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Electrodeposition And Characterization of Ni-Mo Based Electrocatalyst for Efficient Hydrogen Production Via Alkaline Water Electrolysis

Rana Zubair Anwar

Department of Chemistry, University of Agriculture Faisalabad, Pakistan

Abstract

Using the electrodeposition method, this study investigates the efficacy of an alloy based on nickel and molybdenum as an electrode in water splitting applications. A copper substrate with a current density (c.d.) between 1.00 and 4.00 A dm^{-2} was coated with nanocrystalline nickel-molybdenum alloy, and the deposit properties and electrocatalytic behavior in a 1.00 M KOH solution were examined. The electrocatalytic behavior of the coatings in relation to their oxygen evolution reaction (OER) and hydrogen evolution reaction (HER) was assessed using electrochemical methods including cyclic voltammetry (CV) and chronopotentiometry (CP). According to the experimental results, the maximum electrocatalytic potential for HER and OER is found in nickel-molybdenum alloys electrodeposited at 1.00 A dm^{-2} (38.30 wt% of molybdenum) and 4.00 A dm^{-2} (33.20 wt% of molybdenum). When compared to other coatings, nickel-molybdenum alloys coated at 4.00 A dm^{-2} exhibit the greatest corrosion resistance in an alkaline medium. According to SEM and XRD, the greatest electrocatalytic activity of nickel-molybdenum alloys deposits for both OER and HER, depending on electrodeposition c.d. value, were due to their chemical constitution (in terms of nickel and molybdenum concentrations), structures, and surface structure.

Keywords: Nickle-Molybdenum alloys, Hydrogen production, Alkaline water electrolysis, HER and OER

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1. Introduction

A major challenge in modern times is tackling environmental sustainability and climate change while meeting the growing need for energy. The urgent need for clean and renewable energy sources arises from the accelerated consumption of fossil fuels and increasing environmental problems [1]. Hydrogen is regarded as one of the green fuels due to its high gravimetric energy density and lack of emissions [2]. Electrocatalytic water splitting is a sustainable as well as eco-friendly method of producing superior quality hydrogen. Literature reviews reveal that researchers have focused on improving the effectiveness of catalysts employed in electrochemical splitting of water for enhancing hydrogen production [3]. The most effective electrocatalysts during oxygen evolution reactions (OER), in particular ruthenium- or iridium-based complexes, and hydrogen evolution reactions (HER), especially platinum-based electrodes, remain dependent on rare metals [4]. These materials are unsuitable for widespread application due to their rarity and elevated costs. In common water-splitting reactions and procedures, highly efficient electrocatalytic compounds derived from earth are strongly pursued as replacements for noble metal-based catalysts [5]. To do this, numerous efforts have been made over years to create and improve electrocatalysts based on non-noble metals. However, outcomes are significantly less effective than catalysts based on noble metals. Thus, it is still necessary to develop more efficient electrocatalysts using elements which are easily available on Earth.

Transition metal-based compounds have shown significant potential as catalysts in hydrogen evolution (HER) [6]. In particular, Nickel-Molybdenum (Ni-Mo) catalysts have attracted people's interest mainly because of its unique catalytic properties, stability, and accessibility [7]. In this context, numerous significant advancements have been made with Ni-based bifunctional catalysts. Studies reveal that Ni-Mo binary metal catalysts offer the best HER catalytic efficiency among all other bimetallic-based compounds. Ni-Mo binary metal catalysts are more effective because of their intrinsic electrochemical behavior, surface interaction, and processes to enhance hydrogen evolution reactions [8]. Particular difficulties in synthesizing nickel-molybdenum catalytic compounds for hydrogen production involve high precursor costs, difficulty with controlling synthesis parameters, developing undesirable phases, catalyst aggregation and deactivation, and scaling up for industrial applications [9]. To address these limitations, this study synthesizes Ni-Mo catalysts in solid state medium, offering a thorough characterization of the catalyst with an emphasis on its morphological and compositional analysis using XRD and

SEM, respectively. The main objective of the current study is to improve the efficiency and cost-effectiveness of nickel-molybdenum catalyst production, possibly leading to advances in hydrogen generation.

2. Alkaline Water Electrolysis (AWE)

2.1. Basic Principle

The OER and the HER are the two half-cell processes involved in electrochemical water splitting [10]. These reactions occur at the anode and cathode centers that are generally divided by an ion exchange membrane. Both HER and OER undergo reactions differently based on the pH of the electrolyte and other factors like temperature, pressure, electrode type, and cell design [11]. During alkaline conditions, the hydroxide ions produced by water at the cathode serve as carriers of charges. These hydroxyl anions are oxidized at the anode site to produce O_2 and electrons, that are then utilized at the cathode site to produce H_2 [12]. In accordance with Faraday's law of electrolysis, the electrochemical reactions take place at a junction between the surface of the electrode and the electrolyte, and the rate at which gas is produced is exactly corresponding to the current flowing through the electric circuit [13]. Water splitting is an uphill process that requires an energy supply of $\Delta G = 237.1$ kJ/mol and a thermodynamics potential of 1.23 V for electrolysis [14]. In practice, overcoming a number of obstacles requires a significantly higher applied voltage, known as the overpotential (the difference between the applied potential and the theoretical value of 1.23 V) [15]. These include a circuit's electrical resistance, electrochemical reactions' activation energies, gas bubble-related obstacles that increase barrier to ionic transfer and electrochemical reactions, and mass transportation, particularly because of the slow OER [16]. To make the procedure more economical, the overpotentials for each of the half reactions needs to be reduced. Electrocatalysts that can lower the activation energy and energy input can be used to accomplish this.

2.2. Preparation of Ni-Mo Catalyst

The Ni-Mo alloy was produced by reductive annealing or electric heating, which is based on bottom-up synthesis of porous [17]. The evolution of hydrogen serves as a basis for this strategy. This process involved blending 99.0% pure nickel (II) nitrate hexahydrate with 99.0% pure ammonium molybdate in a one on one ratio. A finely ground powder was created by the mixture. Once the powder was fine, it was heated for about 25 to 30 minutes at temperatures between 950°C and 1000°C in a fuming hood [18]. The absence of this yellow odors and green color indicated that the reaction was successfully completed [19]. Figure 1 represents an overview of the multiple stages involved in the production of a solid-state Ni-Mo catalyst.

2.3. Electrodeposition of Ni-Mo Alloy

The nickel-molybdenum electrochemical bath was made with analytical grade reagents and double distilled water. At this point, tri-sodium citrate ($Na_3C_6H_5O_7$) was utilized as a complexing salt and sodium molybdate (Na_2MoO_4) and nickel sulphate ($NiSO_4 \cdot 6H_2O$) as sources of metallic salts [20]. The conventional Hull cell approach was used to determine the ideal bath constitution and operating conditions for depositing of hard adhesive nickel-molybdenum alloy coating [21]. According to what was required, dil. NH_4OH or H_2SO_4 were used to keep the bath's pH at 9.5. Prior to each deposition, the electrolyte was run through Whatmann-40 filter paper to eliminate any suspended anodic impurities along with insoluble molecules [22]. Table 1 shows the optimal bath composition and operation conditions. Nickel-molybdenum coatings were coated on plates of copper as well as a surface of a copper rod (both with identical specification), primarily depending on the characterization requirements [23]. Complete electrocatalytic and electrochemical properties of nickel-molybdenum alloy coatings were studied by applying them to a copper rod's cross-sectional surface area (1.00 cm^2) in a specially designed optical cell [24]. To examine their respective performance, electrodepositions were performed at various c.d. (1.00-4.00 A dm^{-2}) for a fixed time (600.0 s). Following the electrodeposition, coatings were repeatedly washed using distilled water, cured in hot air, and ultimately dried out until additional examination [25].

2.4. Experimental Configuration

A specially designed three-electrode tube-shaped optical cell was used for the electrocatalytic study of nickel-molybdenum alloy [26]. A quantitative assessment of the electrocatalytic behavior of nickel-molybdenum alloy electrodeposited at different c.d. values was made possible by this experimental design. When the electrode is anodic or cathodic polarized, respectively, the specially made cell gathers H_2 and O_2 gas [27]. At one end of tube, a deposited nickel-molybdenum alloy serves as the working electrode, while a platinum electrode with a surface area of 1.0 cm^2 serves as counter electrode. The cell has graduated tubes (burette) that measure amount of easily measurable released gas (oxygen and hydrogen) that displaces the solution [28]. By tracking amount of O_2 and H_2 generated at the cathode and anode, correspondingly, this setup assists in analyzing electrocatalytic effectiveness of nickel-molybdenum alloy Figure 2.

3. Characterization of Ni-Mo Coatings

Nickel-molybdenum alloy coatings were electrodeposited at various current densities (1.0-4.0 A dm^{-2}) using the optimum bath [29]. Despite being a highly electroactive metal, molybdenum (Mo) can only be plated on alongside of another metal using induced co-deposition; it cannot be directly plated from its electrolytic bath alone [30]. As a consequence, nickel, also known as the inducing metal, promotes the deposition process during the deposition of nickel-molybdenum alloy, while molybdenum is referred to as the reluctant metal. The composition of reluctant metal in coating with c.d. is usually found to show no consistent trend in induced co deposition, indicating that variations in the characteristics of electrodeposited nickel-molybdenum alloy coatings with c.d. are highly unreliable [31]. Thus, in current investigation, nickel-molybdenum coatings were deposited at varied c.d. values and were initially evaluated for their fundamental features, such as shape, composition, corrosion resistance and phase structure [32].

3.1. Surface Analysis

The coatings that were plated at 1.00 A dm^{-2} and 2.00 A dm^{-2} were discovered to have a nodular morphology [33]. The surface became smoother and more uniform as the c.d. is elevated because the nodules grew in size but decreased

in quantity. Additionally, several pin holes were seen in nickel-molybdenum alloy coatings plated with elevated c.d., which were related to the formation of hydrogen during co deposition [34]. Therefore, it may be deduced from SEM micrographs of nickel-molybdenum alloy coatings that the surface morphology of the coating is closely related to the applied c.d. It should be noticed that the weight percentage (wt%) of Mo increases with decreasing c.d. As a result, the surface has a granular, nodular structure. As will be mentioned, the higher electrocatalytic activity of the coatings for OER is caused by this elevated nickel concentration at high c.d. range [35].

3.2. XRD Analysis

The phase and crystallite size of the Nickle-Molybdenum alloy coatings at various current densities were determined using XRD approach. The XRD peak values at 2θ = 22.30°, 43.30°, 50.40°, 74.030°, and 89.80° reveal the tetragonal $MoNi_4$ phase, matching to the planes (110.0), (211.0), (130.0), (420,0), and (501.0), respectively, which are in better accordance with the normal morphology of $MoNi_4$ [36]. A steady rise in peak intensity alongside c.d. certainly shows that deposition c.d. has a major impact on the chemical makeup and structural characteristics of the coatings [37]. The Scherrer equation has been used to find out about the particle size of the electroplated nickel-molybdenum alloy coatings at various c.d.

$$d = 0.9\,\lambda/\beta cos\theta$$

In the above equation, d shows the size of the crystallite, λ represents the wavelength of X-rays, β shows the complete breadth at the half height of diffraction peak, and θ indicates Bragg angle. The normal crystallite size for coatings coated across a broad range of c.d. was 22.14 nm [38].

4. Electrocatalytic Study

Material's electrocatalytic behavior can be evaluated based on their abilities to catalyze the HER and OER processes, which are crucial for electrolysis of water and fuel cell applications [39]. When evaluating electrocatalytic properties for water, CV and CP approaches are often thought to provide an abundance of information with specific parameters. According to the CV studies, hydrogen desorption peak region is one of the main factors because it is dependent on the deposit's active specific site; greater active specific surface means that more hydrogen is produced during the reduction of the adsorbed hydrogen on surface of the electrode [40]. According to CP studies, a steady current is given to both the working and counter electrodes, and the working electrode's potential deviation is measured [41]. Therefore, nickel-molybdenum alloy coatings that were plated at various c.d. from suggested bath underwent series of studies to evaluate their electrocatalytic activity against OER & HER using a 1.00 M KOH medium.

4.1. Electrocatalytic Activity for HER

4.1.1. Cyclic Voltammetry Analysis

CV is a potent technique for providing extensive knowledge on both the thermodynamics of redox reactions and the dynamics of heterogeneous electron-transfer events [42]. The CV analysis of nickel-molybdenum coatings obtained at 1.0-4.0 A dm^{-2} for HER is conducted in the range of 1.030 V to -0.570 V, at 50.0 mV s^{-1} scanning rates for 50.0 cycles. About 25.0 cycles later, the value of ipc was observed to be constant, whereas CV curves were seen following the prior cycle [43]. The scenario represents the condition where the adsorption of H atoms on the external surface of an electrode for the generation of H_2 is directly related to the discharge of H_2 gas. The deposited material's greatest i_{pc} value at 1.00 A dm^{-2} can be attributed to its high molybdenum composition (38.30 wt%) comparing to other coatings. The potential of the electrode for HER altered in a favorable direction for production of hydrogen using less over potential as the weight percentage of molybdenum in the alloy gradually elevates [44]. Manazoğlu and colleagues observed a similar discovery. Ultimately, it can be stated that the combination of higher fundamental catalytic activity and increased surface area is responsible for the HER activity rise for nickel-molybdenum alloy coatings Table 2 [45].

4.1.2. Chronopotentiometry Analysis

According to the CP studies on Ni-Mo composite coatings, depositions at varied c.d. (1.00-4.00 A dm^{-2}) were analyzed to see their efficacy toward HER operating at a steady current of 300.0 mA cm^{-2} for a span of 1800 s [46]. The electro-catalysis activity for each Ni-Mo alloy deposit was evaluated by assessing the volume of H_2 liberated for the first 300 seconds. According to experimental findings, alloy plated at 1.00 A dm^{-2} generates more H_2 than coatings coated at greater c.d., indicating that it is highly electro-catalytically active for HER [47]. Furthermore, every sample showed early potential shift right after electrolysis initiated, which was succeeded by a constant potential response [48]. This might be produced by an abrupt degradation of electrolyzed materials at electrode's surface, where hydrogen ions within solution undergoes reduction to release H_2 gas. Eventually, a condition of equilibrium among H_2 and H^+ ions is formed in this procedure [49].

4.2. Electrocatalytic activity for OER

4.2.1. Cyclic Voltammetry Analysis

It is thought that the redox changes of interfacial oxycations among the higher and lower states of oxidation catalyze the OER. Thus, the electrochemical characteristics of the redox pair shortly before the start of oxygen evolution are responsible for OER of nickel-molybdenum alloy coating [50]. Since the OER is an irreversible reaction with a large activation over potential starting potential of oxygen formation is regarded as water's decomposition potential [51]. In comparison to alloy deposited at smaller c.d. (1.420 V vs. RHE), which is comparable to value reported in the literature, evolution of oxygen on Ni-Mo coating coated at 4.00 A dm^{-2} happens at a lesser positive potential. This demonstrates that the alloy coating at 4.00 A dm^{-2} has better OER activity compared others [52]. The positive transition in redox potential arises in a way that oxygen evolution begins very quickly when electrode potentials attains redox potential of this redox pair, which consequently indicates a stronger catalytic activity for OER Table 3 [53].

4.2.2. Chronopotentiometry Study

The CP approach had been utilized to evaluate OER activity for nickel-molybdenum alloy coatings in very same context, following a CV study identical to HER analysis, and amount of O_2 released throughout research was also quantified [54].

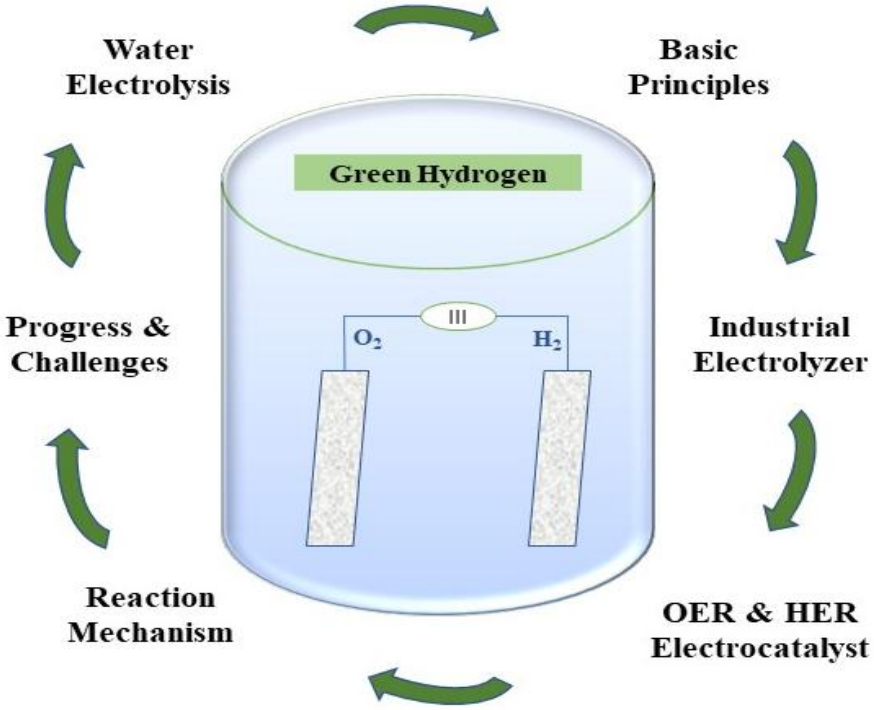

Figure 1. Overview of the key components and stages of alkaline water electrolysis for green hydrogen production

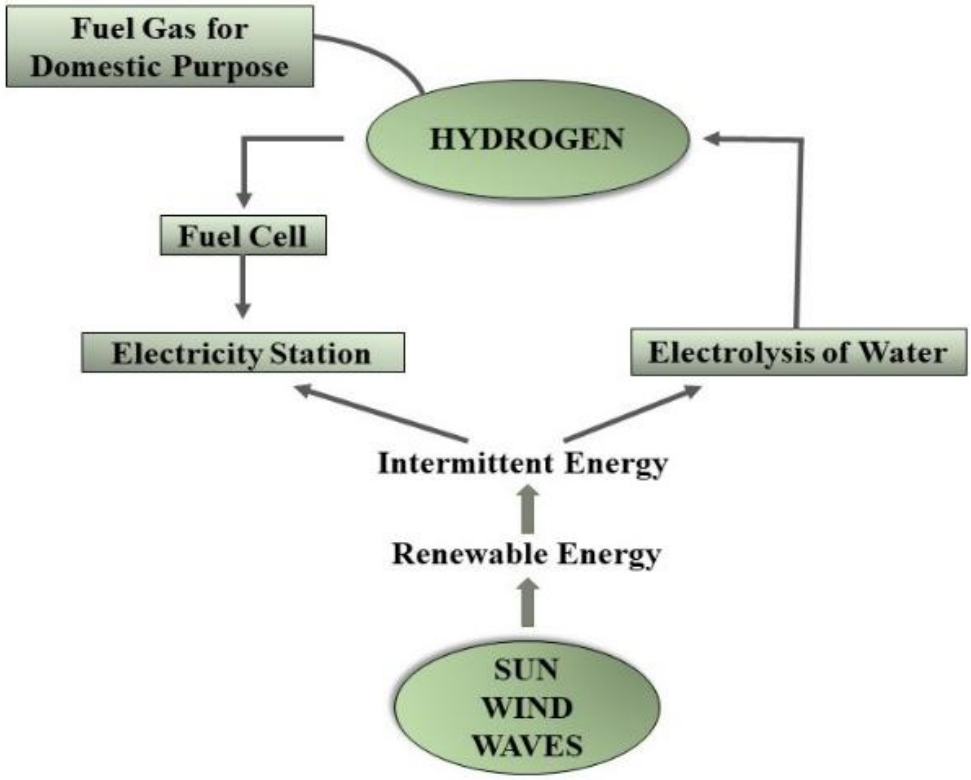

Figure 2. Conceptual energy system using water electrolysis to produce hydrogen for use as a fuel gas or an energy storage medium

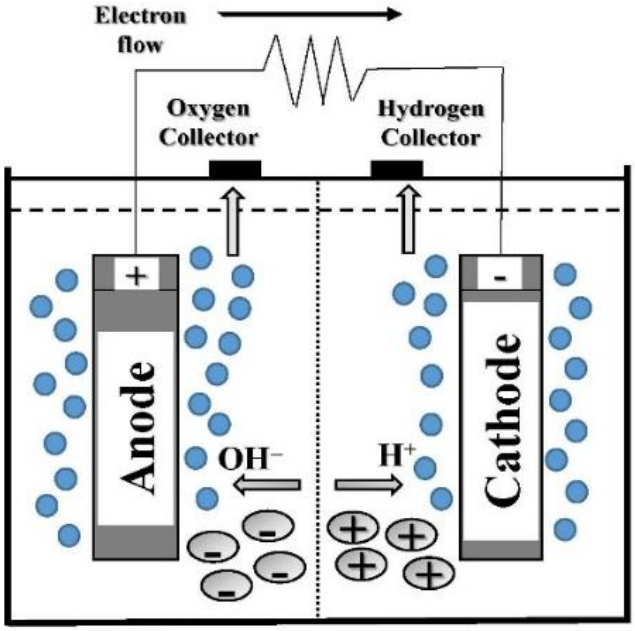

Figure 3. Basic scheme of a water electrolysis system

Table 1: The content and electrodeposition parameters for Nickel-Molybdenum bath.

Bath Components	Amount	Operating conditions
Na_2MoO_4	47 g L^{-1}	Copper cathode
$NiSO_4 \cdot 6H_2O$	17.5 g L^{-1}	Pure Ni plate anode
$Na_3C_6H_5O_7$	104 g L^{-1}	pH: 9.50
		Temp.: 302 K
		Depositing time: 9.5 minute
		c.d.: 1.5 – 4.0 A dm^{-2}

Table 2: HER parameters of nickel-molybdenum alloy coatings formed at various concentrations from optimum bath.

Current density (A $dm^{-2)}$	Cathodic peak c.d. (i_{pc})	OER Over potential	OER Onset potential
1	– 0.197	– 274.3	– 0.29
2	– 0.163	– 301.6	– 0.31
3	– 0.132	– 311.3	– 0.33
4	– 0.113	– 337.4	– 0.35

Table 3: Electrode kinetic variables of OER on nickel-molybdenum coatings deposited at various c.d.

Current density (A dm^{-2})	Anodic peak c.d. (i_{pa})	OER Over potential	OER Onset potential
1	0.096	1.61	1.61
2	0.165	1.59	1.57
3	0.225	1.57	1.55
4	0.315	1.53	1.51

Thus, based on the data derived from i_{pa} and i_{pc} readings, onset potentials, the volume of gases released and their overpotentials, it is possible to determine that deposited the nickel-molybdenum alloy evolved at a lower c.d. (1.00 A dm^{-2}) is better appropriate for the HER and those with the greater c.d. (4.00 A dm^{-2}) are ideal for the OER [55].

5. Water Splitting Mechanism

To gain a better understanding of the process of water electrolysis on the surface of present nickel-molybdenum alloy coatings, alongside the reasons for favored HER and OER under varied conditions, a famous heterogeneous catalysis spillover technique has been introduced. It primarily studies the synergy of transition metal-derived alloys for water electrolysis [56]. As a result, it is also applicable to nickel-molybdenum alloy coatings. This idea proposes that the brief intra- and inter-particle diffusion across the surface of H ad-atoms facilitates the easy cooperative functioning of alloy constituents Figure 3 [57]. It is hypothesized that nickel sites within the nickel-molybdenum surface affect proton emission along with serving as a source of hydrogen for nearby molybdenum sites, which function as hydrogen "trap" sites whereby the ion/atom recombination of a hydrogen atom to produce hydrogen molecules and its adsorption is more effectively encouraged [58]. The Hydrogen Evolution Reaction (HER) process in an alkaline solution involves three fundamental steps:

$$H_2O + M + e^- \rightleftharpoons OH^- + MH_{ads}$$
$$H_2O + MH_{ads} + e^- \rightleftharpoons H_2\uparrow + OH^- + M$$
$$MH_{ads} + MH_{ads} \rightleftharpoons H_2\uparrow + 2M$$

The process begins with an electrochemical reduction of the water molecule, resulting in hydrogen adsorbed on the surface of the electrode via the Volmer reaction (Eq.) [59]. This is followed by the Heyrovsky reaction (Eq.), which produces H_2, and/or a chemical reaction known as the Tafel reaction (Eq.). It can be concluded that the Volmer-Tafel kind of mechanism is followed by nickel-molybdenum alloy coatings for HER [60]. Likewise, the OER process in alkaline medium follows the phases outlined below in sequential order.

$$OH^- \rightarrow OH_{ads} + e^-$$
$$OH^- + OH_{ads} \rightarrow H_2O + O^-_{ads}$$
$$O^-_{ads} \rightarrow O_{ads} + e^-$$
$$2O_{ads} \rightarrow O_2\uparrow$$

This phenomenon might be defined as the absorption of slightly larger OH^- ions on the surface of an electrode, while also overlapping with other processes such as the oxygen reduction process. The highest possible nickel concentration in the coating (66.8%) electrodeposited at 4.0 A dm^{-2} primarily favors OER due to production of NiOOH on surface of electrode caused by OH^- adsorption [61].

6. Conclusion

The hydrogen production studies in this reported work utilizing the nickel-molybdenum catalyst demonstrated its outstanding electrochemical effectiveness. It has been discovered that the manufactured catalyst has low charge transfer resistance, a high turnover frequency, and outstanding stability, making it an ideal choice for sustainable and efficient hydrogen production. Using 1.0 M KOH solution, a nickel-molybdenum alloy bath has been designed to provide the highest coating capability for the splitting of water (both for HER and OER). The findings from experiments indicate that Ni-Mo alloy electrodeposited at 4.0 A dm^{-2} (33.2% Mo) and 1.0 A dm^{-2} (38.3 wt% Mo) exhibits superior electrocatalytic properties for OER and HER, as explained by CV and CP studies. The maximum electrocatalytic activities of Ni-Mo alloy coatings for both HER and OER, dependent on deposition c.d., was related to their content (wt% nickel & molybdenum), surface morphology, and structure, as supported by XRD and SEM investigations. Furthermore, coating of nickel-molybdenum alloy at 4.0 A dm^{-2} improves corrosion resistance in 1.0 M KOH in comparison with other coatings. It was also discovered that the bimetallic (nickel-molybdenum) catalyst manufactured at temperatures varying from 950°C to 1000°C had the highest effective hydrogen evolution.

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