

CdO/Co₃O₄ NANOCOMPOSITE AS AN EFFICIENT ELECTROCATALYST FOR OXYGEN EVOLUTION REACTION IN ALKALINE MEDIA



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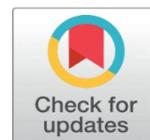
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ABSTRACT

Electrochemical water splitting is one of the promising ways to enhance energy with less outflow. In this regard different electrocatalysts have been reported for Oxygen evolution reaction (OER) to get alternative of noble metal based electrocatalysts. In this work, we have introduced Cadmium-oxide/Cobalt-oxide (CdO/Co₃O₄) nanocomposite by co-precipitation chemical strategy with impressive OER performance in alkaline medium. Almost 310 mV overpotential value is required to achieve 10 mA/cm² current density with Tafel slope value of 62 mV/Dec. The as synthesized nanocomposite has stability of 6h as its longer electrochemical performance.

Keywords: Electrochemistry, Oxygen Evolution Reaction, Electrocatalyst, Cobalt Oxide, Cadmium Oxide

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1. INTRODUCTION

Renewable energy has been playing a vital role since last decade as emerging source of global energy. Electrochemical water splitting is one the green way to get energy with less outflow for growing society [Bhatti et al. \(2021\)](#), [Kang and Kim \(2021\)](#). Electrochemical OER is intensive, critical and productive way for molecular oxygen from water oxidation [Martini and Maia \(2021\)](#). OER requires four electron transfer reaction and high over-potential required for reaction mechanism. That larger over-potential is the major barrier for the researchers to get it at optimum level. Therefore, it is global challenge to reduce the over-potential value to obtain efficient electrocatalyst material for highly active OER performance [Ahamad et al. \(2020\)](#), [Ahmed et al. \(2022\)](#).

Meanwhile, OER activity has been done by several non-precious metal based electrocatalysts such as metal chalcogenide [Majhi and Yadav \(2021\)](#),

metal hydroxide [Kim et al. \(2021\)](#), metal phosphide [Peng et al. \(2018\)](#), nano-carbons [Wang et al. \(2018\)](#) and so on. The over-potential value of various non-precious is not comparable with noble metal-based compound (IrO₂ / RuO₂) [Aftab et al. \(2020\)](#). Among above mentioned transition metals electrocatalysts cobalt oxide is low cost, non-precious active catalyst and has been used broadly [Ibupoto et al. \(2021\)](#). The spinal crystalline structure has electron properties between Co²⁺ and Co³⁺ ions [Lim et al. \(2020\)](#), [Younis and Hou \(2020\)](#). Despite many good features recent research has shown its poor chemisorption and lower catalytic performance. Therefore, to improve its performance by adding other elements have been reported [Chen et al. \(2018\)](#), [Ibupoto et al. \(2021\)](#).

In this work, we have synthesized Cadmium-oxide (CdO) on Cobalt-oxide (Co₃O₄) with simple chemical precipitation method. The as synthesized CdO/Co₃O₄ nanocomposite has revealed promising performance toward OER activity in alkaline environment. For crystalline phase confirmation, functional group study and surface structure analysis XRD, FTIR and SEM techniques were carried out. As previous literature has proved that there is not any reported work of CdO/Co₃O₄ nano-composite for OER in alkaline media. So present work has an encouraging impact for OER performance for various water splitting applications and energy conversion system.

2. EXPERIMENTAL WORK

2.1. CHEMICAL REAGENTS

Cobalt chloride hexahydrate (CoCl₂.6H₂O), Cadmium chloride (CdCl₂), Urea (CH₄N₂O) and Potassium hydroxide (KOH) and Deionized water (D.I) were received from Sigma Aldrich, Karachi.

2.2. SYNTHESIS OF NANOCOMPOSITE MATERIAL

CdO/Co₃O₄ nanocomposites were synthesized by precipitation method by taking 2.37 gm of CoCl₂.6H₂O, 0.6 gm of CH₄N₂O with varying ratio of CdCl₂ as 0.02 gm (sample A) and 0.04 gm (sample B) into 100 ml of deionized water separately. Precursor Solution containing beakers were covered by aluminum foil and placed into an electric oven for 6 hours at 95° C and then precursor solution was cooled at room temperature. As received solutions of Co₃O₄ and CdO/Co₃O₄ were washed several times with D.I water and dried. As received precipitates of hydroxide product were converted into oxide by placing in muffle furnace for calcination at 500° C for 6 hours to get final product.

2.3. PHYSICAL CHARACTERIZATION

The crystalline structure and phase purity was identified by x-ray diffraction (XRD) of model BURKER D8, operating at 40 mA and 40 KV by using Cu K α radiation (λ =1.5418). Scanning electron microscopy (SEM) of model JSM-6380L JEOL was used for morphology of as composed various nanomaterials. The study of chemical bonding between Co₃O₄ and CdO was studied by Fourier transform Infra-Red (FT-IR).

2.4. ELECTROCHEMICAL MEASUREMENTS

Electrochemical measurements of different electrocatalysts were carried out through linear sweep voltammetry (LSV), cyclic voltammetry (CV), chronopotentiometry, and electrochemical impedance spectroscopy (EIS) in 1.0 M KOH solution. All experiments were done by using three electrode configurations,

the modified glassy carbon electrode (GCE) as the working electrode, a silver-silver chloride (Ag/AgCl) as a reference electrode and a platinum wire as counter electrode. To obtain a homogenous catalyst solution for further tests, 10 mg of each catalyst was dispersed into 2 ml of D.I water and 0.2 ml of nafion solution was added as binder. Then the solution was kept on an ultrasonic bath for 20 to 30 minutes. 10 μ l of each catalyst suspension was dropped on cleaned GCE with a diameter of 3 mm by drop-casting method and dried in the air. The loaded mass of catalyst on the GCE was about 0.2 mg. For OER analysis, initially LSV was adopted via 0 to 0.7V vs RHE on potentiostat machine an instrument by VERSA STAT-4. EIS was applied at frequency range from 100 kHz to 0.1 Hz and amplitude of 10 mV vs RHE. Z-view software was used to analyze EIS data by the help of an open equivalent circuit. To measure capacitance and active surface area, CV was performed at a scan rate of 10 mV/s at different scan rates as 30, 50,70 and 90 mV/sec. All the potentials are reported into manuscript are of vs RHE by following the Nernst equation:

$$E_{RHE} = E_{Ag/AgCl} + 0.059pH + E^{\circ}_{Ag/AgCl} \quad (1)$$

3. RESULTS AND DISCUSSION

Different physical tests were carried out for various nanocomposites. Initially, scanning electron microscopy (SEM) was executed on pristine Co_3O_4 and as synthesized sample A and sample B. [Figure 1\(a\)](#) has shown the SEM image of Pristine Co_3O_4 which has nano-wire like structure [Liu et al. \(2021\)](#). Meanwhile, the varying concentration of CdO into sample A and sample B has been analyzed as depicted in [Figure 1 \(a\), \(b\)](#). Both images have different morphologies with mixture of nano wires and particles as well. The addition of CdO has reduced the intensity of nano wires and enhanced the activity of pristine Co_3O_4 with more active sites [Zhang et al. \(2021\)](#). Both structures within one nanocomposite have been analyzed through SEM as successful synthesis of CdO/ Co_3O_4 nanocomposite.

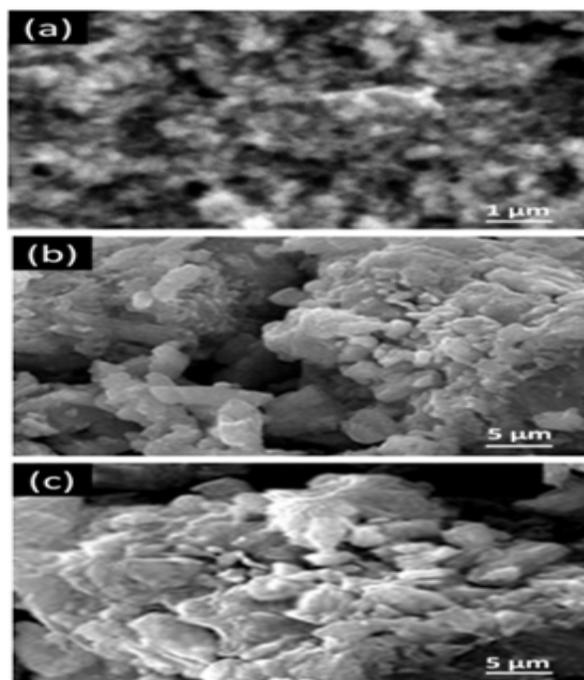


Figure 1 SEM images of various electrocatalysts: (a) Pristine Co_3O_4 (b) Sample A and (c) Sample B

The X-ray diffraction (XRD) was applied for crystallographic study. The pristine Co₃O₄ has revealed well acute patterns of diffraction as depicted in Figure 2(a). Pristine Co₃O₄ has well organized patterns around 31.4°, 36.9°, 38.1°, 44.95°, 55.60°, 59.7° and 65.2° with corresponding reflection of (111), (220), (311), (222), (400), (511) and (440) which are matched with reference card: (01-080-1536) along with cubic phase crystal structure Tian et al. (2022). By increasing the concentration of CdO increase in crystallinity which is evidence in decrease in intensity of the pristine Co₃O₄ diffraction peaks for sample A and small shift of 2 thetas also which meets reference card: (00-005-0640) as shown in Figure 2(b). However, in Figure 2(c) sample B has revealed same patterns with more intensity, which further enhanced the peaks due addition of CdO in increased quantity as result of deformation in structure of Co₃O₄ by doping CdO Gharib and Arab (2021). The XRD JCPDS study was done through High score plus software.

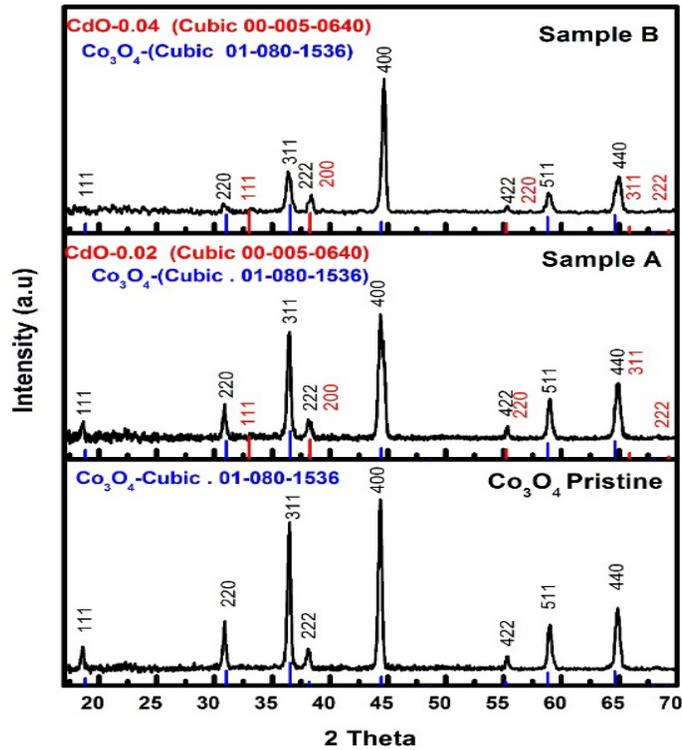


Figure 2 XRD patterns of Co3O4 Pristine, Sample A and Sample B

The Fourier transform infra-red (FT-IR) has been applied for chemical analysis of as prepared nanocomposites. FT-IR has showed spectra of pristine Co₃O₄ and CdO/Co₃O₄ nanocomposites as given in Figure 3. The peaks at 1625 cm⁻¹, 1400 cm⁻¹ and 3600 cm⁻¹ in the spectrum of pristine Co₃O₄ are of molecular water, Co₂ and OH group due to its high surface to volume ratio. Important band of peak at 563 and 444 cm⁻¹ are of metal oxide such as Co₃O₄ and CdO, which has been confirmed from reported literature Rubin and Li (2019). In general, the peaks of CdO introduced have not displayed any additional band, except the small offset of positioning of peaks because of the difference between ionic rays of different metals.

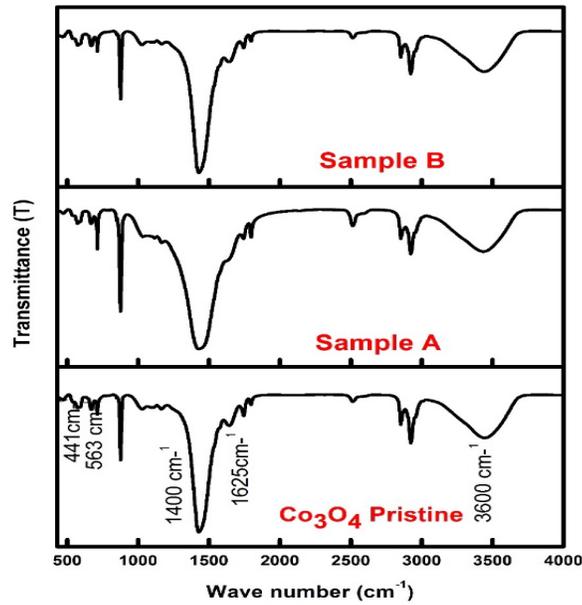


Figure 3 FT-IR spectra of Co₃O₄, Sample A and Sample B

The oxygen evolution reaction (OER) performance of prepared samples was done in 1 M KOH, using linear sweep voltammetry (LSV) at the scan rate of 5 mV/s as depicted in Figure 4. The OER of RuO₂ is taken from previous literature (1.42 V) which have been used for comparative analysis of as received nanocomposites. Through Figure 4(b), it has been analyzed that pristine Co₃O₄ has high over-potential value of 1.62 V at current density of 10 mA/cm² for its OER performance. Whereas, successive addition of CdO in Co₃O₄ up to optimized quantity to 0.04 gm as sample B has revealed low over-potential of 1.54 V to reach at current density value of 10 mA/cm² which is higher than sample A and pristine Co₃O₄ respectively. Sample B has revealed outstanding OER activity except of RuO₂ than others, which has been compared to previous literature work as shown in Table 1.

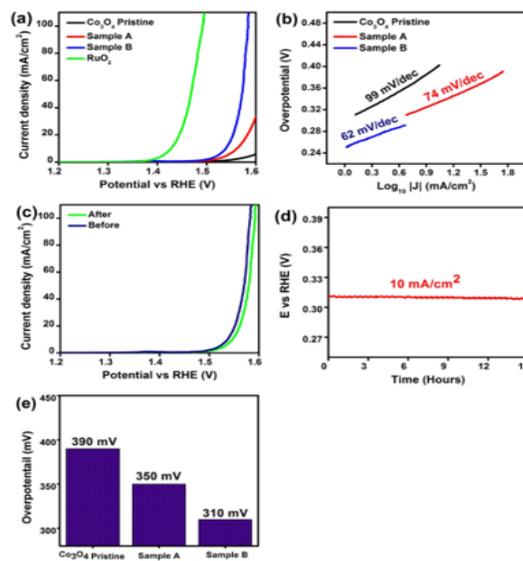


Figure 4 (a) LSV curves of Pristine Co₃O₄, Sample A, Sample B and RuO₂ (b) Corresponding Tafel plots (c) Durability test of Sample B through LSV (d) Chronopotentiometry of Sample B for 15h at 10 mA/cm² (e) Overpotential values given in histogram

As Tafel slope determination, the kinetics of reaction means rate determining step for electrocatalyst behavior. The general rule for Tafel slope is that low value means high rate of reaction. Therefore, as prepared sample B has lower Tafel slope value of 62 mV/dec, which indicates that it has favorable OER kinetics and can be used as a possible candidate for the water oxidation as shown in Figure 4 (b). The durability of an electrocatalyst is an essential parameter for its long-term performance. The durability of sample B was checked before and after chronopotentiometric examination as depicted in Figure 4 (c). While, chronopotentiometry test was done on Sample B at 10 mA/cm² current density for 15h as shown in Figure 4(d). Sample B has shown its stable behavior without any loss of potential value. The overpotential values of various electrocatalysts have been shown in Figure 4(e).

Electrochemical active surface area (ECSA) and electrochemical double-layer capacitance (C_{dl}) was determined by cyclic voltammetry (CV) for various nanocomposites at different scan rates of 30, 50, 70 and 90 mV/s as shown in Figure 5 (a), (b) and (c). The ECSA is measured by CV through linear fitting of mean current density vs scan rate Younis and Hou (2020). The formula is given below for calculation of the ECSA through C_{dl} values;

$$ECSA = C_{dl}/C_s. \quad (2)$$

In this equation C_s is 0.04 mF/cm² for KOH solution Tong et al. (2020). The sample B nanocomposite have higher value of C_{dl} as 16.8 μ F/cm² and ECSA was calculated as 420 cm² which is higher than others as depicted in Figure 5(d). Active edges of catalyst materials are responsible for the adsorption of OH and O species reported in literature Su et al. (2019). In present work sample B has superior OER attempt because of evidence of large ECSA value.

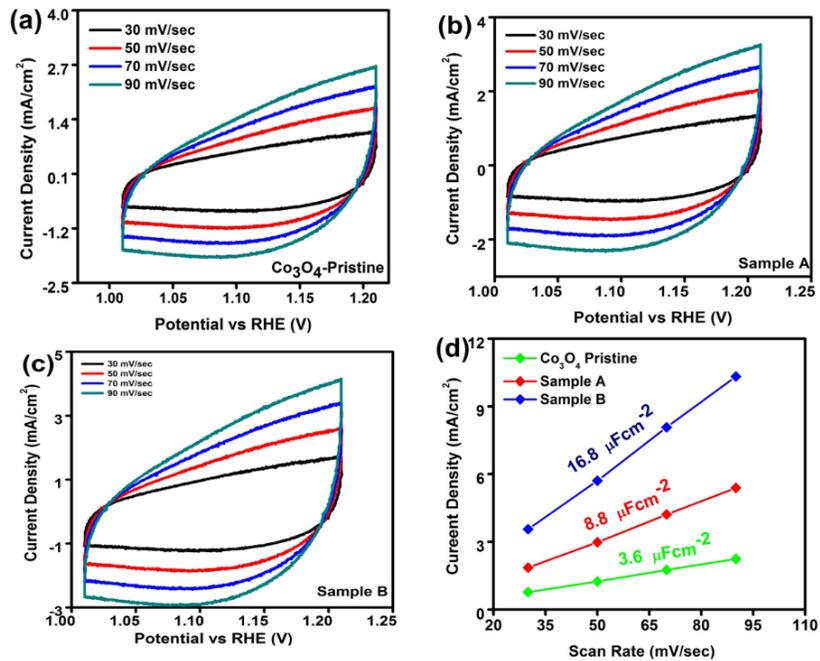


Figure 5 CV curves at different scan rates of 30, 50, 70 and 90 mV/sec (a) Pristine Co₃O₄, (b) Sample A, (c) Sample B and (d) C_{dl} values of various nanocomposites by linear fitting of current density vs scan rates through CV curves

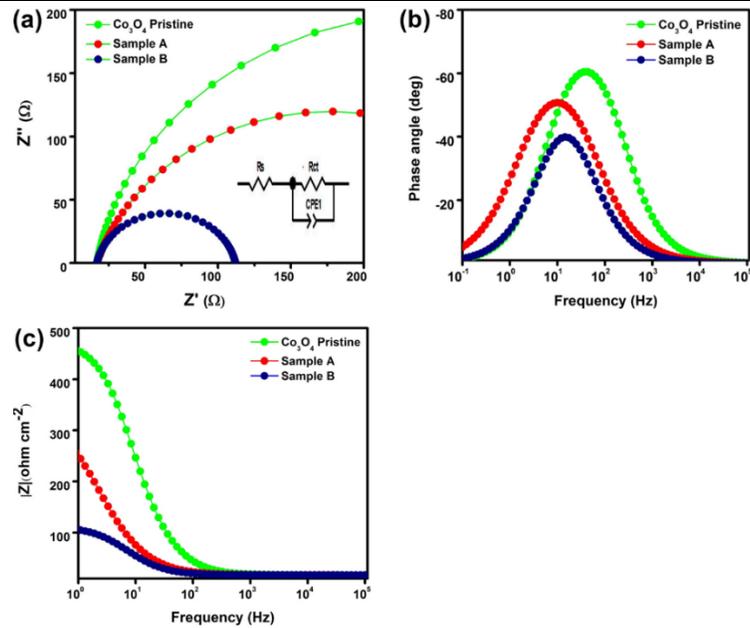


Figure 6 EIS results of as prepared electrocatalysts (a) Nyquist plots, (b) Bode plot (i) and (c) Bode plot (ii)

Additionally, electrochemical impedance spectroscopy (EIS) was done to verify the charge transfer resistance (R_{ct}) as shown in Figure 6. In Figure 6 (a), the Nyquist plots are shown, in which half circle has low radius of sample B and Figure 6(b) and (c) has revealed bode (i) and bode (ii) plots respectively to support Nyquist plots respectively. EIS study has exhibited that sample B has the small charge transfer resistance (R_{ct}) value of 95Ω . Meanwhile, we can say that fast charge transportation and higher capacitance value as 0.4 mF . Sample B has fast charge transport, which is evidence for rapid charge transfer during electrolyte and electrode reaction, which favors the superior OER activity.

Herein, Table 1 described comparative study of previous reported electrocatalysts for OER kinetics for water splitting applications. From literature point of view, it has been observed that Sample B has over all promising electrochemical response for OER to compete other non-noble metal based electrocatalysts.

Table 1 Comparative study of as prepared nanocomposite with previous reported electrocatalysts

Electrocatalyst	Electrolyte	Current Density	Overpotential	Tafel Slope	Ref.
CdO-Co ₃ O ₄	1M KOH	10 mA cm ⁻²	310 mV	62 mV dec ⁻¹	This work
Ni/NiS/NC	1 M KOH	10 mA cm ⁻²	337 mV	52 mV dec ⁻¹	Ding et al. (2019)
Co _{0.85} Se/HPG	0.1 M KOH	10 mA cm ⁻²	385 mV	61.7 mV dec ⁻¹	Zhong et al. (2019)
CoOx-N-C/TiO ₂ C	1 M KOH	10 mA cm ⁻²	350 mV	75 mV dec ⁻¹	He et al. (2019)
MWCNT-CuO-400	1 M KOH	10 mA cm ⁻²	420 mV	99 mv dec ⁻¹	Hou et al. (2016)
Fe (OH) ₃ : Cu (OH) ₂	1 M KOH	10 mA cm ⁻²	365 mV	42 mv dec ⁻¹	Cheng et al. (2015)

Table 2 Some electrochemical features of as synthesized electrocatalyst

Catalyst	Calculated from LSV	Calculated from EIS		Calculated from CV	
	Tafel Slope	Charge Transfer Resistance	Double Layer Capacitance	Double Layer Capacitance	Electrochemical active surface area
	<i>B</i>	<i>R_{ct}</i>	<i>CPE_{cdl}</i>	<i>C_{dl}</i>	<i>ECSA</i>
	<i>mV/dec</i>	Ω	<i>mf</i>	($\mu\text{F}/\text{cm}^2$)	cm^2
Co ₃ O ₄ Pristine	99	450	0.03	3.6	90
Sample A	74	320	0.31	8.8	220
Sample B	62	95	0.4	16.8	420

4. CONCLUSION

In conclusion, for CdO/Co₃O₄ nanocomposite two-step co-precipitation methods were used and analyzed OER activity. The over-potential and Tafel slope of optimized sample B (CdO 0.04 gm) has lower value as 310 mV and 62 mV/dec respectively at current density value of 10 mA/cm². The ESCA and C_{dl} value of sample B has larger value of 420 cm² and 16.8 $\mu\text{F}/\text{cm}^2$. EIS experiment has shown that CdO/Co₃O₄ nanocomposite has small R_{ct} value of 95 Ω and improved C_{dl} value as 0.4 mF. This simple experiment adds the one step for CdO/Co₃O₄ electrocatalyst as progressive one for effective electrochemical water-splitting applications.

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