

Cite this: *RSC Adv.*, 2026, 16, 46590

Delaminated Mo_2CT_x MXene-coupled NiCo-LDH nanosheets as efficient non-precious metal anodes for anion exchange membrane water electrolysis

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Hydroxide-based layered double hydroxides (LDHs) are promising oxygen evolution reaction (OER) electrocatalysts for alkaline water electrolysis; however, their practical application is often limited by poor electrical conductivity and sluggish charge-transfer kinetics. In this work, a hybrid NiCo-LDH@ Mo_2CT_x /NF electrode was developed by integrating conductive, delaminated Mo_2CT_x MXene with NiCo-LDH nanosheets that were directly grown on nickel foam via a hydrothermal process. The delamination treatment effectively increased the interlayer spacing in Mo_2CT_x and provided a conductive platform for the uniform growth of NiCo-LDH, thereby enhancing interfacial contact and electron transport. Structural and surface analyses confirmed the successful formation of the NiCo-LDH@ Mo_2CT_x hybrid architecture with intimate coupling between the LDH and MXene components. Benefiting from the conductive MXene framework and optimized electrode architecture, the NiCo-LDH@ Mo_2CT_x /NF electrode exhibited enhanced OER performance in 1.0 M KOH, requiring overpotentials of 324 and 393 mV to achieve current densities of 50 and 100 mA cm^{-2} , respectively. Furthermore, the hybrid electrode displayed faster reaction kinetics, lower charge-transfer resistance, higher intrinsic activity, and excellent durability, maintaining stable operation for 50 h at 50 mA cm^{-2} . The practical applicability of the catalyst was further validated in an anion exchange membrane water electrolysis (AEMWE) single-cell system. The NiCo-LDH@ Mo_2CT_x /NF anode delivered a current density of 1.15 A cm^{-2} at 2.0 V and maintained stable operation for 100 h at 500 mA cm^{-2} . These results demonstrate that integrating conductive Mo_2CT_x MXene with NiCo-LDH is an effective strategy for improving both catalytic activity and operational stability, offering a promising route toward the development of efficient non-precious-metal anodes for alkaline water electrolysis.

Received 16th June 2026
Accepted 5th August 2026

DOI: 10.1039/d6ra05281a

rsc.li/rsc-advances

1 Introduction

As the demand for carbon-neutral energy systems increases, hydrogen is emerging as a key clean energy carrier,^{1,2} and renewable energy-based water electrolysis is attracting attention as a promising technology capable of sustainably producing high-purity hydrogen.^{3–5} Among various water electrolysis technologies, anion exchange membrane water electrolysis (AEMWE) has been evaluated as a next-generation system capable of simultaneously securing the economic feasibility of alkaline water electrolysis⁶ and the high-performance characteristics of proton exchange membrane water electrolysis.^{7,8} In AEMWE, OH^{+} ions move through the polymer membrane

under alkaline conditions, making it possible to utilize non-precious catalysts.^{9,10} However, despite these advantages, the oxygen evolution reaction (OER) at the anode acts as a major bottleneck for the entire system due to the slow 4-electron reaction, complex reaction intermediates, and high over-potential requirement.^{11,12} Moreover, in the high current density region required under actual driving conditions, charge-transfer resistance, bubble accumulation, and interface deterioration¹³ occur at the same time, further reducing the efficiency and durability of the system.^{14–17}

To overcome these limitations, the development of non-precious metal OER catalysts based on abundant transition metals such as Ni, Co, and Fe has been actively carried out.^{18,19} Among them, layered double hydroxide (LDH) has been widely studied due to its abundant active sites, large specific surface area,²⁰ and flexible controllability of metal composition and electronic structure²¹ based on a two-dimensional layered structure.²¹ In particular, NiCo-LDH has been reported to exhibit superior catalytic activity and structural stability compared to single metal hydroxides due to the synergistic

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effect between Ni and Co.^{22,23} However, LDH-based catalysts essentially have low electrical conductivity and high interfacial charge transfer resistance,^{24,25} and these limitations are more prominent under high current driving conditions.²⁶ Therefore, hybridization with conductive support is attracting attention as a strategy to improve electron transfer and stably maintain catalyst performance.^{27–29} However, existing carbon-based materials may have limitations in terms of LDH growth and structural stability^{30,31} due to low hydrophilicity and limited precursor interaction.³²

MXene has been widely studied as a conductive support for electrochemical catalysts due to its two-dimensional structure, high electrical conductivity, large surface area, adjustable interlayer spacing, and abundant surface termination.^{33–35} These features improve electrolyte accessibility, facilitate ion/electron transport, and enable interfacial electron modulation.^{36–38} In LDH-based catalysts, the negatively charged MXene surface strongly interacts with the positively charged LDH layer, which can promote uniform LDH nanosheet growth and intimate interfacial contact.^{39,40} These interfacial bonds inhibit LDH aggregation, accelerate charge-transfer kinetics, and improve structural stability under alkaline OER conditions.^{41,42} However, most MXene-assisted OER studies have focused on $\text{Ti}_3\text{C}_2\text{T}_x$ MXene,^{43,44} with Mo-based MXene, especially Mo_2CT_x , remaining relatively less explored in LDH-based OER systems. Among the MXene family, $\text{Ti}_3\text{C}_2\text{T}_x$ has been the most extensively investigated conductive support for LDH-based OER electrocatalysts because of its excellent electrical conductivity, hydrophilic surface terminations, and strong interfacial interaction with hydroxide nanosheets, which effectively suppress catalyst aggregation and facilitate charge transport. However, in most $\text{Ti}_3\text{C}_2\text{T}_x$ /LDH hybrid systems, $\text{Ti}_3\text{C}_2\text{T}_x$ primarily serves as a conductive scaffold, while opportunities for further electronic modulation of the active sites remain relatively limited.⁴⁵ In contrast, Mo_2CT_x possesses a distinctive Mo-centered carbide framework together with abundant surface functional groups, providing additional opportunities for interfacial charge redistribution and modulation of the adsorption behavior of oxygenated reaction intermediates.^{46,47} Furthermore, the electronic structure of Mo_2CT_x can be effectively tailored through delamination and surface engineering, leading to enlarged interlayer spacing, increased exposure of accessible active interfaces, and improved interaction with transition-metal hydroxides.^{48–51}

Despite these attractive characteristics, Mo_2CT_x has received considerably less attention than $\text{Ti}_3\text{C}_2\text{T}_x$ in LDH-based OER electrocatalysis, and systematic investigations into its role in regulating interfacial charge transfer and catalytic kinetics remain scarce. More importantly, most previously reported Mo_2CT_x -based OER studies have primarily focused on morphology optimization or simple hybridization strategies, whereas the influence of residual Ga removal and delamination-induced interlayer expansion on the construction of strongly coupled LDH/MXene heterostructures has rarely been explored.^{52,53} Therefore, developing a delaminated Mo_2CT_x platform with expanded interlayer spacing and intimate interfacial coupling provides a rational strategy to simultaneously

enhance electron transport, promote charge redistribution at the heterointerface, and improve catalytic performance under practical high-current-density AEM water electrolysis conditions.

In this study, pristine Mo_2CT_x was used as the starting material to prepare structurally and electrochemically enhanced delaminated Mo_2CT_x (d- Mo_2CT_x) by removing residual Ga. The selective A-layer delamination process effectively removed residual Ga from Mo_2CT_x , resulting in expanded interlayer spacing and enhanced electrochemical properties. The obtained d- Mo_2CT_x served as a conductive platform for the uniform growth of NiCo-LDH by providing enlarged interlayer spacing, increased effective surface area, and improved charge-transfer characteristics.³³ Although NiCo-LDH is a promising OER catalyst with high activity and tunable metal composition, its performance can be limited under high-current-density operation because of its relatively low electrical conductivity and restricted charge-transfer kinetics. To overcome these limitations, highly conductive d- Mo_2CT_x MXene was integrated with NiCo-LDH to improve electron transport within the electrode and reduce interfacial charge-transfer resistance. Subsequently, NiCo-LDH@ Mo_2CT_x was directly grown on nickel foam (NF) *via* a hydrothermal process, forming a binder-free electrode with enhanced charge-transfer kinetics and structural stability for efficient OER operation. In addition, the interfacial interaction between Mo_2CT_x and NiCo-LDH can contribute to the improvement of the OER kinetics by controlling the electronic structure centered on the active metal and inducing charge redistribution. As a result, the prepared NiCo-LDH@ Mo_2CT_x /NF electrode exhibited excellent OER activity and durability not only in the three-electrode evaluation at 1.0 M KOH but also in the driving conditions of an AEM electrolysis unit cell, and proved its potential as a high-performance non-precious metal anode for AEM water electrolysis.

2 Experimental

2.1 Chemicals and materials

Mo_2CT_x (Xinxitechnology Co., Ltd, China), Nickel(II) nitrate hexahydrate ($\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$), Cobalt(II) nitrate hexahydrate ($\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$) and cetyltrimethylammonium bromide (pre), ethanol, methanol, hydrochloric acid (HCl), Tetramethylammonium hydroxide (TMAOH) and Nickel foam (NF) (thickness: 1.6 mm, bulk density: 0.42 g cm^{-3} , Alantum). All chemicals were used as received, without further purification.

2.2 Mo_2CT_x delamination process

The fabrication process of the electrode is schematically illustrated in Fig. 1. Pre-treated Mo_2CT_x (pre- Mo_2CT_x) was prepared through an additional hydrothermal etching treatment based on a previously reported procedure.⁵⁴ First, 21 mL of 6 M HCl was mixed with 19 mL of deionized (DI) water under magnetic stirring, and 7.26 g of CTAB was then added to prepare the acidic etching solution. Subsequently, 0.2 g of pristine Mo_2CT_x powder was introduced into the solution, followed by magnetic stirring and sonication for 1 h to ensure uniform dispersion.

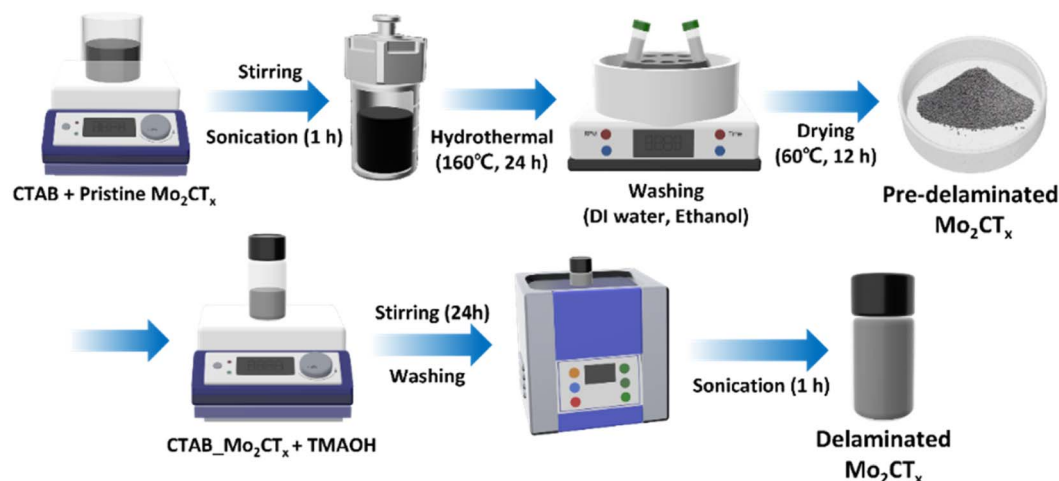
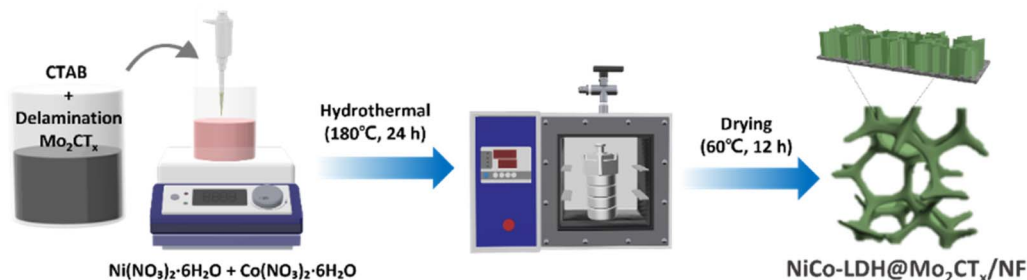
Step 1 : Delaminated Mo_2CT_x **Step 2 : NiCo-LDH@ Mo_2CT_x /NF Synthesis**

Fig. 1 Synthesis procedure for d- Mo_2CT_x and NiCo-LDH@ Mo_2CT_x /NF.

The resulting mixture was transferred into a Teflon-lined stainless-steel autoclave and heated at 160°C for 24 h. After the reaction, the product was collected by centrifugation and washed several times with DI water and ethanol until neutral pH was reached. The obtained precipitate was then dried in a vacuum oven at 60°C for 12 h, yielding pre- Mo_2CT_x powder.

Subsequently, delamination was carried out using tetramethylammonium hydroxide (TMAOH). 30 mg of pre- Mo_2CT_x was dispersed in 5 mL aqueous TMAOH solution (1 : 3) containing 3.33 mmol of TMAOH, followed by continuous magnetic stirring for 24 h to facilitate intercalation and delamination. Then the suspension was centrifuged and washed several times with ethanol and deionized (DI) water, and finally delaminated Mo_2CT_x (d- Mo_2CT_x) was obtained.

2.3 Synthesis of NiCo-LDH@ Mo_2CT_x /NF

Before synthesis, nickel foam (NF) was sequentially cleaned in 3 M HCl, ethanol, and DI water under ultrasonication for 10 min each, followed by drying at 60°C . For hydrothermal growth, 0.90 mmol of $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ and 0.60 mmol of $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ were completely dissolved in 30 mL of methanol under magnetic stirring. Separately, 0.55 mmol of CTAB was added to 6 mL of d- Mo_2CT_x dispersion under stirring to prepare a homogeneous solution. While vigorously stirring the Ni/Co precursor solution, the CTAB-containing d- Mo_2CT_x dispersion

was slowly added dropwise. The cleaned NF was immersed in the mixed precursor solution, followed by sonication, and then transferred into a Teflon-lined stainless-steel autoclave. The hydrothermal reaction was carried out at 180°C for 24 h. After naturally cooling to room temperature, the obtained electrode was washed with DI water and dried at 60°C .

For comparison, NiCo-LDH/NF was synthesized under identical hydrothermal conditions, except that 6 mL of d- Mo_2CT_x dispersion was replaced with 6 mL of DI water. The d- Mo_2CT_x amount was maintained constant throughout this study to enable direct comparison between NiCo-LDH@ Mo_2CT_x /NF and the corresponding NiCo-LDH/NF control prepared under otherwise identical conditions.

2.4 Physical characterizations

The crystallographic phases of the synthesized materials were identified by X-ray diffraction (XRD) using a Rigaku Ultima IV diffractometer (Cu $K\alpha$ radiation, D/MAX-2500). The surface morphology and microstructural features were examined by field-emission scanning electron microscopy (FESEM, TESCAN MIRA3 LM) and field-emission transmission electron microscopy (FETEM, JEOL JSM-6701F). High-resolution imaging was employed to further elucidate the nanoscale structural characteristics of the samples. The surface elemental composition and oxidation states were analyzed by X-ray photoelectron

spectroscopy (XPS) using a Thermo Fisher Scientific NEXSA G2 system. Fourier-transform infrared (FT-IR) spectroscopy was conducted using a PerkinElmer Spectrum Two spectrometer to analyze the chemical bonding characteristics and functional groups of the samples.

2.5 Electrochemical characterizations

All electrochemical characterizations were carried out using a ZIVE SP1 electrochemical workstation in a conventional three-electrode configuration at room temperature with 1.0 M KOH as the electrolyte. The as-prepared NiCo-LDH@ Mo₂CT_x/NF (1 × 1 cm²) was directly used as the working electrode without binders, while a platinum wire and an Ag/AgCl (3.0 M KCl) electrode served as the counter and reference electrodes, respectively. All potentials were converted to the reversible hydrogen electrode (RHE) scale. Linear sweep voltammetry (LSV) was conducted at a scan rate of 1 mV s⁻¹ to evaluate OER activity, and the corresponding Tafel slopes were derived from the polarization curves to assess reaction kinetics. Electrochemical impedance spectroscopy (EIS) was performed over a frequency range of 100 kHz to 0.1 Hz with an AC amplitude of 10 mV to investigate charge-transfer resistance (*R*_{ct}). The electrochemically active surface area (ECSA) was estimated from the double-layer capacitance (*C*_{dl}) obtained *via* cyclic voltammetry in a non-faradaic region at various scan rates. The TOF was calculated from the OER current and the number of electrochemically accessible active sites estimated by integrating the Ni/Co redox peak in the CV curve. A one-electron redox process was assumed for the active-site estimation, while a four-electron transfer process was used for the OER. The calculated values are therefore reported as apparent TOF values. Detailed equations and assumptions are provided in the SI. The long-term durability was examined by chronopotentiometry at a constant current density under alkaline conditions. The AEM single-cell performance was evaluated at 60 °C using 1 M KOH supplied at a flow rate of 15 mL min⁻¹ under dry cathode conditions. The electrodes were fabricated on a 3 cm² nickel foam substrate. A

PiperION membrane (40 μm) was employed as the anion exchange membrane, and Pt/C supported on carbon cloth was used as the cathode. Additional experimental details are provided in the SI.

3 Results and discussion

The structural evolution of Mo₂CT_x during selective Ga removal and subsequent delamination is illustrated in Fig. 2a. Although the initial Mo₂CT_x had already undergone removal of the Ga-containing A-layer, EDS analysis (Table S1) confirmed that a considerable amount of residual Ga still remained. In addition, FE-SEM observations revealed that the sample retained a densely stacked multilayer morphology (Fig. 2c), which is less favorable for the growth of NiCo-LDH because it limits the exposed surface area required for uniform nanosheet formation and intimate interfacial contact.⁵⁵ Therefore, an additional hydrothermal treatment was carried out to further remove residual Ga and induce interlayer separation, yielding d-Mo₂CT_x.

As shown in Fig. 2b, both pristine Mo₂CT_x and d-Mo₂CT_x exhibit the characteristic of MXene phase, and also the MXene framework of d-Mo₂CT_x was preserved after delamination process. Notably, the (002) peak shifted from 8.4° to 6.7° toward a lower diffraction angle, indicating an increase in interlayer spacing after the delamination process.⁵⁶ Since the (002) reflection in layered materials reflects the average distance between adjacent layers,⁵² this low-angle shift suggests weakened interlayer stacking and the formation of a more expanded layered structure, which is consistent with effective delamination.

FE-SEM images further support this structural evolution as shown in (Fig. 2c–e). Pristine Mo₂CT_x exhibited a compact multilayer platelet-like morphology, whereas pre-treated Mo₂CT_x and d-Mo₂CT_x showed progressively thinner, more open, and sheet-like structures, indicating gradual layer separation (Fig. 2c–e). The apparent differences in the lateral

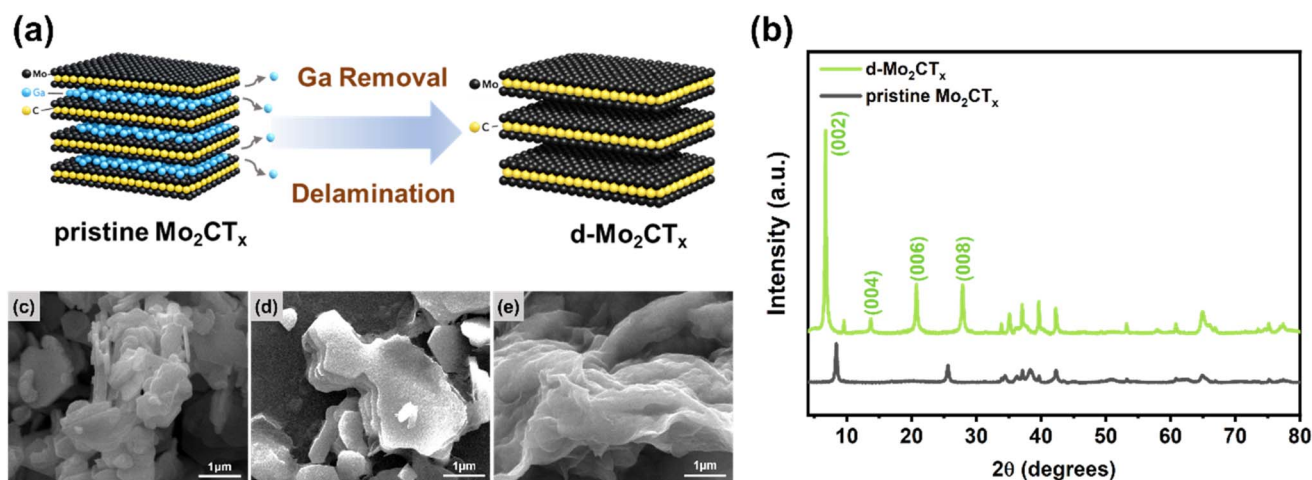


Fig. 2 (a) Schematic illustration of the synthesis process of d-Mo₂CT_x, (b) powder XRD patterns of pristine Mo₂CT_x and d-Mo₂CT_x, (c–e) FE-SEM images of pristine Mo₂CT_x, pre-Mo₂CT_x, and d-Mo₂CT_x, respectively, revealing their typical layered flake-like structure.

dimensions of the MXene flakes mainly originate from the progressive structural evolution during hydrothermal treatment and TMAOH-assisted delamination. The removal of residual Ga and weakening of interlayer interactions facilitate exfoliation into thinner, more separated sheets, while the SEM images represent typical regions of each sample rather than a statistical comparison of flake size. This morphological evolution is in good agreement with the compositional analysis, which revealed that the Ga content markedly decreased from 23.51 wt% (12.68 at%) in pristine Mo_2CT_x to 4.02 wt% (3.62 at%) in d- Mo_2CT_x . Taken together, these results demonstrate that the hydrothermal treatment and subsequent delamination process effectively removed residual Ga while preserving the Mo_2CT_x framework and promoting interlayer expansion and exfoliation.

The hydrothermal etching and TMAOH-assisted delamination parameters employed in this study were selected to obtain Mo_2CT_x with reduced residual Ga content, expanded interlayer spacing, and a more open sheet-like morphology. These conditions are therefore regarded as effective synthesis conditions for constructing the present $\text{NiCo-LDH@Mo}_2\text{CT}_x$ electrode rather than as globally optimized parameters. A systematic investigation of the effects of etching temperature and duration, TMAOH concentration and intercalation time, and Mo_2CT_x loading on the conductivity, morphology, and OER activity will be valuable for further optimization of the composite architecture.

XRD analysis (Fig. 3a) was conducted to compare the crystal structures of NiCo-LDH/NF and $\text{NiCo-LDH@Mo}_2\text{CT}_x/\text{NF}$. Both electrodes showed characteristic diffraction peaks at approximately 11.4° , 22.8° , 34.5° , 39.1° , 46.8° , and 60.5° , corresponding to the (003), (006), (012), (015), (018), and (110) planes of the LDH phase, respectively.³⁷ These peaks are consistent with the standard LDH pattern (PDF#40-0216), confirming the successful formation of NiCo-LDH . Notably, the main LDH peaks of $\text{NiCo-LDH@Mo}_2\text{CT}_x/\text{NF}$ were retained without obvious peak shifts, indicating that the introduction of d- Mo_2CT_x did not significantly alter the LDH crystal structure. In addition, a weak low-angle peak at 6.24° , assigned to the (002) plane of Mo_2CT_x ,

was observed in $\text{NiCo-LDH@Mo}_2\text{CT}_x/\text{NF}$, confirming the incorporation of Mo_2CT_x into the electrode.

The low intensity of this peak can be understood from the delaminated nature and relatively low amount of Mo_2CT_x in the hybrid structure. In addition, a peak feature related to the Mo_2CT_x (100) plane was observed near the region of the LDH (012) peak, further supporting that LDH and Mo_2CT_x . These XRD results show that the composite keeps the original LDH crystal framework while also containing the MXene phase, which is important for building a stable electrode.

FT-IR analysis (Fig. 3b) was performed to examine the bonding features of NiCo-LDH/NF and $\text{NiCo-LDH@Mo}_2\text{CT}_x/\text{NF}$. Both samples showed a broad band centered at about 3448 cm^{-1} , which is related to the stretching vibration of surface $-\text{OH}$ groups and interlayer water molecules. This broad band confirms that both catalysts keep the hydroxyl-rich nature that is typical of LDH structures. The band at 1633 cm^{-1} is assigned to the bending vibration of H-O-H from interlayer or adsorbed water, while the peak at 1381 cm^{-1} is attributed to nitrate ions located in the LDH interlayer space.⁵⁷ These features indicate that the basic layered LDH structure was preserved even after coupling with Mo_2CT_x . In the low-wavenumber region, which is related to metal-oxygen (M-O) vibrations,^{58,59} the $\text{NiCo-LDH@Mo}_2\text{CT}_x/\text{NF}$ sample showed a weaker and slightly shifted M-O band compared with NiCo-LDH/NF . This change means that the local bonding environment around the metal centers was changed after the introduction of Mo_2CT_x . In other words, NiCo-LDH was not simply mixed with Mo_2CT_x , but interacted with it at the interface. Such a change in the metal-oxygen bonding environment supports the formation of a well-connected hybrid structure between LDH and MXene.

Taken together, the XRD and FT-IR results confirm that $\text{NiCo-LDH@Mo}_2\text{CT}_x/\text{NF}$ was successfully formed while preserving the LDH framework and introducing interfacial interaction with Mo_2CT_x .

Using delaminated d- Mo_2CT_x as the support, the $\text{NiCo-LDH@Mo}_2\text{CT}_x/\text{NF}$ electrode was successfully fabricated, and its morphology and microstructure were systematically

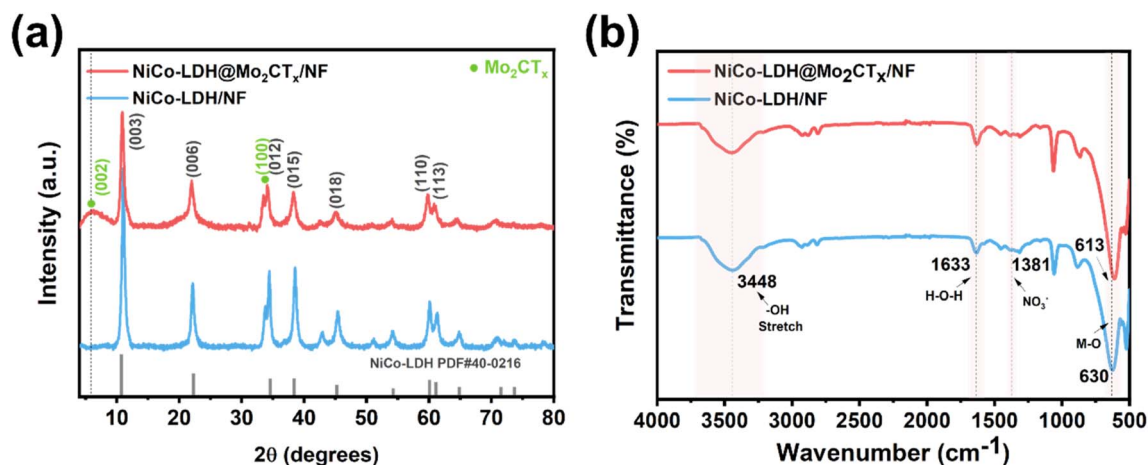


Fig. 3 (a) XRD patterns and (b) FT-IR spectra of NiCo-LDH/NF and $\text{NiCo-LDH@Mo}_2\text{CT}_x/\text{NF}$.

characterized. The low- and high-magnification FE-SEM images (Fig. 4a and b) show that NiCo-LDH nanosheets are uniformly distributed over the entire nickel foam surface, forming an interconnected and open nanosheet architecture. This morphology is clearly distinct from that of NiCo-LDH/NF prepared without d-Mo₂CT_x, which exhibits relatively large and thick plate-like sheets with dense overlap and noticeable local agglomeration (Fig. S1). In contrast, incorporation of d-Mo₂CT_x suppresses excessive sheet aggregation, resulting in a finer and more homogeneous nanosheet assembly on NF.⁶⁰ This structural modulation can increase the electrochemically accessible surface area,⁶¹ exposes more active edge sites, and preserves open transport channels for electrolyte penetration, thereby establishing a favorable electrode architecture for OER under high-current-density conditions.

The EDS elemental mapping results (Fig. 4c) show a relatively uniform distribution of O, Ni, Co, and Mo throughout the electrode, confirming the coexistence of NiCo-LDH and d-Mo₂CT_x on the NF substrate without obvious elemental segregation. FE-SEM and EDS analyses of the Mo₂CT_x@NF electrode (Fig. S2) further confirm that d-Mo₂CT_x is distributed on the NF surface. These observations verify the successful synthesis of the NiCo-LDH@Mo₂CT_x/NF electrode. After the OER test, the

nanosheet morphology was largely retained for all electrodes (Fig. S3), and NiCo-LDH@Mo₂CT_x/NF maintained its overall nanosheet-based framework without severe structural collapse. The microstructure of the NiCo-LDH@Mo₂CT_x/NF electrode was further examined by TEM. The low-magnification TEM image (Fig. 4d) shows an aggregate composed of intertwined nanosheets, while the high-magnification TEM image (Fig. 4e) reveals thin and transparent sheet-like features characteristic of a two-dimensional layered structure. This thin-sheet morphology shortens ion diffusion pathways and enables intimate interfacial coupling⁶² between NiCo-LDH and d-Mo₂CT_x.⁶³ In the HRTEM image (Fig. 4f), lattice fringes with interplanar spacings of 0.23 and 0.15 nm are assigned to the (012) and (110) planes of NiCo-LDH, respectively, while an additional lattice fringe corresponds to the 0.15 nm(100) plane of Mo₂CT_x.⁶⁴ These lattice features are in good agreement with the diffraction peaks identified in the XRD pattern, confirming the coexistence of the LDH phase and d-Mo₂CT_x within the structure. These results directly verify that the crystalline LDH phase is intimately coupled with the preserved MXene framework within the composite.

The SAED pattern (Fig. 4g) shows a clear concentric diffraction ring and confirms that the NiCo-LDH@Mo₂CT_x/NF

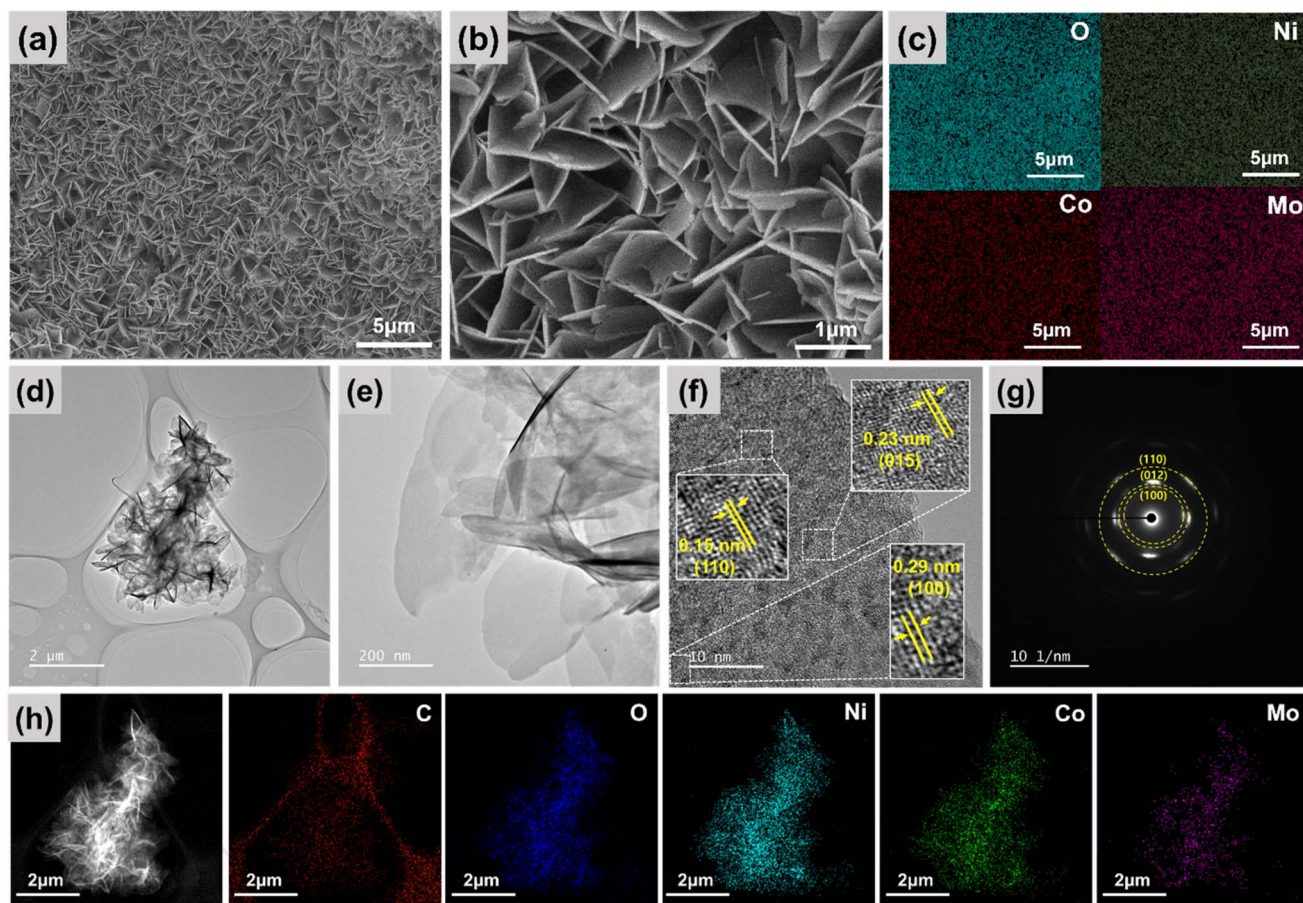


Fig. 4 (a and b) Low- and high-magnification FE-SEM images of NiCo-LDH@Mo₂CT_x/NF. (c) EDS elemental mapping of O, Ni, Co, and Mo. (d and e) Low- and high-magnification FE-TEM images. (f) HRTEM image. (g) SAED pattern. (h) STEM image and corresponding elemental mapping images.

electrode has polycrystalline properties. The observed diffraction ring can be indexed to the (100) plane corresponding to 0.29 nm of the Mo_2CT_x and to the 0.23 nm (012) and 0.15 nm (110) planes of the NiCo-LDH, respectively, which agree well with the HRTEM and XRD analysis results. These results show that the NiCo-LDH phase and the d- Mo_2CT_x structure remain together even after the formation of the composite electrode. In addition, in the STEM element mapping (Fig. 4h), it was confirmed that C, O, Ni, Co, and Mo elements were distributed throughout the nanosheet area, and through this, it can be confirmed that the NiCo-LDH and d- Mo_2CT_x coexist in the electrode. Therefore, the SAED and STEM analysis results support the successful synthesis of the NiCo-LDH@ Mo_2CT_x /NF electrodes.

X-ray photoelectron spectroscopy (XPS) was employed to probe the surface chemical composition and chemical states of the prepared electrodes. Prior to peak deconvolution, all spectra were calibrated against the adventitious C 1s peak at 284.8 eV. As shown in the survey spectra (Fig. S4), NiCo-LDH/NF exhibited the characteristic signals of Ni, Co, O, and C, whereas NiCo-LDH@ Mo_2CT_x /NF showed an additional Mo signal, confirming

the successful incorporation of Mo-containing species into the electrode. This observation is fully consistent with the EDS elemental mapping results, which revealed the homogeneous distribution of Ni, Co, O, and Mo across the electrode surface.

The high-resolution C 1s spectra (Fig. 5a) of both samples can be deconvoluted into components located at approximately 284.5, 285.3, and 287.5 eV, which are assigned to C-C/C=C, C-O, and O-C=O species, respectively. In the case of NiCo-LDH@ Mo_2CT_x /NF, an additional feature appears at 283.4–283.5 eV, which is attributed to the Mo-C bond.⁶⁵ The presence of this component confirms that the carbide-related framework of Mo_2CT_x was retained after formation of the electrode. Moreover, the Mo 3d spectrum (Fig. 5b), detected only for NiCo-LDH@ Mo_2CT_x /NF, further verifies the presence of Mo-based species on the catalyst surface. When considered together, the Mo-C component in the C 1s region and the Mo 3d signal clearly demonstrate that Mo_2CT_x was successfully incorporated into the electrode while maintaining its characteristic carbide structure. The coexistence of oxygen-containing carbon species further indicates that the Mo_2CT_x surface retained functional

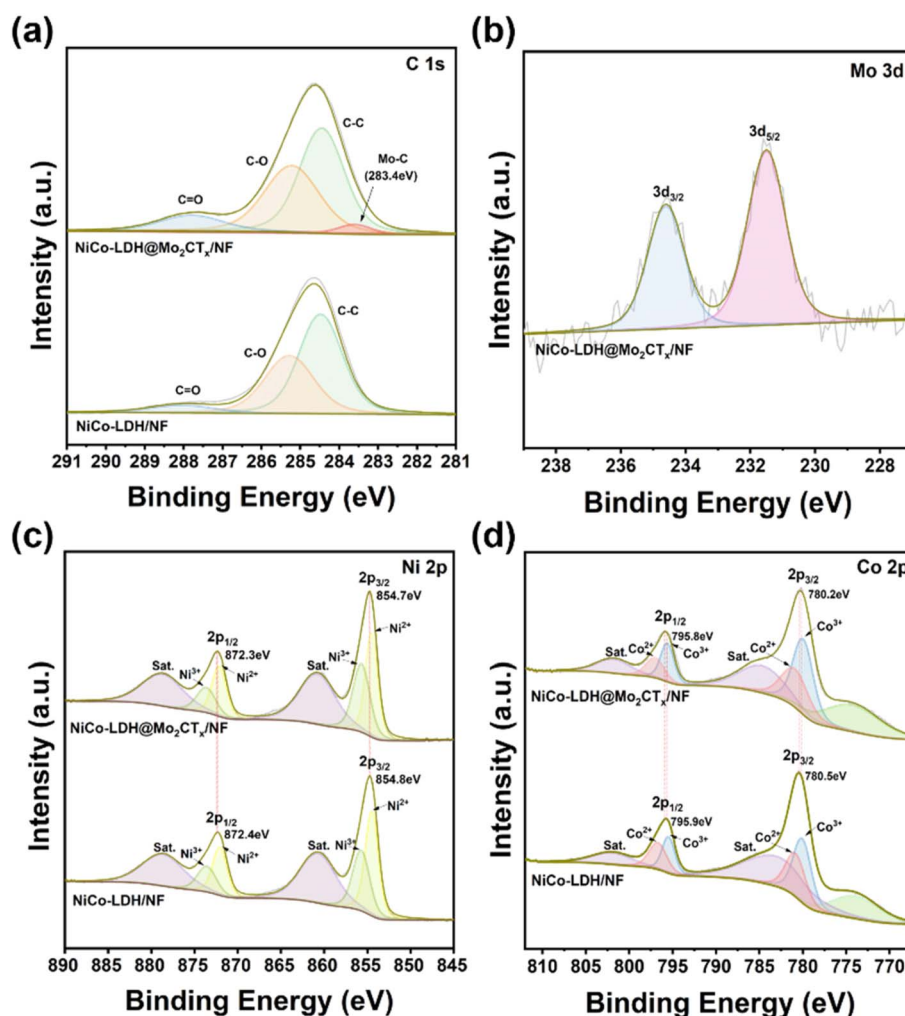


Fig. 5 XPS spectrum results of NiCo-LDH@ Mo_2CT_x /NF and NiCo-LDH/NF (a) C 1s, (b) Mo 3d, (c) Ni 2p, (d) Co 2p.

groups capable of promoting close interfacial contact with the LDH phase.

The high-resolution Ni 2p spectra (Fig. 5c) display the typical mixed-valence characteristics of LDH materials. Both NiCo-LDH/NF and NiCo-LDH@Mo₂CT_x/NF exhibit two principal spin-orbit components at 854.8/854.7 eV and 872.4/872.3 eV, corresponding to Ni 2p_{3/2} and Ni 2p_{1/2}, respectively, together with the characteristic satellite features. These spectral features confirm the coexistence of Ni²⁺ and Ni³⁺ species in both electrodes.⁶⁶ Notably, the Ni 2p peaks of NiCo-LDH@Mo₂CT_x/NF show a slight shift toward lower binding energy by approximately 0.1 eV relative to those of NiCo-LDH/NF. Although modest, this shift indicates a subtle change in the local electronic environment of the Ni centers after incorporation of Mo₂CT_x. At the same time, the overall Ni 2p line shape and mixed-valence characteristics remain essentially unchanged, demonstrating that the intrinsic chemical state of Ni in the LDH phase was largely preserved during composite formation.

A similar but more pronounced trend is observed in the Co 2p spectra (Fig. 5d). Both samples exhibit characteristic Co 2p_{3/2} and Co 2p_{1/2} peaks at 780.5/780.2 eV and 795.9/795.8 eV, respectively, along with satellite features in the higher binding-energy region, confirming the coexistence of Co²⁺ and Co³⁺ species.⁶⁷ Compared with NiCo-LDH/NF, the Co 2p peaks of NiCo-LDH@Mo₂CT_x/NF shift toward lower binding energy by approximately 0.3 eV. This larger shift indicates that the Co centers experienced a more noticeable local electronic perturbation upon introduction of Mo₂CT_x. Nevertheless, because the overall Co 2p spectral profile and mixed-valence characteristics were retained, this change is best interpreted as a local interfacial electronic effect rather than a substantial alteration of the bulk chemical state of Co.

The negative binding energy shifts observed for the Ni 2p and Co 2p spectra indicate electronic interaction between NiCo-LDH and d-Mo₂CT_x, resulting in interfacial charge redistribution within the heterostructure. Such electronic modulation is expected to alter the local electron density around the catalytically active Ni and Co sites, thereby optimizing the adsorption strength of oxygen-containing reaction intermediates (OH*, O*, and OOH*). According to previous theoretical investigations on MXene-based heterostructures, appropriate charge redistribution can bring the adsorption free energies of these intermediates closer to the thermodynamically favorable range, thereby lowering the activation energy of the rate-determining step and accelerating the OER kinetics.⁶⁸ Therefore, the enhanced catalytic performance of NiCo-LDH@Mo₂CT_x/NF can be reasonably attributed to the combined effects of improved electrical conductivity, accelerated interfacial charge transfer, and optimized adsorption behavior resulting from the strong electronic coupling between NiCo-LDH and d-Mo₂CT_x.

Taken together, the XPS results demonstrate that NiCo-LDH@Mo₂CT_x/NF was successfully formed while preserving both the characteristic Mo₂CT_x framework and the intrinsic mixed-valence chemistry of NiCo-LDH. The slightly lower binding-energy shifts observed for Ni and Co indicate that incorporation of Mo₂CT_x induced subtle local electronic tuning of the active metal centers without disrupting the fundamental

LDH chemical states. Such interfacial electronic modulation is favorable for OER operation because it can facilitate charge transport across the electrode interface and create a more favorable surface environment for anodic reaction processes. This interpretation is consistent with the electrochemical results, including the reduced charge-transfer resistance, improved reaction kinetics, and enhanced OER activity observed for NiCo-LDH@Mo₂CT_x/NF.

To evaluate the electrochemical activity of the synthesized NiCo-LDH@Mo₂CT_x/NF, OER performance was measured in a three-electrode system using 1.0 M KOH electrolyte at a scan rate of 1 mV s⁻¹. According to the LSV curves (Fig. 6a), bare nickel foam (NF), used as the substrate, exhibited the lowest current density over the entire potential range, indicating very limited OER activity. Mo₂CT_x/NF, in which conductive MXene was deposited on the substrate, showed a slight increase in current density compared with bare NF; however, its catalytic performance for the OER remained insufficient. This result indicates that Mo₂CT_x mainly functions as a conductive support rather than as a primary active phase for oxygen evolution. In contrast, NiCo-LDH@Mo₂CT_x/NF showed the earliest current onset among the compared electrodes. In particular, the sharp increase in current density with increasing potential indicates more favorable anodic reaction behavior at the catalyst surface.

In addition, the oxidation peak observed in the low-potential region is associated with the electrochemical activation of surface metal species and their transformation into catalytically active oxyhydroxide species. This redox feature was more pronounced for NiCo-LDH@Mo₂CT_x/NF, showing that the introduction of Mo₂CT_x promoted the formation of electrochemically active species. NiCo-LDH@Mo₂CT_x/NF exhibited overpotentials of 324 and 393 mV at 50 mA cm⁻² and 100 mA cm⁻², respectively (Fig. 6b), which are lower than the corresponding values of 357 and 440 mV for NiCo-LDH. In other words, the introduction of Mo₂CT_x reduced the overpotential by 33 mV at 50 mA cm⁻² and by 47 mV at 100 mA cm⁻². To further evaluate the reaction kinetics, the Tafel slopes were derived from the IR-corrected LSV curves (Fig. S5) to evaluate the OER reaction kinetics (Fig. 6c). NiCo-LDH@Mo₂CT_x/NF exhibited a Tafel slope of 97.2 mV dec⁻¹, which is substantially lower than that of NiCo-LDH/NF (157.1 mV dec⁻¹). This lower Tafel slope indicates that the incorporation of Mo₂CT_x effectively accelerates the OER kinetics. The improved kinetic behavior can be attributed to the conductive Mo₂CT_x framework, which provides efficient electron-transfer pathways between the NiCo-LDH nanosheets and the nickel foam substrate, thereby facilitating interfacial charge transport during the OER.

To compare the electrochemically accessible surface area of the electrodes, cyclic voltammetry was carried out in the non-faradaic region at different scan rates (Fig. S6), and the double-layer capacitance (*C*_{dl}) values were obtained from the corresponding capacitive current differences (Fig. 6d). NiCo-LDH@Mo₂CT_x/NF showed a *C*_{dl} value of 41.98 μF cm⁻², slightly higher than that of NiCo-LDH/NF (41.13 μF cm⁻²). Although the increase in *C*_{dl} is modest, this result indicates that the Mo₂CT_x-containing electrode provides a slightly larger electrochemically

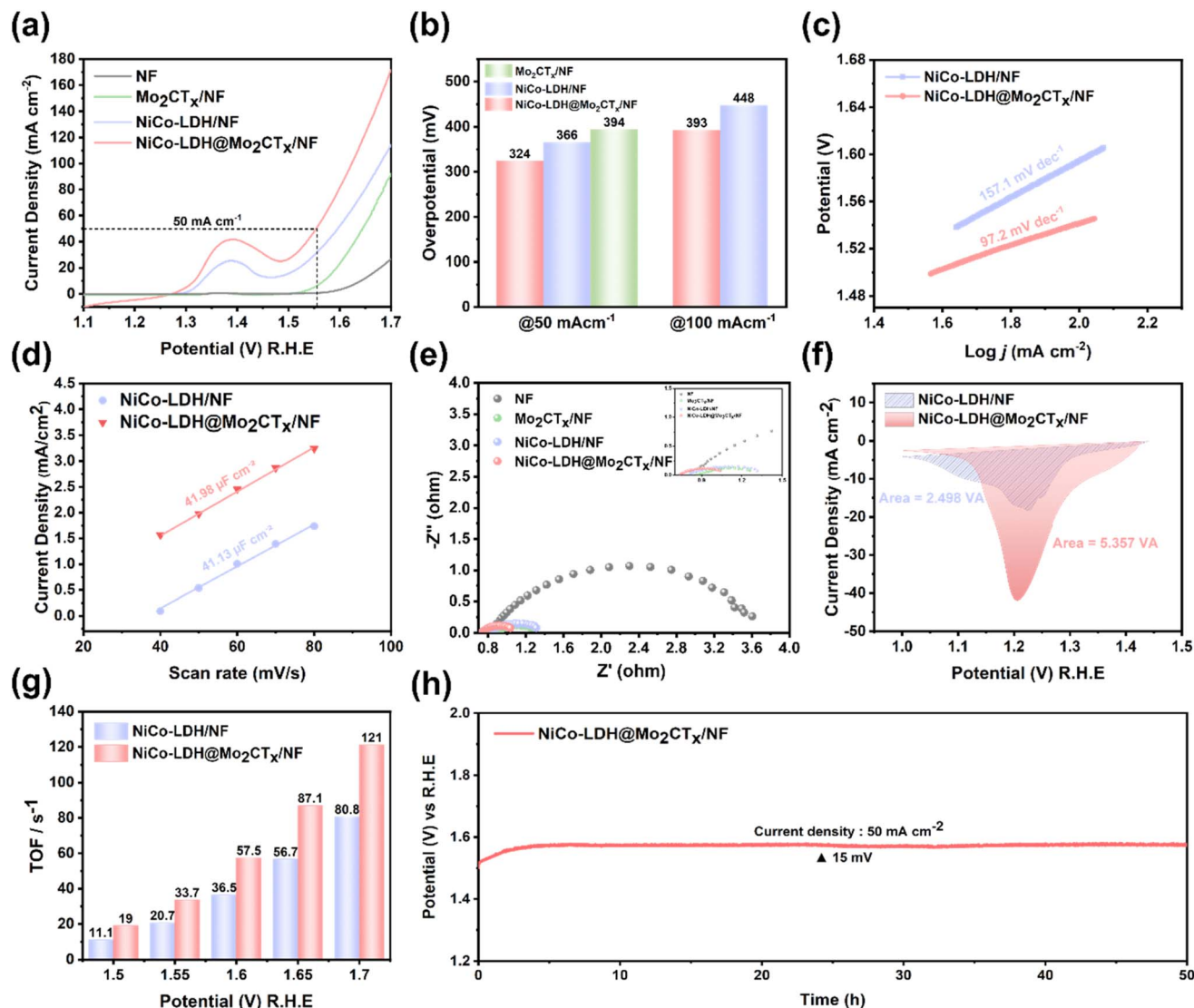


Fig. 6 Electrochemical OER performance of NF, Mo₂CT_x/NF, NiCo-LDH/NF, and NiCo-LDH@Mo₂CT_x/NF: (a) linear sweep voltammetry (LSV) curves, (b) comparison of overpotentials at 50 and 100 mA cm⁻², (c) Tafel slopes derived from the corresponding LSV curves, (d) double-layer capacitance (C_{dl}) plots, (e) Nyquist plots of the electrodes. An enlarged view of the high-frequency region is provided to facilitate comparison of the charge-transfer resistance, (f) cyclic voltammograms used to estimate the electrochemically active surface area (ECSA), (g) TOF values at different applied potentials, and (h) chronopotentiometric stability test at a constant current density of 50 mA cm⁻².

accessible interface. More importantly, the relatively small difference in C_{dl} compared with the clear improvement in Tafel slope suggests that the enhanced OER performance of NiCo-LDH@Mo₂CT_x/NF is mainly associated with improved reaction kinetics and interfacial charge transfer rather than a simple increase in surface area.

Electrochemical impedance spectroscopy was performed at 1.72 V vs. RHE to further examine the interfacial charge-transfer properties of the electrodes (Fig. 6e). In the Nyquist plots, the semicircle diameter is closely related to the charge-transfer resistance (R_{ct}). NF exhibited the largest semicircle with an R_{ct} of 2.789 Ω, indicating sluggish charge transfer at the electrode/electrolyte interface. After introducing Mo₂CT_x, Mo₂CT_x/NF showed a markedly reduced R_{ct} of 0.497 Ω, demonstrating that Mo₂CT_x itself improves the interfacial charge-transport

behavior of the NF-based electrode. NiCo-LDH/NF further decreased the R_{ct} to 0.382 Ω, reflecting the catalytic contribution of the LDH nanosheets. Notably, NiCo-LDH@Mo₂CT_x/NF exhibited the smallest R_{ct} of 0.301 Ω among all electrodes, which is lower than those of NF, Mo₂CT_x/NF, and NiCo-LDH/NF. An equivalent circuit of NiCo-LDH@Mo₂CT_x/NF model consisting of a solution resistance (R_s) connected in series with three parallel resistance-constant phase element (R-Q) components (Fig. S7). This result confirms that the integration of Mo₂CT_x with NiCo-LDH creates a more favorable charge-transfer interface, enabling faster electron transport between the active LDH nanosheets and the NF current collector. The reduced R_{ct} is consistent with the lower Tafel slope, supporting that the improved OER activity of NiCo-LDH@Mo₂CT_x/NF

mainly originates from accelerated interfacial charge-transfer kinetics.

To estimate the amount of electrochemically active reaction sites, the charge was calculated from the integrated area of the CV curves. NiCo-LDH@Mo₂CT_x/NF showed a larger integrated CV area than NiCo-LDH/NF (Fig. 6f), indicating that more electrochemically active sites were available on the electrode surface. In addition, the turnover frequency (TOF) was calculated to further compare the intrinsic reaction activity of the catalysts while minimizing the influence of catalyst loading and electrode area (Fig. 6g). Both electrodes showed a gradual increase in TOF with increasing applied potential, indicating accelerated OER kinetics under higher anodic bias. At 1.50, 1.55, 1.60, 1.65, and 1.70 V *vs.* RHE, NiCo-LDH@Mo₂CT_x/NF delivered TOF values of 19.0, 33.7, 57.6, 87.1, and 121 s⁻¹, respectively, which are consistently higher than those of NiCo-LDH/NF measured at the same potentials, namely 11.1, 20.7, 36.8, 56.7, and 80.8 s⁻¹. Notably, NiCo-LDH@Mo₂CT_x/NF reached the highest TOF value of 121 s⁻¹ at 1.70 V *vs.* RHE. These results demonstrate that the introduction of Mo₂CT_x improves the reaction activity of the NiCo-LDH electrode, indicating that the enhanced OER performance originates not only from improved charge-transfer kinetics but also from more efficient utilization of catalytically active sites.

To evaluate the long-term electrochemical durability of the catalyst, chronopotentiometry (CP) was conducted for 50 h at a constant current density of 50 mA cm⁻² (Fig. 6h). NiCo-LDH@Mo₂CT_x/NF maintained a potential of approximately 1.57 V *vs.* RHE during the stability test, indicating stable OER operation. The slight increase in the operating potential during the initial ~5 h is attributed to the electrochemical activation of the catalyst. During this period, partial surface reconstruction and the gradual formation of catalytically active Ni/Co oxyhydroxide species occur, accompanied by improved electrolyte wetting and stabilization of the catalyst/electrolyte interface. After this activation stage, the potential remains nearly constant throughout the remainder of the durability test, indicating that the catalyst maintains excellent structural integrity and electrochemical stability during prolonged OER operation. After this initial activation period, the potential remained nearly constant, varying only from 1.573 to 1.576 V throughout the subsequent operation. The negligible potential variation of approximately 3 mV demonstrates that NiCo-LDH@Mo₂CT_x/NF possesses excellent electrochemical durability under prolonged OER operating conditions.

To further assess the competitiveness of the present electrode, its OER performance was compared with representative NiCo-LDH/MXene and related MXene-supported non-precious-metal electrocatalysts reported in the literature, as summarized in Table S2. Although several reported catalysts exhibit lower overpotentials at the conventional benchmark current density of 10 mA cm⁻², direct comparison should consider differences in catalyst configuration, electrolyte, current density, loading, and stability conditions. The present binder-free NiCo-LDH@Mo₂CT_x/NF electrode was evaluated at relatively high current densities and required overpotentials of 324 and 393 mV at 50 and 100 mA cm⁻², respectively, while

maintaining stable operation for 50 h at 50 mA cm⁻². These results demonstrate a competitive balance of high-current-density OER activity, accelerated reaction kinetics, and prolonged durability, together with the practical advantage of a directly grown self-supported electrode architecture.

4 AEMWE performance

Based on the excellent OER activity observed in the three-electrode configuration, the practical applicability of the NiCo-LDH@Mo₂CT_x/NF electrode was further evaluated in an AEMWE single-cell system. The cell was assembled using a NiCo-LDH@Mo₂CT_x/NF anode and a commercial Pt/C-coated carbon cloth cathode separated by a PiperION membrane (40 μm thickness) as shown in Fig. 7a. Prior to cell assembly, the membrane was immersed in 1 M KOH for 24 h to ensure complete conversion to the hydroxide form. The AEMWE cell was operated at 60 °C with continuous circulation of 1 M KOH at a flow rate of 15 mL min⁻¹ through the anode compartment.

As illustrated in Fig. 7b, the single-cell polarization curves demonstrate that the incorporation of Mo₂CT_x into the NiCo-LDH electrode significantly improves the overall electrolysis performance. Throughout the entire voltage range, the NiCo-LDH@Mo₂CT_x/NF-based cell delivered higher current densities than the cell employing the NiCo-LDH/NF anode. In particular, the NiCo-LDH@Mo₂CT_x/NF electrode achieved a current density of approximately 1.15 A cm⁻² at 2.0 V, whereas the NiCo-LDH/NF electrode exhibited substantially lower current output under identical operating conditions (Fig. 7b). The superior cell performance is consistent with the three-electrode OER results, which showed lower overpotential, faster reaction kinetics, and reduced charge-transfer resistance for NiCo-LDH@Mo₂CT_x/NF. The conductive Mo₂CT_x framework promotes efficient electron transport within the electrode architecture while maintaining intimate interfacial contact with the active NiCo-LDH nano-sheets, thereby facilitating anodic reaction processes under practical operating conditions. To evaluate operational stability, long-term chronopotentiometric measurements were conducted at a constant current density of 500 mA cm⁻² for 100 h (Fig. 7c). The NiCo-LDH@Mo₂CT_x/NF electrode maintained a nearly constant cell voltage throughout the durability test without abrupt fluctuations or signs of performance degradation. The stable voltage profile indicates excellent structural robustness and interfacial stability during prolonged electrolysis operation. The strong adhesion of the catalyst layer to the nickel foam substrate and the enhanced charge-transfer characteristics introduced by Mo₂CT_x likely contribute to the observed durability.

Furthermore, the AEMWE performance of the present electrode was compared with previously reported transition-metal-based anodes operating under alkaline membrane electrolysis conditions (Table S3). The NiCo-LDH@Mo₂CT_x/NF electrode achieved a current density of 1.15 A cm⁻² at 2.0 V while maintaining stable operation for 100 h at 500 mA cm⁻², demonstrating competitive performance among recently reported non-precious-metal AEMWE anodes. These results confirm that integrating conductive Mo₂CT_x with NiCo-LDH provides an

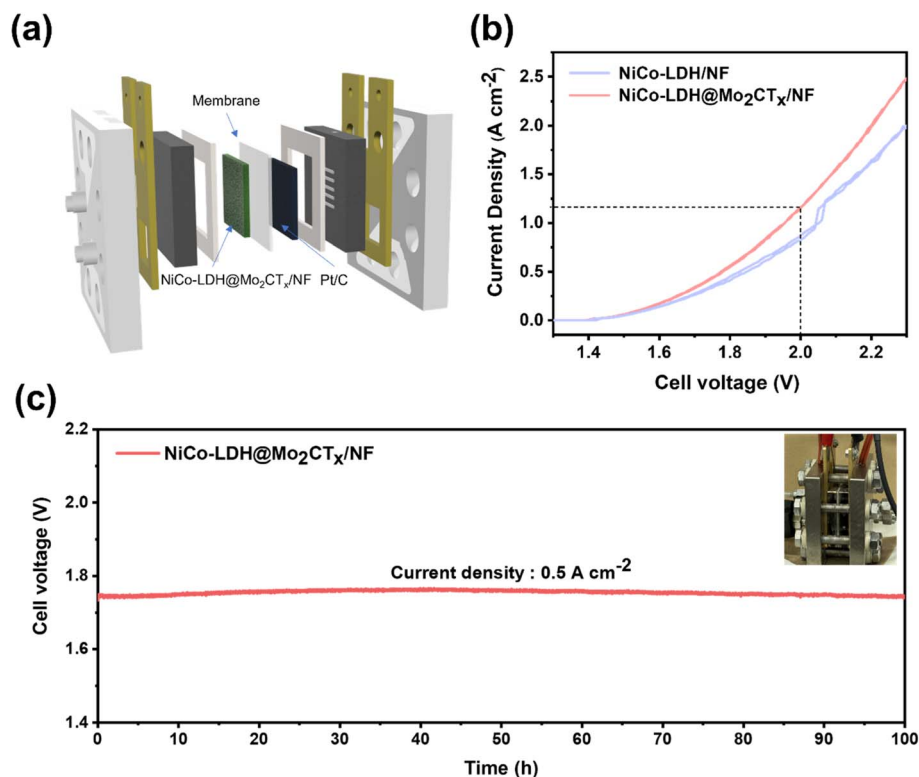


Fig. 7 (a) Schematic illustration of AEM single cell electrolyzer, (b) polarization curves of NiCo-LDH and NiCo-LDH@Mo₂CT_x in an AEM water electrolysis single cell, (c) long-term stability test of NiCo-LDH@Mo₂CT_x.

effective strategy for enhancing both activity and durability in practical AEM water electrolysis systems. Overall, the single-cell results demonstrate that the performance advantages observed in the three-electrode configuration are successfully translated into practical AEMWE operation, highlighting the potential of NiCo-LDH@Mo₂CT_x/NF as a promising non-precious-metal anode for efficient hydrogen production.

5 Conclusion

In this study, a NiCo-LDH@Mo₂CT_x/NF hybrid electrode was successfully developed by integrating conductive delaminated Mo₂CT_x MXene with NiCo-LDH nanosheets to address the intrinsically low conductivity and limited charge-transfer kinetics of LDH-based OER catalysts. The delaminated Mo₂CT_x exhibited an expanded layered structure and served as an effective conductive platform for the uniform growth of NiCo-LDH on nickel foam. Structural and surface characterizations confirmed the successful formation of the hybrid architecture and revealed strong interfacial integration between Mo₂CT_x and NiCo-LDH. The incorporation of Mo₂CT_x improved charge-transfer characteristics and promoted the exposure of electrochemically accessible active sites, resulting in enhanced OER performance. In 1.0 M KOH, the NiCo-LDH@Mo₂CT_x/NF electrode required overpotentials of 324 and 393 mV to achieve current densities of 50 and 100 mA cm⁻², respectively, outperforming the NiCo-LDH/NF electrode. In addition, the hybrid electrode exhibited improved reaction kinetics, lower charge-

transfer resistance, and excellent durability, maintaining stable operation for 50 h at 50 mA cm⁻² in a three-electrode configuration. The practical applicability of the electrode was further demonstrated in an AEM water electrolysis single-cell system. The NiCo-LDH@Mo₂CT_x/NF anode achieved a current density of 1.15 A cm⁻² at 2.0 V and maintained stable operation for 100 h at 500 mA cm⁻², confirming its suitability for long-term alkaline water electrolysis. Overall, this work demonstrates that integrating conductive MXene with transition-metal LDH catalysts is an effective strategy for enhancing both catalytic activity and durability, providing valuable insights for the development of high-performance non-precious-metal anodes for AEM water electrolysis.

Author contributions

Soojin Kwon: conceptualization, methodology, writing – original draft. Yujin Cho: writing – review & editing. Komal Patil: writing – review & editing. Daim Choe: data curation. Jiwon Choi: visualization. Minseo Son: visualization. Dong-Won Kang: supervision, and Jongsung Park: supervision, project administration, funding acquisition.

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

The author confirms that the data supporting the findings of this study are available in the article and its supplementary information (SI). Supplementary information is available. See DOI: <https://doi.org/10.1039/d6ra05281a>.

Acknowledgements

This work was supported by the Korea Institute of Energy Technology Evaluation and Planning (KETEP) and the Ministry of Climate, Energy & Environment (MCEE) of the Republic of Korea (No. RS-2025-25450823) and the National Research Foundation of Korea (NRF) grant funded by the Korea government (MEST) (RS-2023-00257494).

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