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ENHANCING OXYGEN EVOLUTION REACTION PERFORMANCE USING LAYERED DOUBLE HYDROXIDE CATALYSTS: INSIGHTS FROM RECENT ADVANCES IN ELECTRONIC STRUCTURE AND SURFACE ENGINEERING

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ABSTRACT

The oxygen evolution reaction (OER) is a key but slow step in water splitting for green hydrogen production, often limited by the poor stability and sluggish kinetics of conventional electrocatalysts. Layered double hydroxides (LDHs) have recently attracted considerable attention due to their tunable layered structure, large surface area, and efficient charge transport, all of which can be optimised for improved OER activity. This review summarises major advances reported over the past five years in the synthesis, modification, and application of LDH-based catalysts, with particular emphasis on how synthesis strategies and metal cation selection influence catalytic performance. Recent studies demonstrate that tailoring the electronic structure and density of active sites through approaches such as heteroatom doping, defect engineering, and surface modification significantly enhances catalytic efficiency, leading to lower overpotentials and faster reaction kinetics. These findings highlight the strong potential of LDH-based materials as cost-effective and high-performance OER catalysts for water splitting. However, challenges related to long-term structural stability and mass transport limitations remain. Overcoming these challenges via improved catalyst designs and integration strategies will help translate LDH-based materials into practical systems for producing hydrogen sustainably. In a nutshell, the insights summarised in this review guide the rational design of next-generation LDH electrocatalysts.

Keywords: Layered Double Hydroxides (LDHs), Oxygen Evolution Reaction (OER), Electrocatalysis, Water Splitting, Hydrogen Production, Sustainable Energy, SDG-7.

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INTRODUCTION

Growing global energy demand and the uneven distribution of fossil fuel resources have intensified the search for sustainable and environmentally friendly energy alternatives. Among the promising solutions, hydrogen produced through water splitting has attracted considerable attention because it generates only hydrogen and oxygen without emitting greenhouse gases. However, despite its potential as a clean energy carrier, large-scale implementation of water electrolysis still faces several technical and economic challenges.[1–3] The process requires high energy input, durable electrolysers, and affordable catalysts, while hydrogen storage, transport, and infrastructure also remain significant obstacles.[4–6] In this context, developing efficient and stable electrocatalysts is critical for improving the overall efficacy of water-splitting and enabling practical hydrogen production. Advancements in such technologies directly support the goals of the United Nations Sustainable Development Goal-7, which emphasises developing cleaner and sustainable energy systems. A major bottleneck in water electrolysis is the oxygen evolution reaction (OER), which is kinetically sluggish and requires high overpotentials, making the process energy intensive. Traditional catalysts such as IrO_2 and RuO_2 show marked efficiency, but their higher costs and

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limited long-term stability under harsh electrochemical conditions restrict their large-scale application.[7,8] As a result, considerable research efforts have been directed toward developing alternative catalysts that are both efficient and economically viable. Layered double hydroxides (LDHs) are considered promising materials because of their lower cost, compositional flexibility, and high surface area, making these materials viable for OER electrocatalysis.[9–12] They generally follow the formula:

$$M^{+2}\left[M^{+2}_{1-x}\, M^{+3}_{x} OH_2\right] x^{+} . [A^{-n}_{\frac{x}{n}} . mH_2O]^{-x}$$

In this context, M^{+2} denotes divalent cationic metals like magnesium and zinc, while M^{+3} refers to trivalent cationic metals, such as aluminium and iron. A^{-n} represents anions that are intercalated, including chlorides, carbonates, and nitrates.[13] Here, x indicates the fraction of trivalent cations, and m specifies the number of water molecules involved. These materials possess a brucite-like layered structure (Fig.-1) in which metal cations are octahedrally coordinated with hydroxide ions, allowing for the exchange of interlayer anions.[14]

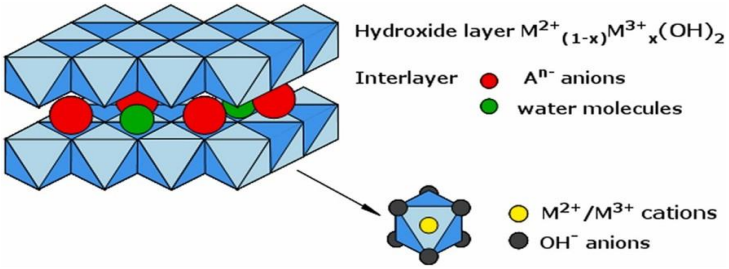

Fig.-1: Structure of LDH. Picture Credit: M.V. Bukhtiyarova, Reproduced with Permission Under CC BY 4.0 License[15]

EXPERIMENTAL

Recent studies on LDHs as OER electrocatalysts have largely focused on optimising their active sites and structural properties to improve catalytic efficiency. However, a comprehensive synthesis of recent developments covering functionalization strategies, catalytic mechanisms, and challenges related to practical deployment is still needed. Therefore, this review presents recent advances in LDH-based OER catalysis, summarising progress in synthesis strategies, structural modifications, catalytic performance, and emerging research directions aimed at developing efficient and stable OER systems for sustainable hydrogen production.[16,17] A A systematic literature review was conducted to identify recent studies on layered double hydroxide (LDH)-based electrocatalysts for the oxygen evolution reaction (OER). Relevant publications were retrieved from Scopus, Web of Science, and arXiv, yielding 330 records. After removing duplicates and out-of-scope entries, 180 records were finalised. The inclusion criteria were focused on the synthesis, structural or compositional modification, and/or electrochemical evaluation of LDHs as OER electrocatalysts and provided primary experimental or computational data. Only peer-reviewed articles published in English between 2020 and 2025 were considered. Studies not investigating LDHs or OER, lacking original data, or not meeting quality standards were excluded. Following full-text assessment, 56 studies were retained. The selection process followed the PRISMA 2020 framework and is illustrated in Fig.-2.

Approaches for improving the OER efficiency of LDHs

Compositional Tuning and Elemental Doping

Incorporating various transition metals into LDHs is a proven strategy for improving OER efficiency.[20] NiFe-LDHs exemplify this, as iron modifies nickel's electronic properties and supplies additional active sites, boosting performance. Advanced designs have further enhanced NiFe-LDHs by adding a third metal. For instance, tungsten (W) improves both structural stability and active sites, as demonstrated by Guo *et al.* (2021).[21] Other metals like manganese (Mn) and cobalt (Co) have also been studied: Mn increases durability, while Co enhances electronic conductivity, both important for activity as reviewed by Sahoo *et al.* (2022).[22] Fan *et al.* (2023) found that molybdenum (Mo) shifts the electronic d-band, helping bind oxygen intermediates and increasing OER efficiency.[23]

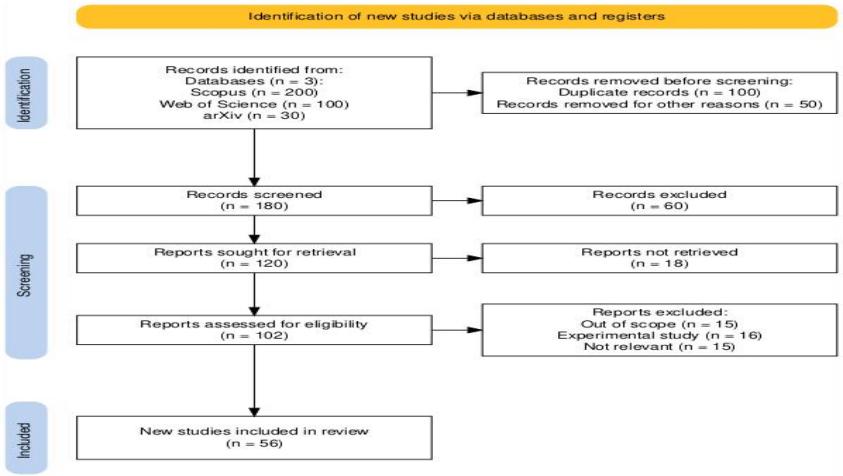

Fig.-2: PRISMA 2020 Flow Diagram Illustrating the Identification, Screening, and Inclusion Process for Studies on LDH-based OER Electrocatalysts[18,19]

Beyond metal composition, architectural engineering also plays a key role. Zhou *et al.* synthesised Fe-doped $Ni_{0.83}Fe_{0.17}(OH)_2$ nanosheets with a porous, defect-rich structure, yielding a low overpotential (245 mV at 10 mA cm^{-2}) and a modest Tafel slope (61 mV dec^{-1}), outperforming conventional NiFe-LDHs.[24] Chen *et al.* used a MOF precursor to design a Co-based LDH catalyst (Co@N-CS/N-HCP@CC), achieving high OER activity at an overpotential of 248 mV and a low Tafel slope of 68 mV dec^{-1}.[25] Further, Liu *et al.* developed a phosphorus-doped 3D catalyst (P-(Ni, Fe)$_3S_2$/NF) exhibiting excellent water electrolysis performance and notable stability, with an exceptionally lower Tafel slope of 30 mV dec^{-1}.[26] This improvement was linked to beneficial surface oxidation during electrolysis, a phenomenon common to transition metal-based sulfides, nitrides, and phosphides.

Morphology and Nanostructure Engineering

The OER performance of LDHs is greatly affected by their morphology and nanostructure. Engineering LDHs into two-dimensional (2D) nanosheets is a common strategy to increase surface area and enhance the accessibility of active sites, thus furthering their reactivity.[27,28] Thinning LDH layers improves mass transport and surface area, as demonstrated by Jiang *et al.* (2020), where ultrathin NiFe-LDH nanosheets showed superior OER activity due to these factors.[29] More complex hierarchical nanostructures also boost performance by facilitating mass and ion flow through interconnected pores or 3D frameworks. Yin *et al.* (2015) created a structure combining nanorods and nanosheets that improved catalyst stability and charge transfer during OER.[30] Sun and colleagues developed monolayer nickel-vanadium LDHs (NiV-LDHs) just 0.9 nm thick, delivering a large surface area that exposed active sites effectively.[31] This catalyst outperformed NiFe-LDH, with vanadium enhancing adsorption of OOH* intermediates and boosting activity as shown by DFT calculations. Similarly, Hu *et al.* synthesised ultrathin cobalt-manganese (CoMn) LDH nanosheets (~3.6 nm thick), which exhibited excellent OER activity, such as a lower overpotential of 324 mV at 10 mA cm^{-2} and a smaller Tafel slope of 43 mV dec^{-1}.[32] After 12 hours of operation, performance further improved to a lower overpotential of 293 mV. This was attributed to amorphous regions and the formation of highly active Co (IV) species on the nanosheet surface, which enhances intrinsic catalytic activity.

Electronic Structure Tuning and Defect Engineering

Modulating the electronic structure of LDHs is a key approach to enhance their OER activity, achieved by elemental doping, defect engineering, or creating oxygen vacancies.[33-35] Cai *et al.* (2019) demonstrated that doping heteroatoms such as sulfur, phosphorus, or nitrogen effectively tunes the electronic environment around metal centres, enhancing interaction with oxygen intermediates and boosting catalytic activity.[36] Similarly, creating oxygen vacancies introduces new active sites that promote oxygen species adsorption and lower OER energy barriers. Liu *et al.* showed that oxygen vacancies in NiFe-LDHs improve both electronic conductivity and catalytic efficiency[37], while Long *et al.* (2024) found

sulfur doping increases oxygen vacancies, further enhancing activity.[38] Song and Hu exfoliated bulk LDHs into single-layer nanosheets (NiFe, NiCo, CoCo), confirmed by XRD and AFM, showing significantly better OER performance than bulk forms. These single layers exhibit smaller Tafel slopes and higher turnover frequencies (TOF), indicating stronger electron conductivity and faster transport. The improved activity stems from a higher density of active sites and enhanced conductivity, with edge sites on nanosheets playing a crucial role by exposing more highly active centres for water oxidation, thus dramatically improving OER efficiency.[39]

Anion Intercalation, Surface Functionalization and Hybridisation with Conductive Supports

Anion insertion into LDH layers, such as carbonate, nitrate, and sulfate, is essential for structural stability and charge redistribution during OER.[40] According to the kinetic study conducted by Berger *et al.* (2023), the type of anion used for intercalation greatly affects the electrochemical activity of the NiFe-LDHs.[41] Sulfate ions significantly reduce overpotential by enhancing electron transport in NiFe-LDHs. Surface functionalization with organic molecules further lowers activation barriers by tuning oxygen intermediate binding.[42] Hybridising LDHs with conductive supports like MOFs, graphene, and carbon nanotubes improves charge transfer and mechanical stability. Graphene derived from algae boosts NiFe-LDH activity by increasing conductivity and clustering active sites.[43] CNT hybrids enhance durability and decrease overpotential. Vertically aligned NiFe-LDH nanosheets grown on nickel foam form porous 3D structures, achieving a low onset overpotential of 230 mV and a Tafel slope of 50 mV dec^{-1}.[44]

Recent Developments in LDH Electrocatalysts

Co and Fe-based LDHs

A pioneering study by Han et al. revealed that CoFe-LDHs outperform the benchmark RuO_2 catalyst for the OER, due to a strong synergistic effect between cobalt and iron combined with their layered nanoscale structure, which enhances ion and mass transport.[45] Cobalt acts as the primary catalytic site, while iron modifies the electronic environment to improve electron mobility, significantly reducing overpotential and boosting catalytic efficiency.[46-48] The 2D LDH structure maximises electrolyte contact and ion conductivity, further improved by tuning interlayer spacing to expose active sites, along with excellent durability in alkaline conditions, making CoFe-LDHs an economically attractive alternative to noble metals.[49,50] Sakita *et al.* developed a rapid one-step synthesis of CoFe thin films coated with CoFe-LDH, forming a hybrid morphology that shows superior OER performance with low overpotential and Tafel slope.[51] Another study synthesised cobalt-iron metal oxides via microwave-assisted hydrothermal methods, with the best catalyst at Co: Fe = 2:1, achieving a low onset potential of 1.56 V at 10 mA cm^{-2} and stable current density of 130 mA cm^{-2} over 16 hours, outperforming noble metal catalysts in cost-effectiveness and stability. [52] Wang *et al.* synthesised a 3D honeycomb CoFe-selenite catalyst via in situ transformation, exhibiting low overpotential, fast kinetics, and stable current density due to a zeolite-like structure, bimetallic active centres, SeO_3 oxyanions, and hierarchical porosity.[53] Nagappan et al. enhanced CoFe-LDH performance by intercalating halide ions, with iodide delivering the best electrocatalytic activity, an overpotential of 264 mV and a Tafel slope of 56.3 mV dec^{-1}.[54] Recently, a ternary CoFe-LDH composite wrapped in carbon nanotubes demonstrated faster electron transfer and lower Tafel slope, emphasising excellent catalytic efficiency. [55]

Ni and Fe-Based LDHs

The performance of NiFe electrocatalysts is enhanced through structural modifications such as element doping, oxygen vacancies, and incorporation of conductive supports like graphene, which improve active site accessibility, electrical conductivity, and mass and electron transfer.[56,57]

Table-1: Comparative Study of the Electrochemical Performance of Co-Fe-Based LDHS

Catalyst	Electrolyte	Current density (mA cm^{-2})	Overpotential (mV)	Tafel slope (mV/dec)	Stability (h)	Ref.
CoFe-LDH sponge with Cl^-	-	-	286	48	-	[51]

as interlayer						
Co-Fe metal oxide nanocomposites	-	130	-	-	-	[52]
CoFe-based zemannite -type selenite	1 M KOH	10	274	45.6	50	[53]
CoFe-LDHs with intercalated anions	1 M KOH	50	264	56.34	-	[54]
CoFe LDH@CNT	1 M KOH	10	170	42	-	[55]

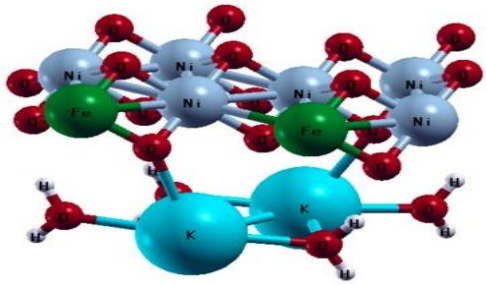

Fig.-3: Structure of NiFe LDH

In 2021, Guo *et al.* synthesised NiFeW LDH, where tungsten boosts iron site efficiency and OER intermediate adsorption, yielding a 3D electrode with excellent overpotential and Tafel slope, ranking among the top OER catalysts.[58] A 2020 study developed a karst-like NiFe LDH directly grown on nickel foil with abundant O_2 defects, showing enhanced OER activity and durability due to better catalyst-substrate binding and conductivity.[59] Qin *et al.* improved NiFe LDH performance by applying a 200 mT magnetic field, enhancing charge transfer and magnetohydrodynamic effects.[60] Dionigi *et al.* revealed that NiFe and CoFe LDHs convert from α- to catalytically active γ-phase under anodic potentials, stabilised by Fe sites' interaction with Ni/Co.[50] A 3D hybrid electrode combining graphene and NiFe LDH showed improved reaction kinetics due to defective graphene oxide promoting electron transfer.[61] Researchers also developed nickel-iron glycerate (NiFeG) structures, with Ni_3Fe_2G/CP exhibiting superior OER performance linked to γ-NiOOH formation as the initial step in OER.[62] Ultrathin NiFeRh-LDH nanosheets grown on Ni foam incorporating rhodium achieved 400 mA cm^{-2} current density at 290 mV overpotential, attributed to electronic structure optimisation enhancing reaction kinetics.[63] Liu *et al.* synthesised amorphous NiFe-LDH with improved active site exposure and OER performance surpassing RuO_2 and crystalline NiFe-LDH due to its unique amorphous phase.[64] Finally, integrating NiFe LDH with the conductive polymer polyaniline (PANI) created an electron-rich catalyst showing superior OER activity, with a small Tafel slope of 44 mV dec^{-1}, low overpotential (220 mV), and excellent stability, thanks to optimised adsorption of reactive oxygen intermediates from strong interfacial interactions. [65]

Table-2: Comparative Study of the Electrochemical Performance of Ni-Fe-Based LDHS

Catalyst	Electrolyte	Current density ($mAcm^{-2}$)	Over-potential (mV)	Tafel slope (mV/dec)	Stability (h)	Ref.
NiFe LDH	KOH (1M)	10	348	-	-	50
NiFeW	KOH (1M)	20	248	68	20	58
NiFe LDH on Ni foil	-	10	272	43	-	59
NiFe LDH	-	-	207	417	-	60
Graphene/NiFe hybrid	KOH (1M)	10	-	109.7	1	61
NiFeGs	KOH (1M)	10	-	320	40	62

NiFeGs/CP	KOH (1M)	100	-	300	40	
NiFeRh-LDH	KOH (1M)	400	290	27	-	63
Amorphous NiFe-LDH	KOH (1M)	10	241	55	-	64
Electron-rich – NiFe LDH	KOH (1M)	10	220	44	-	65

Ni and Co-based LDHs

NiCo-based electrocatalysts are highly effective for water splitting in alkaline environments due to their chemical resilience, high electrical conductivity, and exceptional durability, maintaining performance over thousands of OER cycles. Researchers enhance their efficiency by doping with additional metals or creating oxygen vacancies to expose more active sites and fine-tune their electronic structure.[66] Current focus lies on optimising nanostructures and understanding reaction mechanisms to facilitate large-scale renewable energy applications. Wang *et al.* synthesised NiCo-based LDH and oxide catalysts via co-precipitation and thermal treatment, creating a heterostructure (NiCo–CH) with increased porosity, surface area, metallic concentration, and active sites. This demonstrated improved OER efficiency, with a lower onset potential of 1.51 V, an overpotential of 343 mV at 10 mA cm^{-2}, and a Tafel slope of 66 mV dec^{-1}, outperforming commercial Pt/C.[67] Qian *et al.* developed a NiCo-LDH/NiFe-LDH composite with outstanding OER activity across various aqueous media, including alkaline, seawater, lake water, and wastewater. It exhibited low overpotentials (240–330 mV) and a minimal Tafel slope of 16.6 mV dec^{-1} in 1.0 M KOH, maintaining structural stability over 90 hours.[68] DFT and spectroscopic analyses attribute this performance to efficient electron transfer, an internal electric field, and strong synergistic interactions optimising transition metal redox states. Dhandapani *et al.* enhanced a NiCo-LDH/NF catalyst by doping with Ce^{3+} ions, modifying its microstructure and electronic properties. This resulted in an OER overpotential of 250 mV and a Tafel slope of 98 mV dec^{-1}, with DFT calculations confirming that increased electrical conductance explains improved catalysis.[69] Another study developed CoP@NiCo-LDH dendritic heterosystems via multi-stage growth, outperforming Pt and Ir commercial catalysts in alkaline media with an OER overpotential of 225 mV and a Tafel slope of 46.6 mV dec^{-1}. DFT results indicate favourable charge reorganisation and lower adsorption energy (1.11 eV) at interfaces, enabling tunable catalytic properties. [70]

Table-3: Comparative Study of the Electrochemical Performance of Ni-Co-Based LDHS

Catalyst	Electrolyte	Current density (mA cm^{-2})	Overpotential (mV)	Tafel slope (mV/dec)	Stability (h)	Ref
CO_3^{2-} intercalated NICo LDH	1 M KOH	10	343	66	-	67
NiCo LDH	1 M KOH	50	240	16.6	90	68
Ce^{+3} @ NiCo LDH	1 M KOH	50	250	98	-	69
CoP @ NiCo LDH	1 M KOH	10	225	46.6	14	70

Other LDHs

Of late, wang et al developed a NiCu LDH electrocatalyst modulated with Cu nanoparticles. These systems demonstrated a minimal overpotential of 206 mV and a Tafel slope of 86.9 mV dec^{-1} while maintaining stability for 70 hours at high current densities in unit molar potassium hydroxide solution. XPS studies confirmed greater electronic interactions of nanoparticles with the LDH matrix. DFT studies further revealed that this electronic coupling enhances intrinsic OER performance by improving conductance and adsorption energy of oxygenated intermediate systems. The main innovation of the reported work is the development and characterisation of the catalytic heterostructure interface, a previously unreported design, offering valuable insights for creating vigorous and lasting OER catalysts in an alkali medium.[71] In another study, Liu *et al.* employed a hydrothermal technique for creating two-dimensional NiMn- LDHs on 2D Ti_3C_2 MXene, yielding a hybrid structure characterised by a thin NiMn-LDH layer that develops outward. The resulting NiMn-LDHs/Ti_3C_2-MXene composites (NT-10) demonstrate impressive electrocatalytic activity for OER (overpotential 294 mV and Tafel slope 83.7 mV dec^{-1}). Compared to pure NiMn-LDHs and commercial IrO_2 catalysts, these composites exhibit enhanced

performance due to the effective reduction of the bandgap and alteration of electronic structures facilitated by the interaction between Ti_3C_2 MXene and NiMn-LDHs.[72.]

CONCLUSION

This review highlights recent advances in LDH materials, focusing on their development, synthesis strategies, and electrochemical applications for the OER. Different synthesis approaches, including topological transformations and oriented assembly, have enabled the design of LDHs with better activity and structural stability. Bimetallic LDHs based on transition metals show significant promise as efficient OER electrocatalysts. The catalytic efficiency of these materials is considerably impacted by the electron configuration of transition-metal cations, particularly near one-electron occupancy in the e.g., orbital. In this context, cobalt (II) systems such as CoFe-LDH/carbon composites demonstrate considerable potential for OER electrocatalysis.[73] Similarly, nickel–iron (Ni–Fe) electrocatalysts exhibit excellent activity due to their cost-effectiveness, abundance, and synergistic interaction, where nickel acts as the primary active site while iron modulates the electronic structure to enhance electron transfer and reduce the energy barrier for water splitting. Nickel–cobalt (Ni–Co) LDHs also benefit from a complementary synergy in which cobalt optimises the electronic structure and nickel provides active catalytic sites. [74] The nanosheet structure of LDH catalysts offers a large surface area, improving active site accessibility, ion diffusion, and mass transport. Precise tuning of interlayer spacing can further enhance catalytic efficiency by facilitating better access for electrolytes and reactants. Despite these advances, challenges remain in achieving long-term stability and large-scale practical application. Future research should therefore focus on improving structural stability, electronic conductivity, and scalable synthesis strategies. Overall, the continued development of efficient LDH-based electrocatalysts can contribute to sustainable hydrogen production through water splitting, supporting the goals of the United Nations Sustainable Development Goal 7 and advancing clean energy technologies.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

The present work is related to SDG-7: *Affordable and Clean Energy*. All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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