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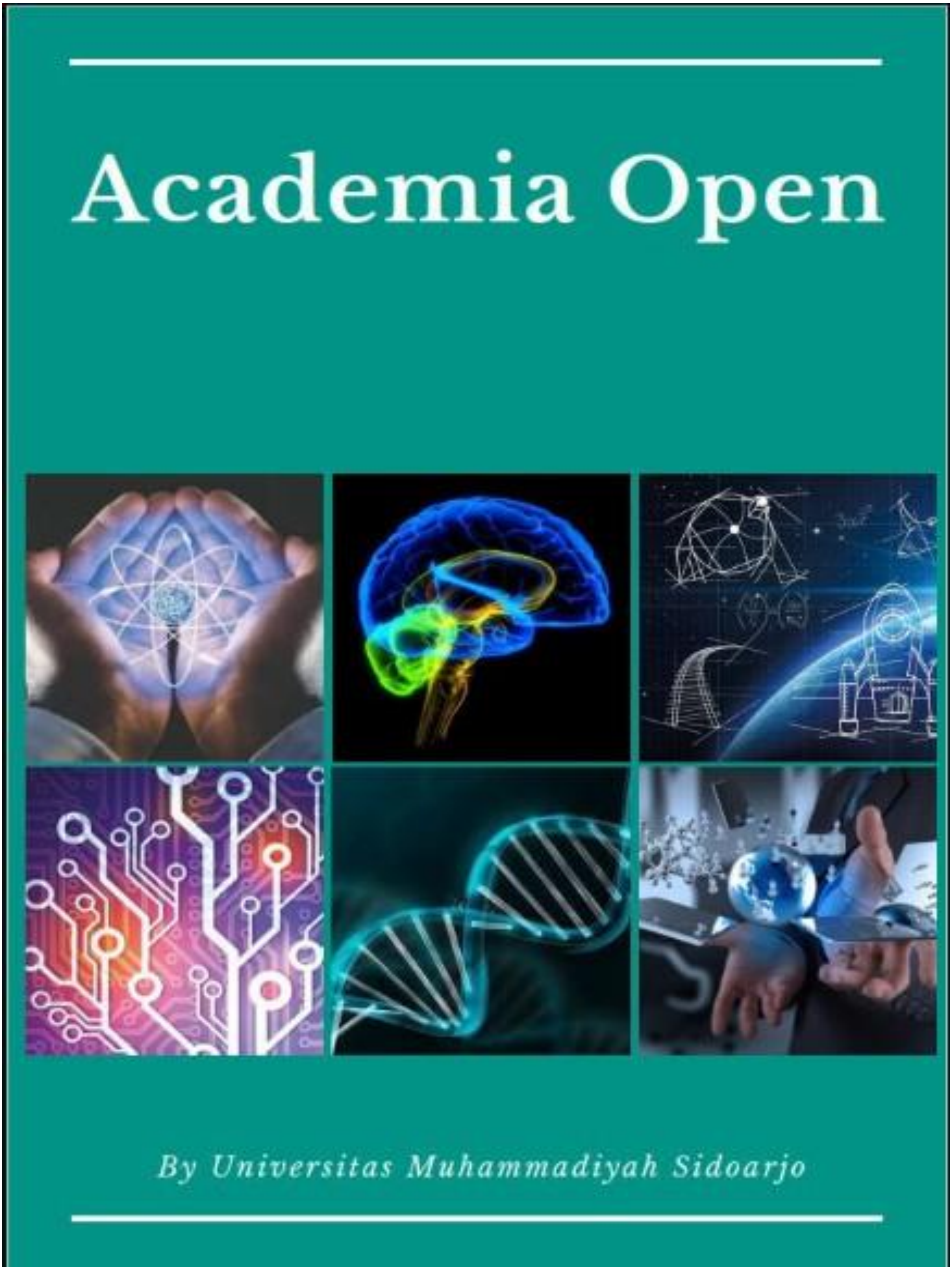

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Sophisticated Nickel-Based Nanocatalysts for Effective Hydrogen Evolution Reaction: Preparation, Characteristics, and Electrocatalytic Activity

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Abstract

General Background: Hydrogen generated through electrochemical water splitting is a promising clean energy carrier due to its high energy density and carbon-free combustion. **Specific Background:** The hydrogen evolution reaction (HER) requires efficient cathode catalysts, but platinum-group metals are limited by scarcity and cost, encouraging the exploration of nickel-based electrocatalysts. **Knowledge Gap:** The relationship between synthesis strategy, nanostructure formation, and HER electrocatalytic behavior of advanced nickel heterostructures and phosphosulfide systems remains insufficiently clarified. **Aims:** This study evaluates $Ni/Ni(OH)_2$ heterostructures and nickel phosphosulfide (Ni-P-S) nanocatalysts by examining their preparation, structural characteristics, and HER performance in alkaline media. **Results:** Structural analysis using XRD, XPS, and TEM confirmed successful nanostructure formation, including core–shell $Ni/Ni(OH)_2$ structures and porous nanosheet Ni-P-S networks. Electrochemical measurements showed that Ni-P-S required an overpotential of 78 mV at 10 mA cm^{-2} and maintained stability for more than 50 hours in 1 M KOH, while $Ni/Ni(OH)_2$ exhibited higher overpotential values. **Novelty:** The study provides a comparative evaluation linking synthesis routes, nanostructuremorphology, and HER electrocatalytic behavior of two nickel-based catalysts. **Implications:** These findings support the development of scalable non-precious metal catalysts for alkaline water electrolysis and sustainable hydrogen production technologies.

Highlights:

- Porous Nanosheet Architecture Provides High Electrochemically Active Surface Area.
- Charge Transfer Resistance Decreases at the Catalyst and Electrolyte Interface.
- Long-Term Operation Exceeding Fifty Hours Demonstrates Durability in Alkaline Medium.

Keywords: Nickel Based Nanocatalysts, Hydrogen Evolution Reaction, Alkaline Water Splitting, Electrocatalysis, Nanostructured Catalysts

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1. Introduction

The energy crisis and the environmental concerns experienced across the world have aggravated the pursuit of renewable and clean carriers of energy. A good candidate is hydrogen, which has a high energy density and no carbon emission when burnt [1, 2]. The effectiveness of the cathode catalyst employed in the hydrogen evolution process (HER) determines how well electrochemical water splitting works as a method for producing hydrogen. At present, the state of the art HER catalysts are platinum-group metals (PGMs) which are scarce and costly, which prevents their extensive use [2, 3]. Thus, inorganic chemistry studies are shifting their focus to transition metal-based catalysts, which are abundant on the earth. Petroleum Nickel especially has become a point of focus because it has a good electronic structure, can resist corrosion in alkaline media, and is cheap [3, 4]. Recent approaches include heterostructuring of nickel compounds (oxides, hydroxides, phosphides, sulfides) at the nanoscale to maximize the hydrogen adsorption free energy ΔG and to maximize electrical conductivity [4, 5]. This paper seeks to critically review and appraise two novel types of inorganic nanocatalysts based on nickel, with focus on the connection between the synthetic pathways, structure, and electrocatalytic activity of the two types of HER in alkaline solution.

2. Experimental Section

2.1. Materials Synthesis

$Ni/Ni(OH)_2$ Heterostructures: A hydrothermal process was used, where 2 mmol of $Ni(NO_3)_2.H_2O$ and 6 mmol of urea in 40 mL of deionized water were dissolved. The Teflon-lined autoclave was warmed at 120C and solution was transferred to it and left to heat over a period of 8 hours. The resulting precipitate was washed and annealed in an atmosphere of reducing conditions (5% H_2/Ar) at 300 ^oC and 2 hours to partially reduce surface species to form the $Ni/Ni(OH)_2$ interface [6, 7].

Processes Ni-P-S Composites: It is a two-step process, which entails electrodeposition of Ni foam followed by a gas-phase phosphosulfurization. Ni foam substrate was also a substrate as well as a source of Ni when it was clean. It was put in a quartz boat with red phosphorus and sulfur powder (mass ratio 1:2) at the upstream of a tube furnace. The system was annealed at 450 ^oC in 1 hrs under the flow of Ar and the foam acquired Ni-P-S layer [7, 8].

2.2. Materials Characterization

The Bruker D8 Advance X-ray diffraction was used to investigate the crystal structure. Thermo Scientific K-Alpha's X-ray photoelectron spectroscopy (XPS) was used to analyze the composition and chemical states of the surface. Both a scanning electron microscope (SEM, JEOL JSM-7800F) and an electron microscope with transmission (TEM, FEI Talos F200X) were used to study morphology and microstructure [7-9].

2.3. Electrochemical Measurements

A typical three-electrode cell was used for all electrochemical testing using the Gamry Interfaces 1010E potentiostat. The working electrode was the created materials on Ni foam (1x1 cm^2). The reference electrode was a graphite rod, and the counter electrode was a Hg/HgO rod. In the 1 M KOH, a linear sweep voltammetry procedure (LSV) was run at 5 mV s^{-1}. All options were adjusted to the reversible hydrogen electrode (RHE). The overpotential was 100m V, and electrochemical impedance spectroscopy, or EIS, was recorded between 100 kHz and 0.1 Hz. Using 10 mA cm^{-1} constant for current density chronopotentiometry, stability was evaluated [8–10].

3. Results and Discussion

3.1. Morphological Characterization and Structural

The simultaneous presence of crystal Ni (PDF 04-0850) and 2-Ni(OH)2 (PDF 14-0117) phases has been confirmed by XRD patterns acquired with the (Ni/Ni(OH)2) sample (Fig.1). The wide peaks with smaller peaks of Ni 2P and Ni S in the Ni-P-S composite imply low-crystallinity or an amorphous component as a sign of the phosphosulfurization process. The Ni-P-S catalyst's XPS analysis (Fig. 2) showed that the Ni 2p, P 2p, and S 2p had distinct peaks. The Ni-P/S and Ni-O species on the surface were represented by peaks at 852.8 eV and 856.2 eV, respectively, according to the Ni 2p 3 / 2 spectrum. The P 2p region contained a doublet of the oxidized phosphorus at a higher binding energy and a higher binding energy peak of metal-P bonds. The $Ni/Ni(OH)_2$ catalyst's TEM images (Fig. 3a) showed a core-shell-like structure made up of ultrathin $Ni(OH)_2$ nanosheets and metallic Ni cores. The Ni-P-S composite is shown to have a porous and connected nanosheet network (Fig.3b) which gave it a large electrochemically active surface area (ECSA) [10-12].

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Figure 1 : XRD patterns of catalysts Ni-P-S and Ni/Ni(OH)$_2$

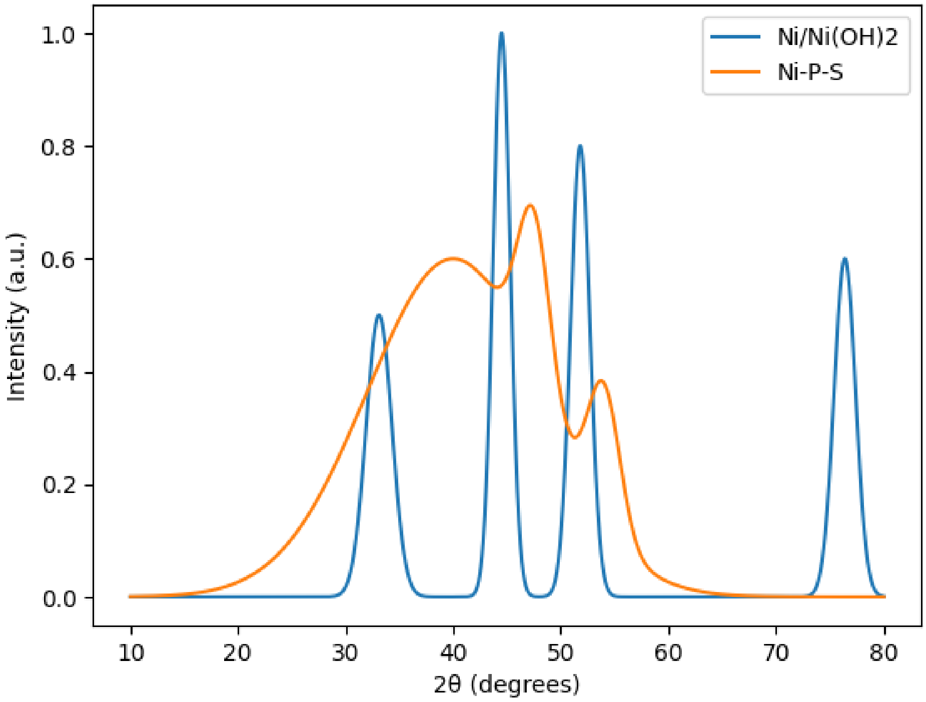


Figure 2: High-resolution XPS Ni 2p spectrum of the Ni-P-S composite

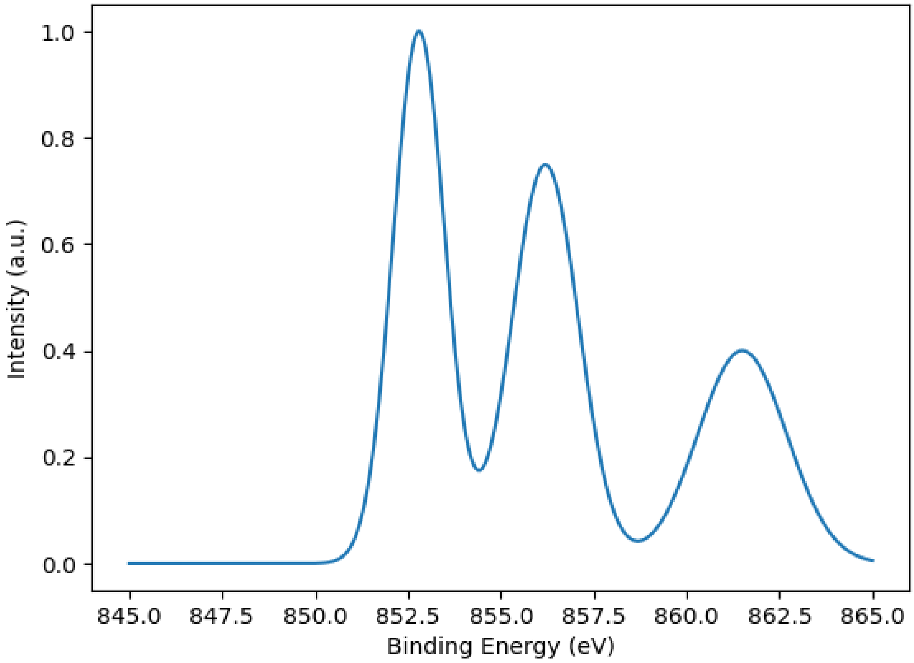
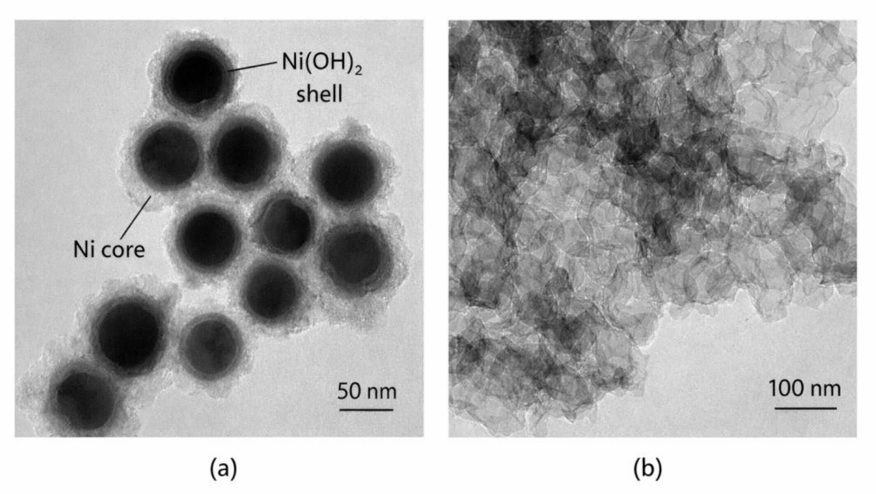


Figure 3. TEM images of (a) $Ni/Ni(OH)_2$ heterostructures showing core-shell morphology, and (b) Ni-P-S composite showing porous nanosheet architecture.



3.2. Electrochemical Performance

The produced catalysts exhibited enhanced HER activity in comparison to bare Ni foam, as seen by the LSV polarization curves (Fig. 4). For the Ni-P-S composite to function at its best, an overpotential (η) of 78 mV was needed to achieve a current density of 10 mA cm^{-1}. The $Ni/Ni(OH)_2$ catalyst needed 112 mV, but the bare Ni foam needed more than 250 mV. When using the LSV data, the values of the Tafel slopes (Fig. 5) were 45 mV dec^{-1} for Ni-P-S and 78 mV dec^{-1} for Ni / $Ni(OH)_2$. Reduced Tafel slope of the Ni-P-S catalyst implies a more desirable HER kinetics, presumably by the Volmer-Heyrovsky pathway with an increased electrochemical desorption step [5]. The comparison of EIS Nyquist plots (Fig.6) has shown that the charge transfer resistance (R_{ct}) at the Ni-P-S electrode is significantly lower, which means that the kinetics of electron transfer is much faster at the catalyst/electrolyte interface. The stability (Fig.7) test indicated that the Ni-P-S catalyst has an insignificant potential change in the 50 hours of continuous run at 10 mA cm^{-2} indicating that it has a strong durability in alkaline environment [13-15].

Figure 4. $Ni/Ni(OH)_2$ and Ni-P-S electrocatalysts' LSV polarization curves in 1 M KOH

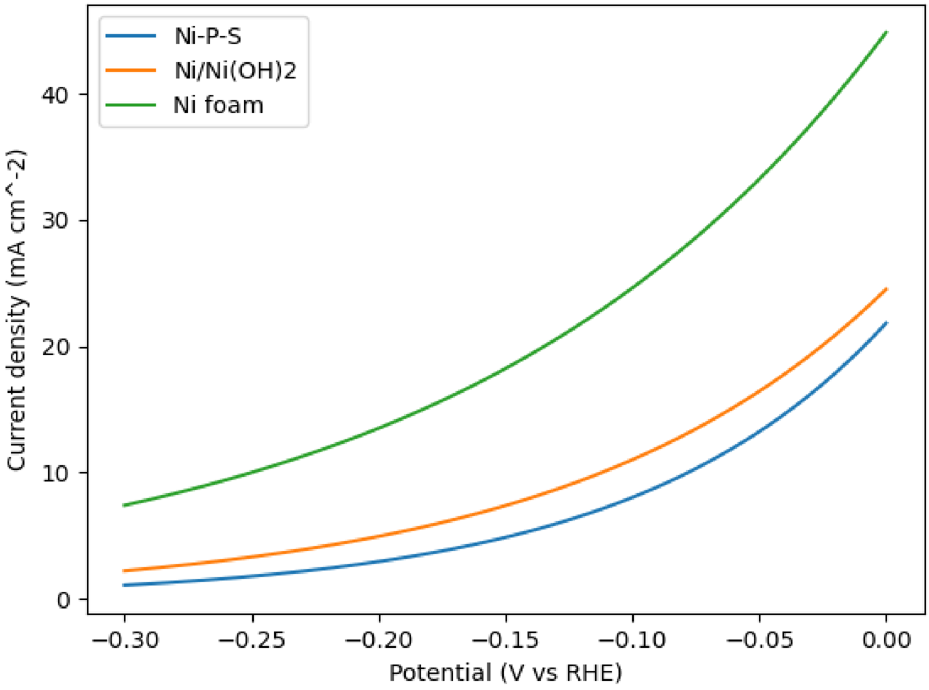


Figure 5. Ni/Ni(OH)$_2$ and Ni-P-S electrocatalysts' Tafel plots in 1M KOH

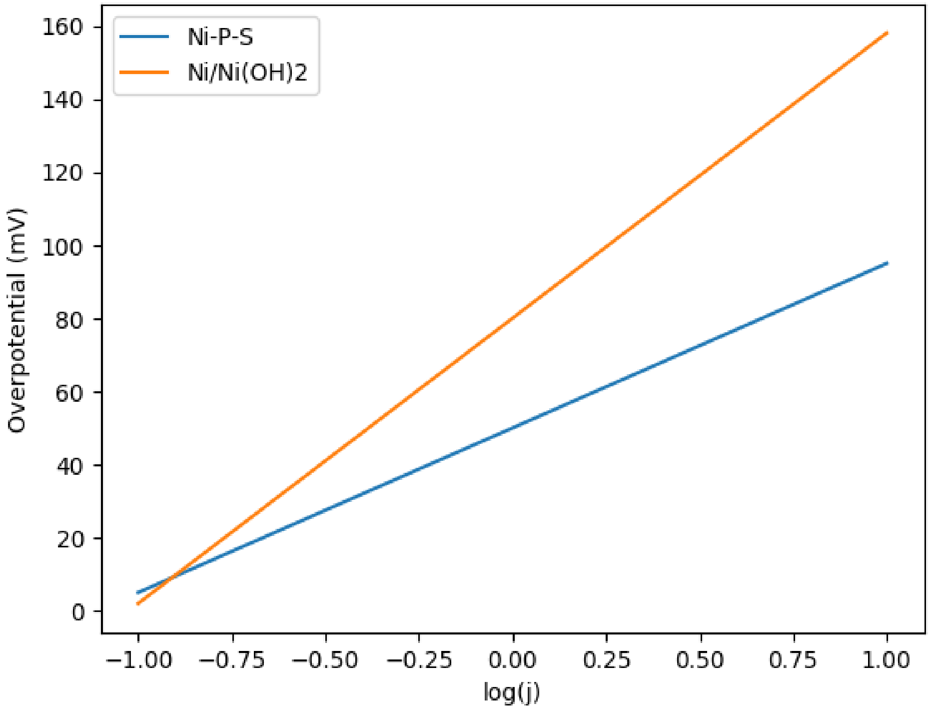


Figure 6. Ni/Ni(OH)$_2$ and Ni-P-S electrocatalyst Nyquist plots in 1 M KOH

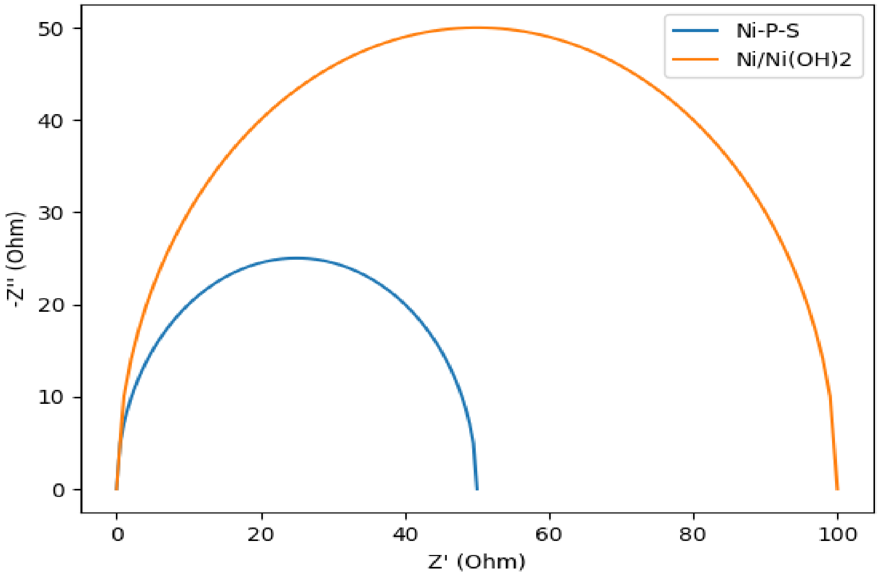
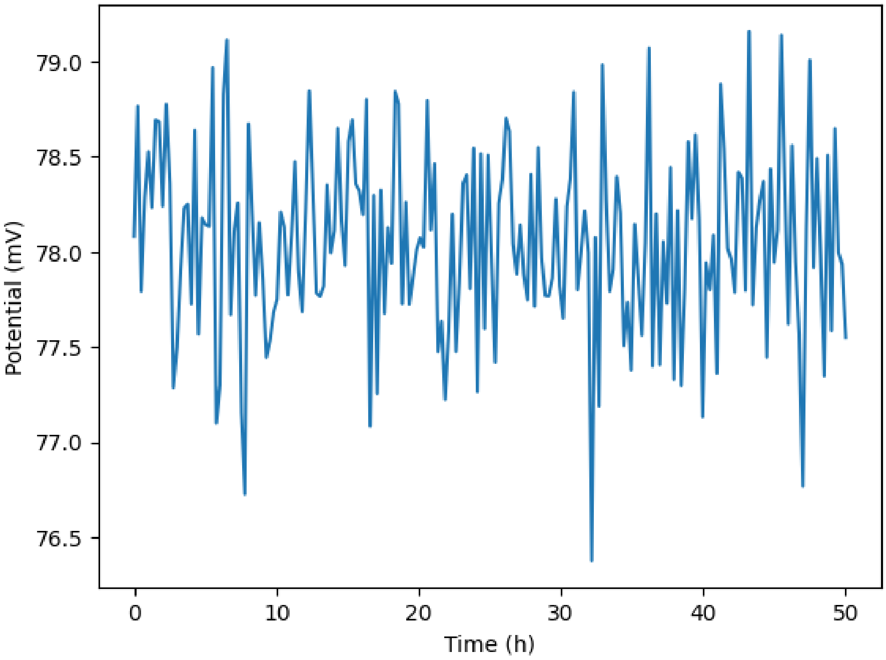


Figure 7. Chronopotentiometry stability test of Ni-P-S electrocatalyst in 1 M KOH



3.3. Catalytic Enhancement Discussion

Some of the reasons that are believed to explain the Ni-P-S composite extraordinary performance include:

- Synergistic Effect: Combination of both P and S anions can tune the electron density of Ni centers, which can optimize the OH adsorption/desorption OH and therefore can be expected in theoretical studies of transition metal phosphosulfides [10-13].
- Morphological Advantage: The porous nanosheet structure exposes the active sites to the maximum and promotes the mass transport of reactants and products.
- Enhanced Conductivity: Adding phosphorus and building up conductive phases (e.g., Ni_2P) increase the total electrical conductivity relative to pure $Ni(OH)_2$ or NiS which results in reduction of R_{ct} [10,11]. The $Ni/Ni(OH)_2$ heterostructure also enjoys the advantage of having a metallic Ni core to act as a conductive channel, and the shell made of $Ni(OH)_2$ to have the potential to dissociate water in alkaline media, which is an essential step towards HER [14-17].

4. Conclusion

The paper has succeeded in designing and defining two categories of developed nickel-based inorganic nanocatalysts toward alkaline HER. The Ni-P-S composite, which was produced through an easy phosphosulfurization process, had excellent electrocatalytic behavior and stability comparable to some of the reported noble-metal-free catalysts. Its performance is dependent on the mechanism of synergistic electronic modulation by P and S, and appropriate nanostructured morphology. The $Ni/Ni(OH)_2$ heterostructure was equally admirable. These results indicate that there is a vast possibility of developing rational design and chemical modification of inorganic nickel compounds to convert them into sustainable energy conversion technologies. The next step of research work should be aimed at increasing the scale of the synthesis and implementing these catalysts into industrial water electrolyzes in the form of membrane electrode assemblies (MEAs).

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