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Original research article

Dip-Coated Cobalt Oxide Thin Film: A Facile Approach for Oxygen Evolution Reaction

Sharmila Pradhan[1], Mani Ram Kandel[1*], Leela Pradhan Joshi[2], Dinesh Kumar Chaudhary[2], Sheikh Manjura Hoque[3], Chhabi Lal Gnawali[4], Shankar Prasad Shrestha[5*]

[1] Department of Chemistry, Amrit Campus, Tribhuvan University, Kathmandu 44600, Nepal
[2] Department of Physics, Amrit Campus, Tribhuvan University, Kathmandu 44600, Nepal
[3] Material Science Division, Atomic Energy Centre, Dhaka, Bangladesh
[4] Institute of Engineering, Pulchowk Campus, Tribhuvan University, Lalitpur 44700, Nepal
[5] Department of Physics, Patan Multiple Campus, Tribhuvan University, Lalitpur 44700, Nepal

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Abstract: This study presents a low-cost and scalable dip-coating approach for synthesizing nanoparticles of cobalt oxide (Co_3O_4) thin films on stainless-steel substrate and assesses electrochemical performances in alkaline water electrolysis (AWE). Electrochemical assessment via linear sweep voltammetry (LSV) exhibited that the 10L Co_3O_4-coated steel exhibited reduced overpotentials of 258, 350, and 430 mV at current densities of 10, 50, and 100 $mAcm^{-2}$, respectively, compared to uncoated steel, highlighting the influence of film thickness on OER performance. The OER activities of the as-synthesized electrocatalyst are due to the intrinsic electronic structure, promising oxidation states of Co, and catalytic properties. This study brings a cost-effective and controllable synthesis approach for cobalt oxide thin films, presenting insights into the influence of layer deposition on electrocatalytic activity.

Keywords: dip-coating, nanoparticles, linear sweep voltammetry, oxygen evolution reaction, thin film

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शोधसार: यो अध्ययनले स्टेनलेस स्टिल सब्सट्रेटमा कोबाल्ट अक्साइड (Co_3O_4) न्यानोपार्टिकलहरू समाविष्ट थिन फिल्महरू तयार गर्नका लागि कम लागतमा र ठूलो परिमाणमा उपयोग गर्न मिल्ने डिप-कोटिङ प्रविधि प्रस्तुत गर्दछ । साथै यसले उक्त सामग्रीको क्षारीय जल इलेक्ट्रोलाइसिस (AWE) मा इलेक्ट्रोरासायनिक कार्यसम्पादनको मूल्याङ्कन पनि गर्दछ। लिनियर स्वीप भोल्टामेट्री (LSV) मार्फत गरिएको परीक्षणमा १० लिटर कोबाल्ट अक्साइड-लेपित स्टिलले १०, ५०, र १०० मिलिएम्पेएर प्रति वर्ग सेन्टिमिटर धारा घनत्वमा क्रमशः २५८, ३५०, र ४३० मिलिभोल्टको घटाइएको ओभरपोटेन्सियल देखाएको छ, जुन लेप नगरिएको स्टिलको तुलनामा उल्लेखनीय सुधार हो। यसले थिन फिल्मको मोटाइले अक्सिजन इभोलुसन रिएक्सन (OER) कार्यसम्पादनमा महत्वपूर्ण प्रभाव पार्ने कुरा स्पष्ट गर्छ। तयार गरिएको इलेक्ट्रोक्याटालिस्टको OER सक्रियता यसको अन्तर्निहित इलेक्ट्रोनिक संरचना, कोबाल्टको अनुकूल अक्सिडेशन अवस्थाहरू, र उत्कृष्ट उत्प्रेरक गुणहरूका कारण देखिन्छ । समग्रमा, यो अध्ययनले कोबाल्ट अक्साइड थिन फिल्महरू निर्माणका लागि लागत-प्रभावकारी र नियन्त्रणयोग्य संश्लेषण विधि प्रस्तुत गर्दछ । साथै यसले तह-लेपनको इलेक्ट्रोक्याटालिटिक कार्यसम्पादनमा पार्ने प्रभावसम्बन्धी उपयोगी जानकारी पनि प्रदान गर्दछ।

INTRODUCTION

Fossil fuel combustion is a major contributor to air pollution, posing severe environmental challenges [1–3]. As global energy demands continue to rise, transitioning to sustainable energy sources is essential to mitigate ecological damage [4,5]. Renewable alternatives such as solar, wind, and hydrogen energy offer practical solutions, as they produce power with minimal environmental impact [6]. Among these, oxygen and hydrogen are particularly promising energy carriers, capable of meeting the world's growing energy needs. Their production is primarily driven by water-splitting reactions, namely the oxygen evolution reaction (OER) and hydrogen evolution reaction (HER), which are electrochemical processes facilitated by electrocatalysis at the electrode-electrolyte interface [7,8]. However, achieving long-term stability under operational conditions remains a significant challenge, as only a few electrocatalytic materials

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* Corresponding authors, E-mail: mrk.shringa4@gmail.com (MRK), shankarpds@yahoo.com (SPS)

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demonstrate the necessary durability [9]. To improve reaction efficiency, both cathodic and anodic processes typically require catalysis, with noble metals (Pt, Ru, and Ir) and their oxides (PtO_2, IrO_2, and RuO_2) being the most used electrodes [10–12]. Nevertheless, the scarcity and high cost of noble metal-based materials demand the exploration of alternative, efficient, and eco-friendly electrocatalysts based on transition metals [13,14]. Exploring such alternatives opens new avenues for developing cost-effective, high-performance catalysts, which are crucial for advancing hydrogen-based energy systems [4,15,16].

Transition metal oxides (TMO) are promising electrocatalysts due to their excellent mechanical and chemical stability, as well as their high charge transfer capacity, even in amorphous states, highlighting their versatility. Their electronic structure can also be finely tuned, making them ideal candidates for water splitting applications [10,17]. For example, NiO/NF exhibits an overpotential of 310 mV at 10 mA cm^{-2}, while $NiCo_2O_4$ synthesized via the hydrothermal method shows an overpotential of 390 mV under the same conditions [18]. Another study found that mesoporous $NiCo_2O_4$ achieved a lower overpotential of 350 mV at 10 mA cm^{-2} [19]. However, challenges such as stability issues and relatively high overpotentials of transition metal-based monometallic oxides during OER must be addressed through various synthetic strategies [10]. The synthesis methods for metal oxides play a crucial role in determining their morphology and particle size. Various techniques, including the sol-gel method, chemical vapor deposition (CVD), co-precipitation, electrodeposition, and hydrothermal synthesis, have been employed [20]. However, these methods have limitations such as temperature control issues in sol-gel processes, impurity incorporation and low crystallinity in co-precipitation, and hydrolysis-related contamination in hydrothermal synthesis [20,21].

Thin-film deposition offers an alternative and effective approach for synthesizing OER transition metal oxide electrocatalysts, as it enables precise control over film thickness, morphology, and composition, all of which significantly impact catalytic performance and stability [22,23]. This technique allows us to produce uniform, defect-free films with adjustable electronic properties [24]. Furthermore, thin films enhance the active surface area, electrical conductivity, and long-term durability, making them ideal for water-splitting applications [25]. Previous literature has shown the synthesis of various transition metal-based electrocatalysts. For instance, $CoFeO_x$ and CoO_x nanowires synthesized exhibited superior overpotential due to their tuned electronic structure and charge transfer capacitance. Similarly, cobalt oxides and their composites exhibit high reactivity and self-repair capabilities. Additionally, their nano-sized hierarchical structures and doping potential enhance their efficiency and cost-effectiveness as electrocatalysts [25]. Several studies have demonstrated enhanced OER performance of transition metal oxide thin films. For instance, the NiO thin films displayed better OER performance, attributed to their good electrocatalytic behavior [26]. Similarly, spray-assembled nanoscale CoO_x exhibited better OER performance due to their high electrochemically active surface area and favorable electrode kinetics [27]. Wu et al. reported mesostructured Co_3O_4 thin film delivered a low OER potential 340 mV@10 mA cm^{-2}, owing to enhanced electrode-electrolyte interface [28]. In another study, CoO_x thin films fabricated by two-step electrolytic method exhibit an overpotential of 362 mV at 10 mA/cm^2 for OER, further highlighting the effectiveness of tailored thin-film architectures in boosting activity [29]. Although the overpotential reported here is slightly higher than that of the reported value, the simplicity of the preparation technique could undoubtedly reduce the cost of the electrode for OER. Further, cobalt oxide is recognized as an effective electrocatalyst for water splitting due to its unique characteristics, including the formation of heterogeneous interfaces, nanoscale structures, and the dynamic oxidation states of Co^{2+} and Co^{3+} during electrochemical reactions [30,31]. Their nanostructured and porous nature increases the available surface area and improves reaction kinetics [17]. However, challenges such as low electrical conductivity and stability must be addressed through structural modifications, surface treatments, or hybridization with conductive materials. These improvements make Co_3O_4 a promising candidate for efficient and durable OER applications. Therefore, this work focuses on the effect of cobalt oxide layers on water electrolysis.

Herein, a thin film of Co_3O_4 was synthesized using the cost-effective dip coating technique, which was chosen for its low cost, simplicity, and non-hazardous nature. Co_3O_4 nanoparticles are highly effective catalysts for the oxygen evolution reaction (OER) due to their spinel structure, which contains both Co^{2+} and Co^{3+} ions that facilitate electron transfer. During OER, Co^{3+} is converted to Co^{4+}, generating active sites for oxygen formation through a sequence of hydroxide adsorption, oxidation, and molecular oxygen release. Given these characteristics, the as-synthesized electrocatalyst is considered an effective OER electrocatalyst prepared via the dip coating method.

MATERIALS AND METHODS

Chemicals

Cobalt chloride hexahydrate ($CoCl_2.H_2O$, Purity > 99%, Sigma Aldrich) of A. R grade, acetone (99%, Sigma Aldrich), ethanol (99%, Sigma Aldrich), potassium hydroxide (KOH), distilled water was used for the experiment. Stainless steel pieces were brought from the local market.

Precleaning of Stainless-Steel Substrate

Prior to deposition, contaminants such as dirt, grease, and others organic residues were removed from the stainless-steel substrate. Initially, the substrate was immersed in 30 ml acetone and ultrasonicated for 15 minutes to remove organic residues and grease, followed by cleaning with distilled water under sonication for 10 minutes. Next, the cleaned substrate was immersed in 30 ml ethanol and sonicated for 15 minutes to disregard the residual contaminants. After that, the substrate was dried and stored in a dust free chamber at room temperature.

Synthesis of Electrocatalyst Cobalt Oxide (Co_3o_4)

A thin film of cobalt oxide (Co_3O_4) was synthesized using cost-effective dip coating technique. Briefly, 0.1 M cobalt chloride hexahydrate ($CoCl_2.6H_2O$) solution was prepared by dissolving 1.22 g of $CoCl_2.6H_2O$ in distilled water, followed by continuous stirring at 60 °C for 2 hours. Pure stainless-steel substrates (5 × 2.5) cm^2 were employed for thin-film deposition. The dip-coating process was conducted at a withdrawal rate of 50 mm/min, with an immersion time of 1 s and an air-drying period of 1 min. The cycle of dipping, withdrawal, and drying at 100 °C was repeated multiple times to fabricate films with varying thicknesses. Thin films comprising 5, 10, and 15 layers (5L, 10L, and 15L) of Co_3O_4 were synthesized and subsequently subjected to annealing at 300 °C for 2 hours.

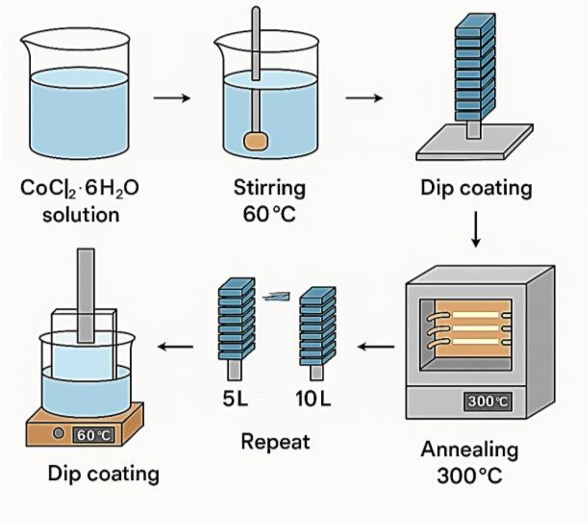
Physicochemical Characterization and Electrochemical Characterization

X-ray diffraction (XRD) analysis was achieved using a Rigaku Ultima IV diffractometer (Rigaku Corporation, Japan) with Cu Kα radiation (λ = 1.5406 Å) to investigate the crystal structure, phase composition, and particle size of OER electrocatalysts, supporting the identification of active phase metal oxides [32,33]. Variations in peak positions and broadening provide information on structural distortions and nanoscale characteristics, which are crucial for electrocatalytic efficacy. Likewise, UV-Vis spectroscopy is utilized to explore the optical properties and electronic structure of the as-synthesized electrocatalysts. By examining absorption spectra and employing Tauc plots, the bandgap energy can be determined, offering insights into charge transfer processes and metal oxidation states. This technique is principally convenient in assessing the light absorption potential of photo-assisted OER electrocatalysts and understanding their electronic interactions and catalytic behaviour.

For electrochemical characterization of the OER, a workstation typically includes an electrochemical setup designed to operate experiments and assess electrocatalytic activities. The core component is a potentiostat which regulates the applied potential or current while recording electrochemical responses, enabling methods such as LSV (34). Electrochemical measurements were conducted using a CORRTEST CS310M electrochemical workstation (Wuhan Corrtest Instruments Corp., Ltd., China). The system employs a three-electrode setup, where the working electrode (WE) consists of the catalyst-coated electrode. The counter electrode (CE) is platinum wire, while the reference electrode (RE) is a stable standard like Hg/HgO to ensure precise potential measurements. The electrochemical cell is operated with a suitable 1M KOH electrolyte at room temperature. This setup enables the reliable evaluation of OER electrocatalysts in terms of their efficiency, durability, and reaction kinetics.

RESULTS AND DISCUSSION

Cobalt oxide thin films were successfully synthesized using a dip-coating method, offering a cost-effective and controllable approach to film deposition (**Scheme 1**). The synthesis involved dissolving cobalt chloride hexahydrate in water to create a uniform precursor solution. During dip coating, the substrate was immersed in the solution, forming a thin layer that underwent gradual hydrolysis and oxidation upon exposure to air. To ensure complete phase transformation, the coated films were annealed at 300°C, promoting the conversion of precursor materials into the spinel Co_3O_4 phase through oxidation. The number of coating cycles (5L, 10L, and 15L) directly influenced the thickness and crystallinity of the films. Based on the controlled dip-coating procedure and consistent solution concentrations, we anticipate that the film thickness increases proportionally with the number of coating layers (i.e., 5L, 10L, and 15L). This relative increase is also supported by the observed trends in electrochemical performance. Thermal treatment further enhanced crystallite formation, improved film adhesion, and eliminated residual solvents. The presence of mixed-valence cobalt ions (Co^{2+}/Co^{3+}) within Co_3O_4 contributes to its electrocatalytic performance, particularly in the OER, by facilitating electron transfer and providing active catalytic sites.



Scheme 1. Schematic diagram of the synthesis of Co_3O_4 electrocatalyst.

Crystallography and Morphological Analysis

The structural properties of cobalt oxide thin films were analysed using the X-ray diffraction technique over a Bragg's angle (2θ) range from 10° to 80°, as displayed in **Figure 1**. The diffraction pattern shows sharp peaks at 43.27° and 74.44°, corresponding to the (400) and (620) crystal planes of Co_3O_4, in agreement with JCPDS file no. 43 – 1003. The XRD results confirm the fruitful formation of the cobalt oxide phase, consistent with earlier reported literature [35,36]. Furthermore, the diffraction pattern reveals the formation of Co_3O_4 thin film with face-centred cubic spinel crystal. An extra peak observed at 50° is attributed to reflection from the steel substrate. The additional peak observed at around 50° is attributed to the underlying stainless-steel substrate, which contributes to the overall diffraction pattern of the coated sample. Since the cobalt oxide thin films were deposited directly on a stainless-steel substrate, certain substrate reflections (especially from Fe or Cr constituents) can appear in the XRD pattern, especially if the film thickness is not sufficient to completely mask substrate contributions.

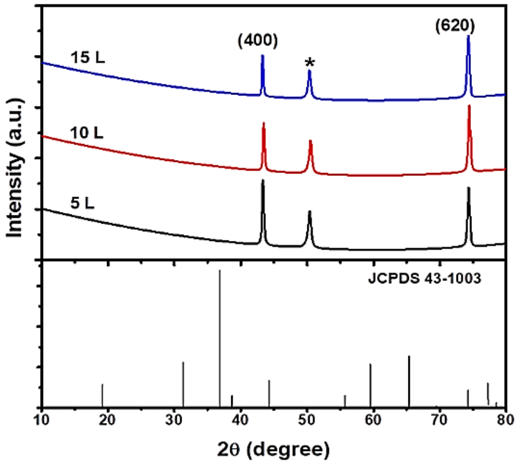


Figure 2. X-ray diffraction pattern of Co_3O_4 electrocatalyst.

Table 1. Structural parameters of thin layers of cobalt oxide Co_3O_4.

Sample (L)	B	Obs d	Rep. d	β	D	Avg. Crys.
	43.36	2.084	2.087	0.285	30.00	
5	74.34	1.28	1.27	0.377	26.48	28.24
10	43.34	2.085	2.02	0.2903	29.46	
	74.44	1.27	1.27	0.4139	24.10	26.78
15	43.30	2.089	2.087	0.2741	31.26	
	74.29	1.27	1.27	0.4493	22.50	26.88

B=Bragg's angle (2θ, degree), obs d=observed d spacing (Å), Rep. d= reported d spacing (Å), β=FWHM (degree), D=Crystallite size(nm), Avg Crys. = Average crystallite size (nm).

The crystallite size of he as fabricated electrode material was determined using Debye Scherrer's equation (37).

$$D = K\lambda/\beta cos\theta \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \text{(1)}$$

where K= 0. 9, λ = 0.154 nm, and β = full width at half maximum (FWHM) obtained from Gaussian fitting of the diffraction peak. **Table 1** displays the observed and stated values of d-spacing along with crystallite size. The calculated average crystallite size of 5, 10 and 15L Co_3O_4 thin film is found to be 28.24, 26.78 and 26.88 nm, respectively. The reduction in crystallite size for 10 and 15 L films proposes an augmented active surface area compared to 5L films, which could be improve the electrocatalytic performance.

UV-Vis Spectroscopy

Optical properties of cobalt oxide thin film were explored using transmittance spectra within the wavelength range 420 to 900 nm. Fig. 2(a) shows the variation of the absorbance of Co_3O_4 film with wavelength. The figure depicts two broad absorbance at wavelengths of 440 nm and 760 nm lying in the visible light region, attributing for the two optical transitions. In general, the edge of the absorbance is determined by the Tauc formula, equation (2)(37).

$$(\alpha h\nu) = A\left(h\nu - E_g\right)^n \ldots\ldots\ldots\ldots\ldots\ldots\ldots \text{(2)}$$

where

α is the absorption coefficient

hʋ - the photon energy

E_g - the optical band gap

A – the constant depending on the effective masses of electrons in the conduction band and holes in the valence band. n- takes the values ½, 2, 3/2, or 3..

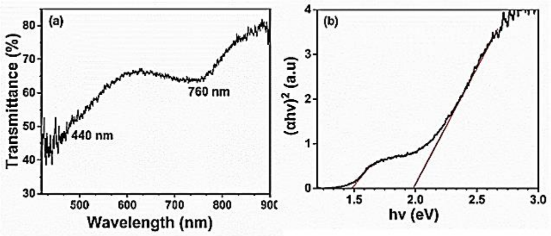


Figure 2. (a) Transmittance vs wavelength of 5L cobalt oxide film prepared by dip coating technique; (b) Tauc plot of the same sample.

It is n=1/2 for direct allowed one, n=2 for indirect allowed, n=3/2 for direct unallowed, and n= 3 for indirect unallowed transition [37]. Fig. 2(b) shows the Tauc plot of $(\alpha h\upsilon)^2$ versus *hʋ* of Co_3O_4 thin films. The extrapolation of the linear portion to zero absorption coefficient gives band gap energy (E_g) of the sample. The plot consists of two straight line regions, designated as A) at low energy side, and B) at higher energy side. The extrapolation of low energy region (A) leads to the intercept at 1.48 eV whereas in region (B), the extrapolation leads to the intercept at 1.98 eV. These intercepts give band gap (E_{g1} and E_{g2}) of deposited films which quite match with that of the literature [38].

ELECTROCATALYTIC CHARACTERIZATION FOR OER

Linear Sweep Voltammetry Analyses

Oxygen Evolution Reaction (OER) of cobalt oxide thin film of different layer was studied using 0.1 M KOH electrolyte. The OER of alkaline water electrolysis was explored with linear sweep voltammetry (LSV) at applied voltage range of 0 V to 0.08 V and scan rate of 10 mV/s. The voltammograms of cobalt oxide thin films of 5, 10 and 15 L, along with uncoated (0 L) steel are presented in **Figure 3.**

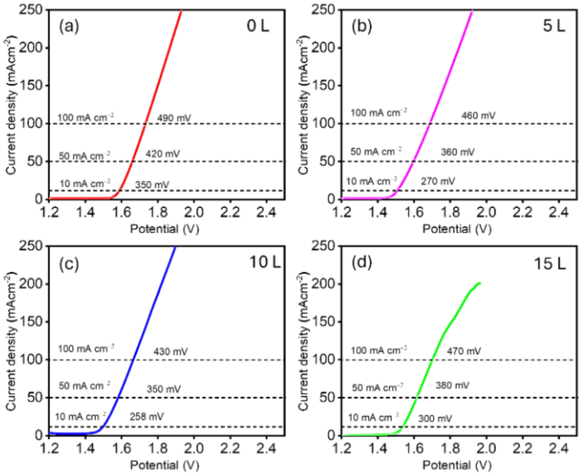


Figure 3. Linear sweep voltammogram of various layered dip-coated cobalt oxide films (a) 0 L, (b) 5 L, (c) 10 L, (d) 15 L measured at 10 mV/s scan rate.

The figure clearly shows that the variation of OER overpotentials of cobalt oxide-coated thin films is lower than that of uncoated steel. It indicates that cobalt oxide-coated thin films evolve oxygen with less energy. E_{RHE} is calculated from the Nernst equation as depicted in equation 3.

$$E_{RHE} \quad = E_{observed} \; + E^0 \text{ (SCE)} + 0.059^* \text{ pH} \ldots\ldots\ldots\ldots.\text{[3]}$$

The overpotential (η) of thin films at 10 mAcm^{-2} current density was calculated using the relation, overpotential, η (V) = E_{RHE} - 1.23

In **Figure 3**, the LSV curves show the variation in OER overpotentials. For the 0 L electrode, the overpotentials are 350, 420, and 490 mV at current densities of 10, 50, and 100 mA cm^{-2}, respectively. In addition, For the 5 L electrode, the overpotentials are 270, 360, and 460 mV at current densities of 10, 50, and 100 mA cm^{-2}, respectively. For the 10 L electrode, the overpotentials are 258, 350, and 430 mV at current densities of 10, 50, and 100 mA cm^{-2}, respectively. For the 15 L electrode, the overpotentials are 300, 380, and 470 mV at current densities of 10, 50, and 100 mA cm^{-2}, respectively. From the as aforementioned electrode and respective OER potentials, it can be observed that the overpotential at current densities 10, 50, and 100 mAcm^{-2} gradually decreased with an increase in the number of layers of cobalt oxide up to 10L, beyond that, it tends to fluctuate.

The variation in overpotentials with increasing cobalt oxide layers can be attributed to changes in active surface area, charge transfer efficiency, and material properties. Up to 10L, the decreasing overpotential suggests improved catalytic performance due to an expansion of the electrochemically active surface area, as supported by XRD findings. This enhancement facilitates better charge transfer and increases the accessibility of active sites, optimizing OER activity. However, beyond 10L, the overpotential begins to fluctuate, likely due to mass transport constraints, higher charge transfer resistance, and structural issues arising from excessive film thickness. A thicker layer may reduce electrical conductivity and limit active site exposure, thereby lowering catalytic efficiency. Additionally, the increase in current density at a fixed potential with more layers indicates enhanced charge transport and surface reactivity up to 10L. Overall, the LSV results suggest that 10L provides the ideal balance between the surface area, conductivity, and charge transfer, making it the most effective for OER.

Conclusions

In this study, cobalt oxide (Co_3O_4) thin films were successfully synthesized using a simple yet effective dip-coating technique. The optical absorbance peaks at 440 nm and 760 nm in the visible light spectrum confirm the formation of Co_3O_4, further validated by the Tauc plot, which revealed two distinct band gaps of 1.48 eV and 1.98 eV, corresponding to direct allowed and forbidden transitions. XRD analysis confirmed the formation of nanostructured Co_3O_4 thin films with well-defined crystallinity. Electrochemical assessment via linear sweep voltammetry (LSV) exhibited that the 10L Co_3O_4-coated steel exhibited reduced overpotentials of 258, 350, and 430 mV at current densities of 10, 50, and 100 mA cm^{-2}, respectively, compared to uncoated steel, highlighting the influence of film thickness on OER performance. LSV also demonstrated that increasing the number of Co_3O_4 layers leads to a significant reduction in overpotential, enhancing OER efficiency. Moreover, a notable increase in current density at a fixed voltage with additional layers indicates improved charge transport and an expanded electrochemically active surface area. These findings underscore the potential of Co_3O_4 thin films as efficient electrocatalysts, highlighting the importance of precise thickness control to optimize performance in energy conversion applications.

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Author Contributions

SP: Methodology, Investigation, Formal analysis, Data curation, Writing-original draft preparation **MRK:** Conceptualization, Writing-original draft preparation, Formal analysis, Data curation **LPJ:** Formal analysis and Writing-review and Editing **DKC:** Formal analysis and Editing **SMH:** Formal analysis and Editing **CLG:** Formal analysis and Editing **SPS:** Conceptualization, Resources, Writing-review and Editing, supervision

Conflicts of Interest

The authors declare no conflict of interest.

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