JNCS Journal of Nepal Chemical Society
January 2026, Vol. 46, No. 1, 32-40
ISSN: 2091-0304 (print)
DOI: https://doi.org/10.3126/jncs.v46i1.91120
RESEARCH ARTICLE

Doped Layered Double Hydroxides for Efficient Hydrogen Evolution Reaction

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(Received: December 29, 2025, revised: January 15, 2026, accepted: January 25, 2026)

Abstract

Hydrogen is considered a primary solution to meet the global energy demand of the present and future generations, as it is clean and renewable. To produce hydrogen efficiently and cost-effectively, layered double hydroxide (LDH)-based electrocatalysts have been widely employed to enhance the hydrogen evolution reaction (HER). In this study, a hybrid bimetallic iron–cobalt–layered double hydroxide (Fe–Co–LDH) synthesized on a clean nickel foam (NF) substrate to develop an active electrocatalyst for HER. In an alkaline medium, the Fe–Co–LDH/NF electrocatalyst delivered low overpotentials of 175 mV at 10 mA cm^{-2} and 238 mV at 50 mA cm^{-2}, and the Tafel slope of 91 mV dec^{-1}. The high activity is attributed to the synergistic effect of Fe and Co, the layered structure providing abundant active sites, good electrical conductivity, and strong metal–hydroxide interactions facilitating water dissociation. These findings suggest that the synthesized Fe–Co–LDH electrocatalyst holds great promise for efficient HER.

Keywords: Electrocatalyst; Fe-Co-LDH; Hydrogen evolution reaction; Precipitation; Water-splitting

Introduction

Energy is the essence of economic and technological development [1] [2]. The extensive use of fossil fuels for commercial and public purposes has led to the continuous rise of carbon dioxide and other greenhouse gas emissions, such as methane, carbon monoxide, and nitrous oxides, which contribute to environmental degradation and health risks [3] [4]. Transitioning to affordable, accessible, and sustainable energy sources is essential to support economic growth, improve energy security, and mitigate climate change impacts [5]. Among various sustainable approaches, electrochemical water splitting has emerged as a promising strategy for green hydrogen production [6]. Water splitting involves two key reactions: HER, OER. In electrolytic cells, HER and OER at the cathode and anode generate molecular hydrogen and oxygen gases [7] [8]. Water electrolysis, consisting of the cathodic HER and anodic OER half-reactions, is inherently slow due to the high activation energy and sluggish charge transfer at the electrode surface, requiring an applied overpotential to achieve the desired current density [9]. Consequently, designing effective electrocatalysts is crucial to reducing activation potentials and improving water-splitting efficiency. Although noble metals like Pt, Ru, and Pd are highly efficient for HER and OER, their high cost and limited availability restrict large-scale applications [10]. Recent research has focused on utilizing earth-abundant transition metal such as Fe, Co, Ni, Cu, and Mn,

and their derivatives, including alloys, nitrides, carbides, sulfides, borides, phosphides, and layered double hydroxides (LDHs) as electrocatalysts [2] [11]. Among these, LDHs composed of first-row transition metals have received significant attention for water-splitting applications due to their tunable structures and high electrochemical activity [12]. Several studies have demonstrated the effectiveness of LDH-based catalysts: For instance, an amorphous B-incorporated Ni–Co LDH supported on nickel foam (A-NiCo LDH/NF) exhibited remarkable HER performance, delivering 100 mA cm^{-2} at an overpotential of 151 mV and maintaining excellent durability for over 72 hours in alkaline medium, surpassing commercial Pt catalysts. This study demonstrated that structural amorphization through B-doping effectively optimizes LDH electrocatalysts for practical water electrolysis applications [13]. An iron-doped cobalt-based layered double hydroxide (Fe–Co LDH) electrocatalyst was synthesized and characterized for hydrogen evolution reaction (HER) applications. The Fe incorporation enhanced the catalytic activity and stability, making it a promising candidate for efficient water splitting [14]. Similarly, A novel CoMo-LDH ultrathin nanosheet was synthesized as a low-cost, bifunctional electrocatalyst for both HER and OER in water splitting. The Mo^{6+} doping and ultrathin structure enhanced its activity and stability, achieving high efficiency and durability for overall water splitting [15]. These studies highlight the importance of bimetallic synergy and structural engineering in enhancing the catalytic performance of LDHs.

Layered double hydroxides, also known as anionic clays, consist of brucite-like layers with divalent and trivalent metal cations coordinated by hydroxide ions, while interlayer spaces contain anions and water molecules [16]. The general chemical formula is $M(II)_{1-x}M(III)_x(OH)_2(An^-)_{x/n}\cdot yH_2O$, where M(II) and M(III) are divalent and trivalent metal cations, An^- represents interlayer anions, and yH_2O denotes interlayer water [17]. LDHs are typically synthesized via co-precipitation, hydrothermal, or sol-gel methods [2]. Their unique adjustable structure allows for high surface area, abundant active sites, and tunable electronic properties, making them excellent electrocatalysts [18]. Doping strategies, such as substituting a portion of Co^{3+} with Fe^{3+}, can modify the electronic structure, enhance charge transfer, and optimize hydrogen adsorption, thereby improving HER performance [19].

Herein, we report a facile and scalable co-precipitation strategy to directly grow Fe-doped Co-layered double hydroxide (Fe–Co–LDH) nanosheets on clean nickel foam at low temperature, producing a binder-free and self-supported electrocatalyst for the hydrogen evolution reaction. In contrast to previously reported Fe–Co–LDH systems that often rely on hydrothermal synthesis, high-temperature treatment, or complex structural engineering, the present approach enables effective electronic modulation through controlled Fe^{3+} incorporation using a simple precipitation route. A direct comparison with pristine Co-LDH prepared under identical conditions allows the specific role of Fe doping in enhancing charge transfer kinetics and HER activity to be clearly elucidated. The synergistic interaction between Fe and Co, combined with the layered architecture and intimate electrical contact with the nickel foam substrate, results in reduced overpotential and a significantly lower Tafel slope. This work demonstrates that rational compositional tuning of LDHs via a straightforward synthesis method can deliver efficient HER electrocatalysts for alkaline water splitting.

Materials and Methods

Chemicals

Cobalt (II) nitrate hexahydrate [$Co(NO_3)_2.6H_2O$, S.D. Fine-chem Ltd., Ferric (III) nitrate nano hydrate [$Fe(NO_3)_3.9H_2O$, Thermo Fisher Scientific India Pvt. Ltd.], Ammonium fluoride [NH_4F, Merck Specialties Pvt. Ltd.,

Urea [NH_2CONH_2, Blulux Laboratories Pvt. Ltd.], Conc. Hydrochloric acid [HCl], ethanol [C_2H_5OH], and distilled water. All the chemicals stated above were provided by the chemical lab in Amrit Campus. Nickel foam (NF) was also provided from the campus.

Fabrication of Fe-doped Co-LDH Electrocatalyst

The nickel foam (NF) of (1 × 1) cm^2 was added to a beaker containing 1 mL of HCl and 250 mL of distilled water, sonicated for 1 hour, washed seven times with distilled water, and rinsed with ethanol, then dried in an oven for 6 hours [20]. Separately, 5 g of $Co(NO_3)_2$, 0.5 g of $Fe(NO_3)_3$, 5 g of NH_4F, and 3 g of NH_2CONH_2 were dissolved in 50 mL of distilled water in a 150 mL beaker and sonicated for 1 hour. The pre-cleaned NF was then immersed in this solution, the beaker sealed with aluminum foil, and the mixture placed on a hot plate at 60°C for 8 hours. After cooling, the NF was retrieved, and the remaining solution was filtered to collect any residue. The samples were dried in an oven for 6 hours before characterization. The Co-LDH was synthesized using the same procedure with identical precursors, omitting the Fe precursor.

Electrocatalyst Characterization

Physicochemical characterization of the Fe-Co-LDH electrocatalyst for HER was carried out using Field Emission Scanning Electron Microscopy (FESEM) and X-ray diffraction (XRD) [21]. FESEM employed a concentrated electron beam to scan the sample surface and capture its topography, revealing surface structures and microfeatures. XRD, particularly powder X-ray diffraction (PXRD), was used to determine the crystal phases of the LDH. Electrochemical characterization was performed using linear sweep voltammetry (LSV), electrochemical impedance spectroscopy (EIS), and chronopotentiometry (CP) [22]. LSV assessed the electrocatalyst's activity and electron transfer rates by applying a linearly varying potential and measuring the resulting current [23]. All electrochemical measurements were performed using a three-electrode configuration, where an Ag/AgCl electrode was used as the reference electrode, and a graphite rod served as the counter electrode. The measured potentials were iR-corrected to account for solution resistance, as obtained from electrochemical impedance spectroscopy. All potentials have been converted to the reversible hydrogen electrode (RHE) scale using the standard conversion equation:

$$E_{\mathrm{RHE}} = E_{\mathrm{reference}} + 0.059\,\mathrm{pH} + E^{0}_{\mathrm{reference}}$$

EIS evaluated the charge transfer resistance to study the electrochemical properties and efficiency of Fe-Co-LDH, while CP measured the kinetics of electrochemical reactions and the stability of the electrocatalyst by recording potential changes under a constant applied current over time [24].

Results and Discussion

The formation of a vertical nanosheet of cobalt hydroxide on the nickel foam through the reactions of specific precursors marked the start of the fabrication of Fe-Co-LDH@NF heterostructure. On the hot plate, urea decomposed into ammonia and carbon dioxide. The ammonia then underwent hydrolysis at elevated pressure and temperature, slowly releasing hydroxide ions into solution, resulting in the formation of Fe-Co-LDH nanosheets on the nickel foam [25]. NH_4F helped in refining the material's morphology by boosting the binding energy of the metal ions, aiding the development of the LDH structure within the nickel foam [20]. Furthermore, urea and ammonium fluoride created an alkaline condition that directed the morphological growth. Leftover carbon and nitrogen did not influence the chemical composition [9]. Post-synthesis, with distilled water and ethanol, any impurities were rinsed off several times, guaranteeing a clean Fe-Co-LDH [12]. In this study, NF was used as a substrate, as it has high conductivity and is of low cost [19]. **Figure 1** displays the synthesis processes for Fe-Co-LDH. The growth of Fe-Co-LDH on the NF resulted in a color shift of dark pink to brown

because of the hydrolysis of Fe and Co ions.

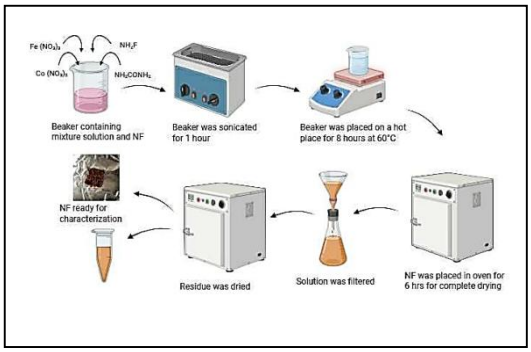

Figure 1. Schematic diagram of the fabrication of Fe-Co-LDH.

Physicochemical Characterization

The FESEM image revealed that the Fe-Co-LDH designed for the HER exhibited a distinct layered nanosheet structure (**Figure 2a**). The image showed a porous and well-defined layered morphology, indicating a high surface area and abundant active sites [26]. A network of interconnected nanosheets and voids was observed, which is crucial for efficient HER performance [20]. The porous and layered configuration facilitates electron transfer and gas diffusion pathways, thereby enhancing catalytic efficiency [10].

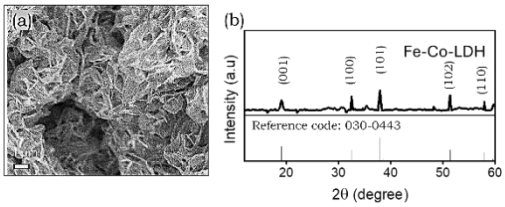

Figure 2. **a**) FESEM image of Fe-Co-LDH and **b**) XRD of Fe-Co-LDH.

The powder X-ray diffraction (PXRD) analysis was performed to confirm the successful synthesis of the desired electrocatalyst [27]. The crystalline phase of Fe-Co-LDH was verified through PXRD patterns (**Figure 2b**). As described in the experimental section, Fe-Co-LDH was synthesized using a simple aging method at room temperature. The diffraction peaks observed at 2θ values of 19.0°, 32.4°, 37.9°, 51.4°, and 57.9°, corresponding to the (001), (100), (101), (102), and (110) crystal planes, respectively (Reference code: 030-0443), confirmed the formation of Fe-Co-LDH [16].

Electrochemical Characterization

Linear Sweep Voltammetry (LSV)

LSV provides current density data as the applied potential changes, offering insight into the catalytic capability of Fe-Co-LDH for reactions such as the HER [2]. The onset potential and efficiency can be determined from the position and shape of peaks on the LSV curve. These data are crucial for comparing the performance of Fe-Co-LDH with other electrocatalysts and evaluating its catalytic activity. The electrochemical performance of the Fe-Co-LDH electrode was tested in 1.0 M KOH solution. The LSV curve (**Figure 3a**) for Co-LDH demonstrated an overpotential of 187 mV to achieve a current density of 10 mA cm^{-2} and 275 mV for 50 mA cm^{-2}, indicating relatively poor electrocatalytic activity due to higher overpotential requirements.

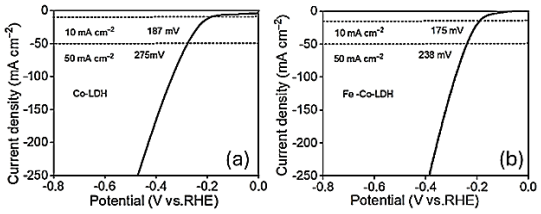

Figure 3. **a**) Voltammetry curve of Co-LDH and **b**) Voltammetry curve of Fe-Co-LDH.

In comparison (**Figure 3b**), Fe-Co-LDH exhibited a lower overpotential of 175 mV at 10 mA cm^{-2} and 238 mV at 50 mA cm^{-2}. This demonstrates that Fe-Co-LDH exhibits better electrocatalytic performance compared to Co-LDH, owing to reduced overpotential and faster kinetics. The synergistic interaction between Co atoms and Fe dopants within the LDH lattice enhances charge transfer and intrinsic catalytic activity [9]. Furthermore, the reaction kinetics of the catalysts toward the HER were analyzed using Tafel plots (**Figure 4**). The Fe–Co–LDH electrode delivers a Tafel slope of 91 mV dec^{-1}, which is markedly smaller than that observed for Co-LDH (133 mV dec^{-1}). This decrease in Tafel slope reflects a notable enhancement in HER kinetics after Fe incorporation, indicating more efficient electron

transfer during the catalytic process. The improved performance can be attributed to the electronic interaction between Fe and Co within the LDH lattice, which optimizes the adsorption and subsequent evolution of hydrogen intermediates. The obtained Tafel slope value implies that the HER on Fe–Co–LDH proceeds predominantly through a Volmer–Heyrovsky pathway, with the electrochemical desorption step governing the overall reaction rate. These results clearly demonstrate the superior intrinsic catalytic activity of Fe–Co–LDH compared with the undoped Co-LDH counterpart.

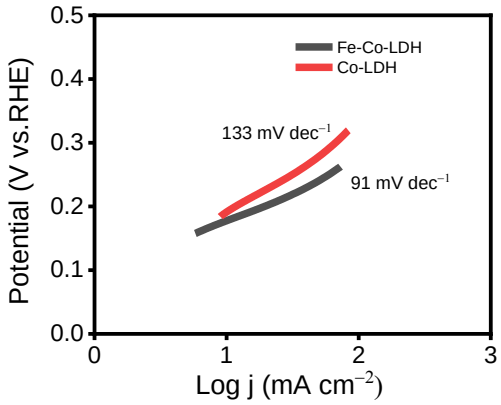

Figure 4. Tafel slope of Co-LDH and Fe-Co-LDH.

Electrochemical Impedance Spectroscopy (EIS)

EIS was performed to investigate the electrical and interfacial properties of Fe-Co-LDH during HER [9]. This technique measures the impedance of the material in response to an alternating current signal. For Fe-doped Co-LDH, EIS provided key insights into charge transfer resistance (R_{ct}), indicating how easily electrons move between the electrode and electrolyte. Lower R_{ct} values correspond to improved catalytic efficiency [6]. Additionally, EIS evaluates capacitance, reflecting the material's ability to store and release charge rapidly, which is crucial for effective catalysis. The Nyquist plot of Co-LDH (**Figure 5a**) began near the origin, forming a small loop around 0.5 Ω (R_s) and a semicircle of approximately 4 Ω (R_{ct}) extending to 4.5 Ω. This pattern indicated significant charge transfer resistance but adequate ion transport, suggesting moderate catalytic activity. In contrast, Fe-doped Co-LDH (**Figure 5b**) exhibited a smaller semicircle, starting at 0.4 Ω (Rs) with a diameter of approximately 2 Ω (Rct), indicating lower charge transfer resistance and enhanced ion diffusion [9] [10]. These improvements confirm that Fe doping effectively enhances the electrical conductivity and catalytic kinetics of Co-LDH, consistent with the polarization data [28].

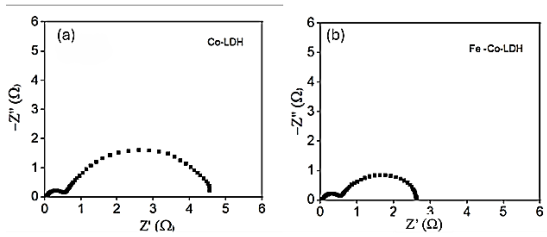

Figure 5. a) EIS curve of Co-LDH and **b**) EIS curve of Fe-Co-LDH

Chronopotentiometry (CP) and Stability Test

The stability of an electrocatalyst reflects its ability to maintain structural and electrochemical integrity during operation [29]. The kinetics of the electrochemical reaction were analyzed by applying a constant current over time. The chronopotentiogram (**Figure 6**) plotted potential versus time and showed a stable potential of –0.25 V for Fe-Co-LDH over 12 hours, indicating excellent electrochemical durability. The incorporation of Fe dopants improved the structural robustness and chemical stability of the Co-LDH electrode.

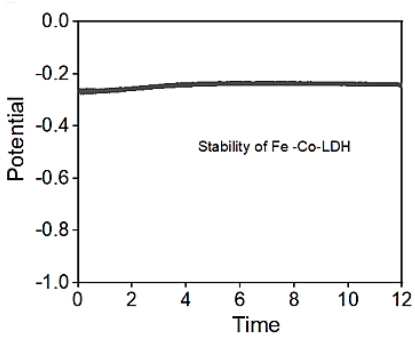

Figure 6. Chronopotentiogram of Fe-Co-LDH.

Conclusions

A facile room-temperature aging method was successfully employed to synthesize Fe-Co-LDH electrocatalysts for HER. XRD confirmed the incorporation of Fe into the Co-LDH lattice without disturbing the crystal structure, while FESEM images revealed uniformly distributed nanosheets with a large surface area, providing abundant active sites. Electrochemical analyses demonstrated that Fe doping significantly enhanced HER activity, with overpotentials of 175 mV and 238 mV at current densities of 10 and 50 mA cm^{-2}, respectively. EIS results showed reduced charge transfer resistance, and CP confirmed excellent long-term stability. The synergistic interaction between Fe and Co optimized the electronic structure, improved conductivity, and increased the number of active sites, establishing Fe-Co-LDH as a highly efficient and durable electrocatalyst for hydrogen evolution.

Acknowledgements

The authors extend their gratitude to Amrit Campus (ASCOL), Tribhuvan University, for providing laboratory facilities. They would also like to thank Mr. Ganesh Bhandari and Keshav Chapaingai at the JBNU, South Korea, for their assistance with sample analysis.

Author's contribution statement

J. Phagami: Methodology, Investigation, Formal analysis, Data curation, Writing-original draft preparation. **P. Koirala:** Formal analysis **D. Oli:** Formal analysis **B. Bhatta:** Formal analysis **K. Swarnakar:** Formal analysis **B. Karki:** Formal analysis and Editing **A. Chaudari:** Review and Editing **M. Ghimire:** Formal analysis **M. R. Kandel:** Conceptualization, Data curation, Writing-original draft preparation, Writing-review and Editing, Resources, Supervision, Project handling

Conflict of interest

The authors declare no conflict of interest.

Data availability statement

The data supporting the findings of this study are available from the corresponding author upon reasonable request.

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