

Synthesis of Co_3O_4 electrodes by electrochemical deposition for water splitting reaction

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ABSTRACT

Introduction: The use of Ni foam substrates for the growth of catalysts is a common practice in electrochemical water splitting reactions, although their stability in some electrolytes can be problematic, hindering the scalability of synthesis. This study aims to explore alternative substrates for catalyst growth, focusing on cobalt oxide (Co_3O_4) due to its potential in enhancing electrochemical water splitting efficiency. **Methods:** Co_3O_4 was synthesized on various conductive substrates including fluorine-doped tin oxide (FTO), indium-doped tin oxide (ITO), and carbon cloth (CC), employing electrochemical deposition techniques. The morphological and crystalline properties of the Co_3O_4 coatings on these substrates were characterized and analyzed to understand their influence on the catalyst's performance in water splitting reactions. **Results:** The electrochemical deposition resulted in a more condensed coverage of Co_3O_4 on the CC substrate, attributed to the crystal's oriented aggregation. The crystallization and lattice development of Co_3O_4 varied significantly across different substrates, exhibiting high crystallization on FTO and ITO substrates but poorer crystallization on the CC substrate. Notably, the $\text{Co}_3\text{O}_4/\text{CC}$ electrode demonstrated superior performance in hydrogen evolution reaction, achieving the lowest overpotential of -382 mV at a current density of 10 mA cm^{-2} . **Conclusion:** The findings suggest that CC presents a promising alternative to Ni foam substrates for the growth of Co_3O_4 catalysts in electrochemical water splitting applications. The enhanced performance of $\text{Co}_3\text{O}_4/\text{CC}$ electrodes, particularly in terms of overpotential and crystallization behavior, highlights the potential of using CC substrates to improve the efficiency and scalability of water splitting reactions for sustainable hydrogen production.

Key words: Co_3O_4 , electrochemical deposition, water splitting, substrate, carbon cloth

INTRODUCTION

Cobalt oxide (Co_3O_4) has emerged as a prominent catalyst in electrochemical water splitting (EWS) applications, demonstrating significant efficacy in both hydrogen evolution reaction (HER) and oxygen evolution reaction (OER) due to its impressive charge transfer capabilities and large surface area¹⁻⁵. Recently, Liu *et al.* synthesized Co_3O_4 quantum dots and combined them with TiO_2 materials to improve the efficiency of charge transfer between the two materials to increase the water-splitting activity of the material⁶. Similarly, Yuan *et al.* have developed a composite material combining Co_3O_4 with nitrogen-doped carbon, supported on Ni foam, to facilitate comprehensive water-splitting for both HER and OER reactions. Despite Co_3O_4 's efficiency, the use of Ni foam substrates has been noted to pose stability issues in acidic environments, suggesting a preference for basic conditions for optimal water splitting reactions⁷.

Recently, La *et al.* developed $\text{Co}_3\text{O}_4/\text{CC}$ for overall water splitting and demonstrated that this material has an efficient catalytic performance^{8,9}. Furthermore, $\text{Co}_3\text{O}_4/\text{CC}$ also expressed a high ESW performance at a Na_2SO_4 electrolyte that does not react or interfere with most of the target electrode or electrochemical reactions, respectively¹⁰. Moreover, Na_2SO_4 solution is a stable electrolyte over electrodes based on SnO_2 -glass¹¹.

Various methods, including hydrothermal synthesis, sol-gel, electrochemical deposition, and sputtering, have been explored for synthesizing Co_3O_4 on substrates¹²⁻¹⁷. Among these, electrochemical deposition stands out as a straightforward, eco-friendly approach that minimizes chemical use and production time. This method is especially beneficial for fabricating electrodes for EWS reactions, which necessitate the integration of catalysts with conductive substrates to ensure efficient electron transfer. The selection of an appropriate conductive electrode—be it graphite, carbon-based materials like carbon nanotubes (CNTs) or carbon cloth (CC), metal foams or

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meshes, conductive polymers, or transparent conductive oxides such as FTO and ITO—is crucial for optimizing EWS efficiency and paving the way for future practical applications^{18,19}.

This study aims to synthesize Co_3O_4 on various conductive substrates (FTO, ITO, and CC) using electrochemical deposition under low potential and temperature conditions. Characterization of the Co_3O_4 coatings was conducted via X-ray diffraction (XRD) patterns and SEM observations. Furthermore, a Na_2SO_4 solution was used as an electrolyte for the EWS evaluation over catalytic electrodes.

METHODS

Materials

The following chemicals and materials were utilized: Cobalt (II) nitrate hexahydrate ($\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, GHTECH, 98.6%), ethylene glycol (EG, $\text{C}_2\text{H}_6\text{O}_2$, Xilong, > 98%), potassium hydroxide (KOH, Merck, 99%), sulfuric acid (H_2SO_4 , Xilong, > 98%), commercially available carbon cloth (Viet Nam), deionized (DI) water (18 M Ω .cm), and ethanol ($\text{C}_2\text{H}_5\text{OH}$, Thermo Fisher Scientific, 99%).

Electrochemical Deposition Synthesis of Co_3O_4

Initially, 2.91 g of $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ was dissolved in 100 mL of DI water to prepare a 0.1 M $\text{Co}(\text{NO}_3)_2$ solution. Substrates including FTO, ITO, and CC were cleansed using DI water and ethanol through ultrasonication sequentially, followed by oven-drying. Electrodeposition was performed in a standard three-electrode system comprising the conductive electrode (FTO, ITO, or CC), a Pt wire, and an Ag/AgCl electrode (in saturated KCl solution) as the working electrode, counter electrode, and reference electrode, respectively. The procedure was conducted for 5 minutes at a constant voltage of -1.0 V, as previously described⁸. The process resulted in the formation of blue precipitate, presumed to be $\text{Co}(\text{OH})_2$, on the working electrodes. The $\text{Co}(\text{OH})_2/\text{CC}$ obtained was oven-dried and subsequently annealed in air at 400°C for 2 hours to yield Co_3O_4 -decorated electrodes, following optimized parameters from our earlier study⁸.

Characterizations

XRD was employed to ascertain the crystalline phases of the materials, utilizing a Bruker D8 instrument with a $\text{Cu K}\alpha$ radiation source ($\lambda = 1.5406 \text{ \AA}$), an electron accelerating voltage of 45 kV, current of 45 mA, and a scanning rate of 0.02° . Surface morphology was

examined using a JSM-IT500 scanning electron microscope (JEOL), with samples being Au-coated prior to insertion into the measurement chamber. An electron accelerating potential of 20 kV was applied for all SEM imaging. Elemental distribution and quantification were conducted via EDX mapping with the Oxford Instruments.

Linear Sweep Voltammetry Experiments

Linear sweep voltammetry (LSV), a potentiometric method measuring current while linearly scanning potential over time, was utilized to identify oxidation or reduction peaks indicative of HER and OER activities. Co_3O_4 -decorated substrates (1 cm^2 total area) were evaluated in a standard three-electrode system using a Biologic SP-200 potentiostat. LSV was conducted at a scan rate of 10 mV s^{-1} in 1.0 M Na_2SO_4 solution, with Ag/AgCl (saturated KCl) as the reference electrode and Pt wire as the counter electrode. HER polarization curves were recorded from -0.5 V to -1.5 V (vs. Ag/AgCl), and OER polarization curves from -1.0 V to 1.0 V (vs. Ag/AgCl)²⁰.

RESULTS

X-ray Diffraction Analysis

The XRD patterns of Co_3O_4 decorated on various substrates are depicted in Figure 1. The patterns reveal peaks corresponding to the underlying substrates of CC, FTO, and ITO. Additionally, distinct peaks at 2θ values of 19° , 31.2° , 36.8° , 44.8° , 59.3° , and 65.2° are observed, which correlate with the (111), (220), (311), (400), (511), and (440) lattice planes of Co_3O_4 , respectively [JCPDS #80-1532]. Notably, the (440) and (311) planes of Co_3O_4 are prominently featured when deposited onto FTO and ITO substrates, respectively. Conversely, the crystalline intensity of Co_3O_4 on the CC substrate appears to be weaker.

Scanning Electron Microscopy Observations

SEM analysis was conducted to examine the morphology of Co_3O_4 on the substrates, as illustrated in Figures 2, 3 and 4. Figure 2 presents $\text{Co}_3\text{O}_4/\text{ITO}$ with a porous structure distinctly different from the smooth structure of the underlying ITO. Figure 3 highlights the morphology of $\text{Co}_3\text{O}_4/\text{FTO}$, indicating slight variations with non-uniform Co_3O_4 particle sizes. In contrast, Figure 4 shows $\text{Co}_3\text{O}_4/\text{CC}$ densely covered with a uniform layer of Co_3O_4 , approximately 4-5 μm thick, exhibiting a consistent porous structure across the CC substrate.

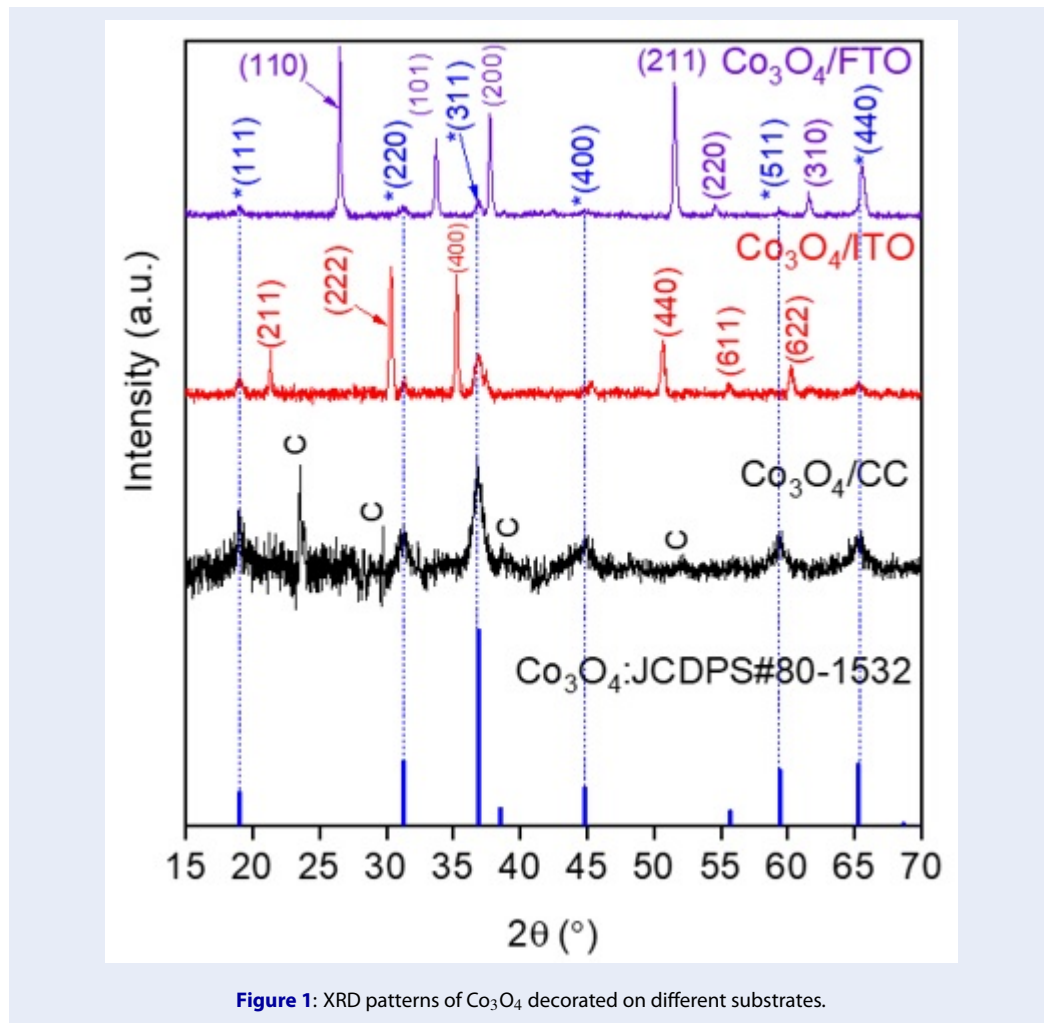


Figure 1: XRD patterns of Co_3O_4 decorated on different substrates.

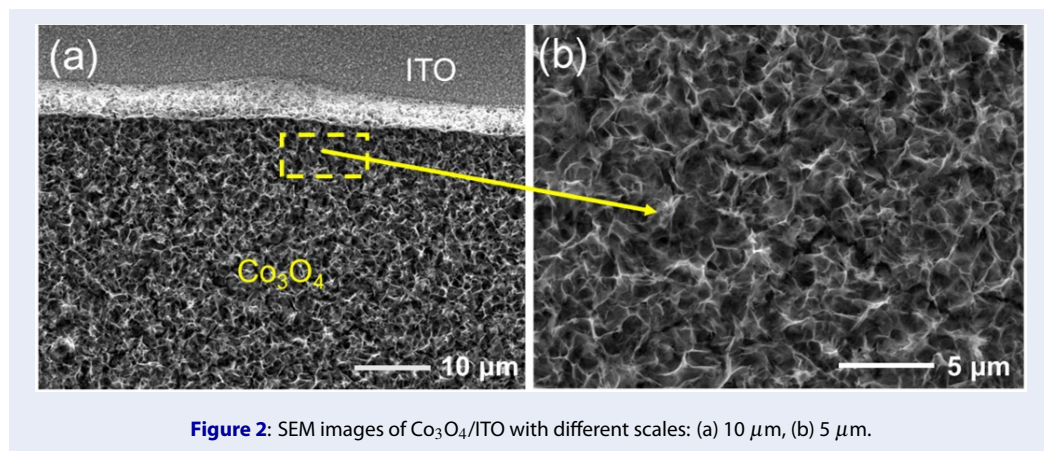


Figure 2: SEM images of $\text{Co}_3\text{O}_4/\text{ITO}$ with different scales: (a) $10\ \mu\text{m}$, (b) $5\ \mu\text{m}$.

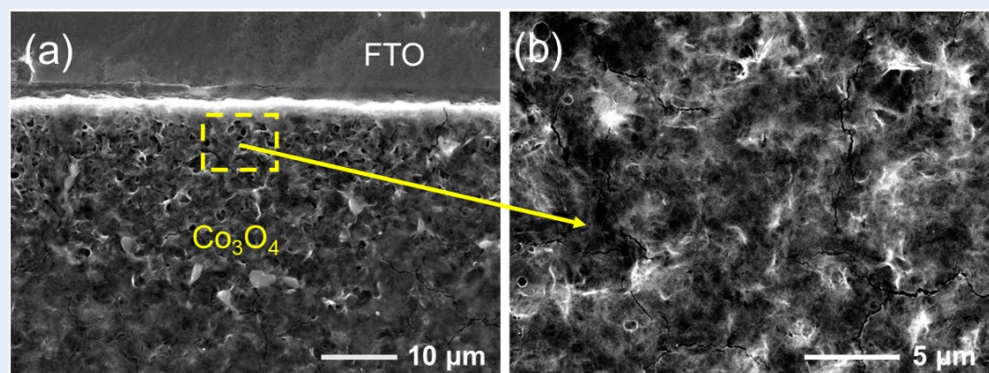


Figure 3: SEM images of $\text{Co}_3\text{O}_4/\text{FTO}$ with different scales: (a) $10\ \mu\text{m}$, (b) $5\ \mu\text{m}$.

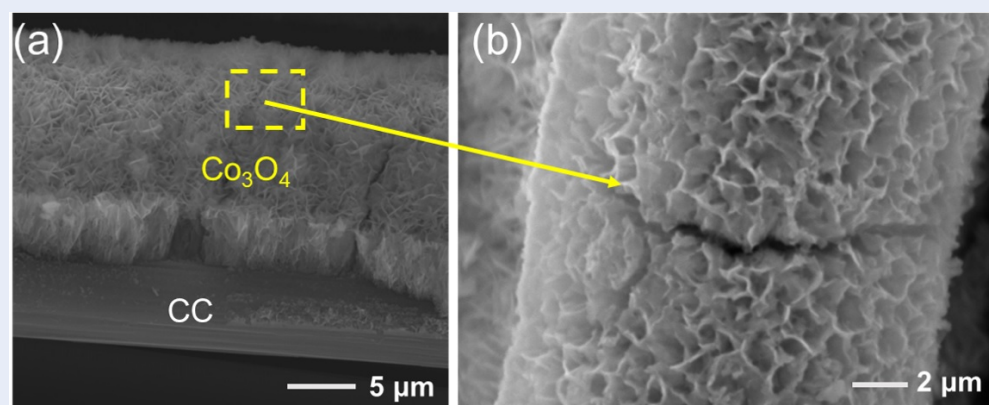


Figure 4: SEM images of $\text{Co}_3\text{O}_4/\text{CC}$ with different scales: (a) $5\ \mu\text{m}$, (b) $5\ \mu\text{m}$.

Linear Sweep Voltammetry Analysis

LSV measurements were utilized to evaluate the EWS activity of Co_3O_4 on various substrates, as depicted in Figure 5. The HER performance, showcased in Figure 5(a), reveals $\text{Co}_3\text{O}_4/\text{CC}$ as the most active electrode, achieving the lowest onset potential of $-382\ \text{mV}$ at a current density of $10\ \text{mA cm}^{-2}$. Comparatively, $\text{Co}_3\text{O}_4/\text{FTO}$ and $\text{Co}_3\text{O}_4/\text{ITO}$ require significantly higher overpotentials of $-975\ \text{mV}$ and $-1610\ \text{mV}$, respectively, to reach $-10\ \text{mA cm}^{-2}$, underscoring the superior HER efficiency of $\text{Co}_3\text{O}_4/\text{CC}$.

OER activities, shown in Figure 5(b), indicate that none of the electrodes—FTO, ITO, or CC—exhibit effective EWS performance for OER, as measured in $1.0\ \text{M Na}_2\text{SO}_4$ at a scan rate of $10\ \text{mV s}^{-1}$. This suggests that while $\text{Co}_3\text{O}_4/\text{CC}$ demonstrates promising HER capabilities, improvements are needed to enhance OER performance across all evaluated substrates.

DISCUSSION

XRD patterns depicted in Figure 1 reveal distinctive crystal structures among the conductive substrates used. Notably, both FTO and ITO exhibit high crystallinity, with predominant orientations at the (110) and (222) planes, respectively. This variance in diffraction peaks can be attributed to the unique crystalline characteristics of each substrate, which in turn influence the nucleation and growth of Co_3O_4 during the electrochemical deposition process. Specifically, the pronounced crystalline growth of Co_3O_4 along the (440) and (311) planes on FTO and ITO substrates, respectively, contrasts with the weaker crystal intensity observed on the CC substrate. However, the presence of both (440) and (311) planes of Co_3O_4 on the CC substrate suggests a nuanced texture development that directly impacts Co_3O_4 's morphology²¹. The observed disparities in Co_3O_4 crystal growth across different substrates might be elucidated by oriented aggregation phenomena, where crystals tend to

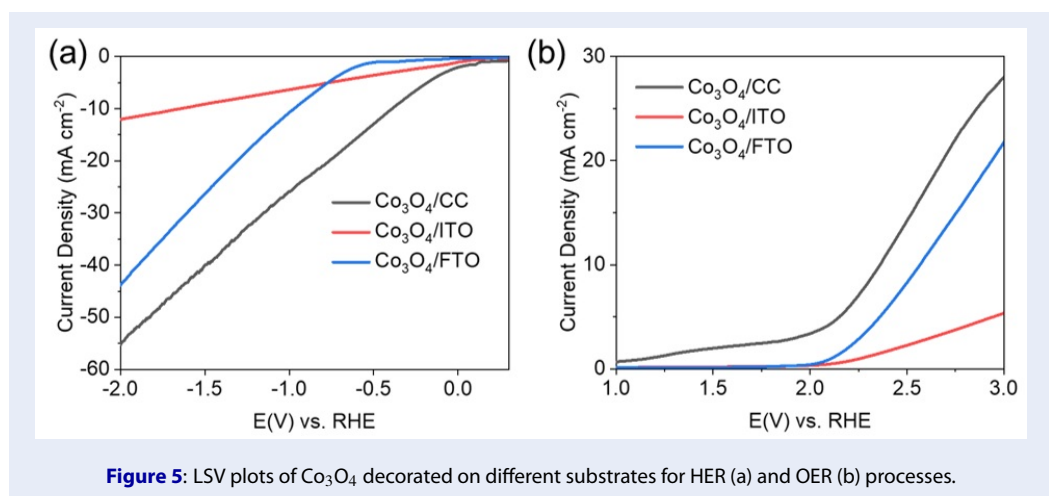


Figure 5: LSV plots of Co₃O₄ decorated on different substrates for HER (a) and OER (b) processes.

cluster around emerging seed sites. This process results in a non-uniform size distribution of Co₃O₄, as evidenced by the SEM images in Figure 4. These images further demonstrate that the CC substrate facilitates a more uniform distribution of Co₃O₄ compared to FTO and ITO substrates, enhancing the HER performance of Co₃O₄.

Moreover, the electrochemical measurements highlighted in Figure 5 and Table 1 indicated that Co₃O₄/CC and Co₃O₄/FTO electrodes require relatively low cell voltages of 1.13 V and 1.34 V, respectively, to achieve a current density of 10 mA cm⁻². This contrasts with the difficulty in determining the necessary voltage for the Co₃O₄/FTO electrode, underscoring the effectiveness of the electrochemical deposition method in preparing Co₃O₄ on various substrates.

The comparative analysis of overpotentials for HER and OER across Co₃O₄-decorated substrates reveals significant variations, possibly due to differences in electrolyte environments. While prior studies predominantly employed 1.0 M KOH, this research utilized 1.0 M Na₂SO₄, showcasing the Co₃O₄/CC electrode's superior activity and lower overpotential in comparison to other electrodes. This suggests that the choice of electrolyte can markedly influence the EWS performance of Co₃O₄ electrode systems, with the Co₃O₄/CC configuration exhibiting enhanced activity at reduced overpotentials.

CONCLUSION

In summary, we have successfully synthesized Co₃O₄ on various substrates, including FTO, ITO, and CC, through an electrochemical deposition, as confirmed by XRD and SEM analyses. The findings reveal that Co₃O₄ exhibits distinct (440) and (311) planes when

deposited on the CC substrate, which significantly influences its morphology. Notably, the Co₃O₄/CC electrode demonstrates superior EWS performance in both HER and OER. Specifically, the Co₃O₄/CC electrode achieved the lowest observed overpotentials of -382 mV for HER and 1130 mV for OER at a consistent current density of 10 mA cm⁻², utilizing a 1.0 M Na₂SO₄ electrolyte. These results underscore the potential of Co₃O₄/CC as a highly effective catalyst for EWS applications, highlighting its promising capacity for energy conversion processes. The study paves the way for further exploration into the optimization of Co₃O₄-based electrodes for sustainable hydrogen and oxygen production.

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COMPETING INTERESTS

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.

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Table 1: Overpotential at 10 mA cm⁻² of Co₃O₄ decorated on different substrates in 1.0 M Na₂SO₄ solution

Material	Substrate	Synthesis methods	HER (mV)	oER (mV)	Electrolyte	Ref.
Co ₃ O ₄	Pt/ SiO ₂ /Si	pulsed laser deposition	263	402	1.0 M KOH	22
Co ₃ O ₄	N doped carbon	heat treatment	237	401	1.0 M KOH	23
Co ₃ O ₄	Nickel foam	hydrothermal	316	520	1.0 M KOH	24
Co ₃ O ₄ clusters	Ag	laser ablation	51	206	1.0 M KOH	25
Co ₃ O ₄ -δ quantum dots	Carbon fiber paper	lithiation/delithiation	-	270	1.0 M KOH	26
Co ₃ O ₄	polypyrrole/MWCNT	mechanical mixing and heat treatment	490	340	1.0 M KOH	27
Co ₃ O ₄	Ti ₃ C ₂ Tx MX-ene@Nickel foam	Electrodeposition	350	520	1.0 M KOH	28
Co ₃ O ₄	CC	Electrodeposition	382	1130	1.0 M Na ₂ SO ₄	This study
Co ₃ O ₄	ITO	Electrodeposition	1610	-	1.0 M Na ₂ SO ₄	This study
Co ₃ O ₄	FTO	Electrodeposition	975	1340	1.0 M Na ₂ SO ₄	This study

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