁻², indicating it has a larger electrochemical surface area, which promotes OER activity, with an ECSA value is 0.728 cm². Electrochemical impedance spectroscopy (EIS) (in Figure 5b) further demonstrated that Ni₁Co₁Mo_{0.125}P/MXene possessed the smallest semicircle diameter, confirming its lowest charge-transfer resistance and superior interfacial electron transport kinetics [14]. The chronoamperometric stability test of the highest-performance Ni₁Co₁Mo_{0.125}P/MXene, as shown in Figure 5c, revealed only a slight current decay over extended operation, highlighting the excellent durability of the catalyst under continuous OER conditions. The kinetic parameters for all electrocatalysts in OER activities are presented in Table 1.

Table 1. Electrochemical parameters of prepared electrocatalysts NiCoMoP/MXene towards OER.

Electrocatalyst	η_{10} (mV)	Tafel slope (mV dec ⁻¹)	Exchange current density (mA cm ⁻²), α	Turnover frequency (TOF, s ⁻¹)	C_{dl} (μ F cm ⁻²)	ECSA (cm ²)
Ni ₁ Co ₁ Mo _{0.125} P/MXene	314.6	77	1.497×10^{-4}	0.04664	18.2	0.728
Ni ₁ Co _{0.75} Mo _{0.1} P/MXene	0	178	2.735×10^{-3}	6.317×10^{-4}	1.3	0.052
Ni ₁ Co _{0.5} Mo _{0.1} P/MXene	419.0	167	0.0140	5.8536×10^{-3}	5.2	0.208
Ni ₁ Co _{0.4} Mo _{0.1} P/MXene	409.13	81	2.504×10^{-5}	5.5763×10^{-3}	7.5	0.3

The benchmarking analysis in Figure 5d showed that the OER performance of NiCoMoP/MXene is similar to other electrocatalysts like CoS [15], CoP/N-CNT [16], Ni_{2.5}Fe_{2.5}-P/Ti₃C₂T_x [17], FeCoB₂ [18], Co₃O₄/MXene [19], Ni-MOF@CNT [20], Na₂Co_{0.75}Fe_{0.25}P₂O₇C NPs [21], and Ni-Fe-S/D-Ti₃C₂ [22]. This confirms that hierarchical Mo-doping and MXene coupling

effectively improve catalytic activity and stability. The initial study of the NiCoMoP/MXene electrocatalyst showed promising activity for the OER, suggesting it could be a strong candidate for further optimization. Additionally, testing different operating conditions might also enhance catalytic activity.

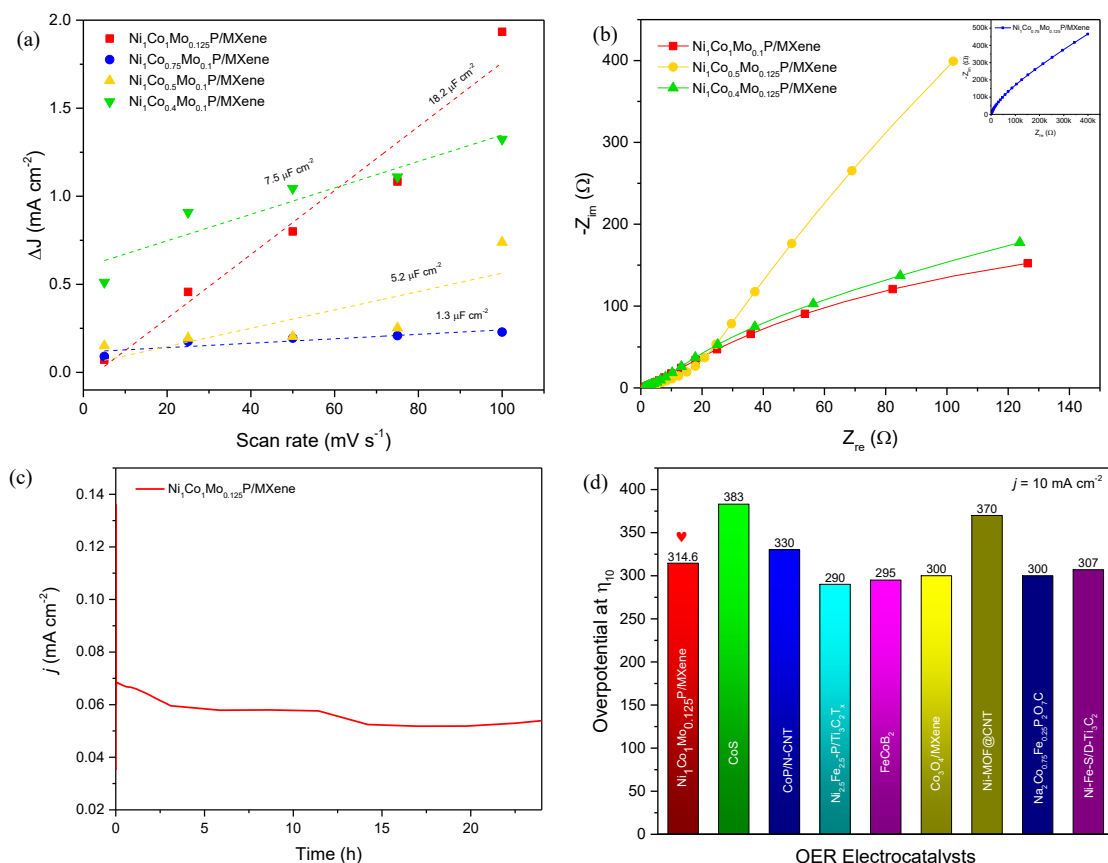


Figure 5. Electrochemical characterization of the NiCoMoP/MXene composite: (a) electrochemical double-layer capacitance (C_{dl}) from different scan rate, (b) electrochemical impedance spectroscopy (EIS), (c) chronoamperometry (CA) tests the stability of the electrocatalyst at a constant overpotential, and (d) comparison of the overpotential of the NiCoMoP/MXene with previously reported OER electrocatalyst performance.

Therefore, it is suggested to demonstrate the NiCoMoP/MXene functions in the OER mechanism reaction as illustrated in Figure 6. The oxygen evolution reaction (OER) over the Ni₁Co₁Mo_{0.125}P/MXene composite proceeds via a four-step mechanism. A highly conductive platform made possible by the multilayer MXene substrate speeds up electron transport to the active regions. Ni and Co atoms anchored on the MXene surface act as the primary catalytic centers, where hydroxide ions (OH⁻) from the alkaline electrolyte are initially adsorbed. The nearby Mo atoms modulate the electronic structure of the Ni/Co sites, promoting charge redistribution and enhancing the adsorption strength of intermediates. Phosphorus incorporation further improves electronic conductivity and stabilizes the active sites. During the OER process, adsorbed OH⁻ species undergo stepwise deprotonation and O-O bond formation to generate the OOH* intermediate. This intermediate then evolves into O₂ and is released into the

solution. MXene, Ni/Co, Mo, and P interact synergistically to enhance the kinetics of each elementary step, enhancing the overall catalytic activity and stability of the composite.

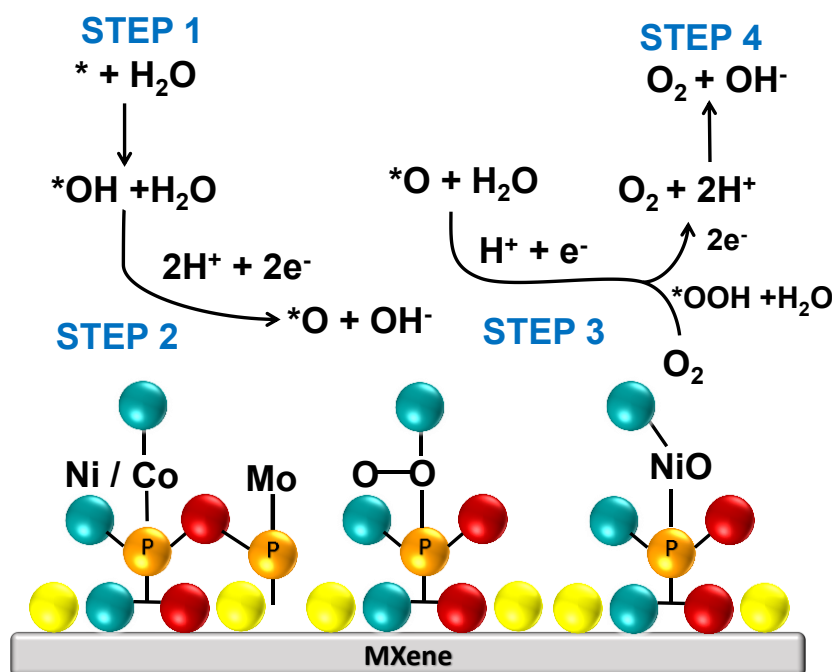


Figure 6. The schematic for NiCoMoP/MXene in the oxygen evolution reaction (OER) mechanism.

4. Conclusion

This study investigates the development of a hierarchical NiCoMoP/MXene nanohybrid electrocatalyst. It combines Mo-doped NiCo-based phosphides with the highly conductive and chemically active 2D $\text{Ti}_3\text{C}_2\text{T}_x$ MXene. The resulting flower-like nanostructures, averaging 200 to 300 nm, create plenty of electroactive sites and efficient electron transport pathways. Among the samples, the $\text{Ni}_1\text{Co}_1\text{Mo}_{0.125}\text{P}/\text{MXene}$ showed good OER activity, achieving an overpotential of 314.6 mV to reach 10 mA cm^{-2} in the alkaline medium. It has a low Tafel slope of 77 mV dec^{-1} , a low exchange current density, α ($1.497 \times 10^{-4} \text{ mA cm}^{-2}$), and a high turnover frequency (TOF) of 0.04664 s^{-1} at $\eta = 400 \text{ mV}$. These factors highlight its effective catalytic performance. The catalyst also has high double-layer capacitance (C_{dl}), which means it has a large electrochemically active surface area (ECSA) and a greater density of exposed active sites. Electrochemical impedance spectroscopy (EIS) showed that it has the lowest charge-transfer resistance among the tested samples, indicating fast charge transport and improved interfacial kinetics. Additionally, chronoamperometry (CA) demonstrated excellent stability with minimal performance decline, showing it maintains high structural integrity during extended OER operations. Overall, these results emphasize the advantages of incorporating Mo and using MXene support, creating a strong platform for developing non-noble electrocatalysts in water electrolysis and future energy conversion and storage technologies.

Acknowledgments

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