Preparation of $\beta$-Ni (OH)$_2$ Nanosheet–Modified Glassy Carbon for Pseudocapacitors

Muhammad Buchari Setia Putra  
*Department of Chemistry, Faculty of Mathematics and Natural Sciences, Universitas Indonesia, Depok 16424, Indonesia*

Aliyah Aliyah  
*Department of Chemistry, Faculty of Mathematics and Natural Sciences, Universitas Indonesia, Depok 16424, Indonesia*

Afiten Rahmin Sanjaya  
*Department of Chemistry, Faculty of Mathematics and Natural Sciences, Universitas Indonesia, Depok 16424, Indonesia*

Respati Kevin Pramadewandaru  
*Department of Materials and Metallurgical Engineering, Faculty of Industrial Technology and Systems Engineering, Institut Teknologi Sepuluh Nopember, Surabaya 60111, Indonesia*

Hoel Chung  
*Department of Chemistry and Research Institute for Convergence of Basic Science, Hanyang University, Seoul 04763, South Korea*

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Preparation of β-Ni (OH)₂ Nanosheet–Modified Glassy Carbon for Pseudocapacitors

Muhammad Buchari Setia Putra¹, Aliyah Aliyah¹, Afiten Rahmin Sanjaya¹, Respati Kevin Pramadewandaru², Hoei Chung³, and Tribidasari Anggraningrum Ivandini¹*

1. Department of Chemistry, Faculty of Mathematics and Natural Sciences, Universitas Indonesia, Depok 16424, Indonesia
2. Department of Materials and Metallurgical Engineering, Faculty of Industrial Technology and Systems Engineering, Institut Teknologi Sepuluh Nopember, Surabaya 60111, Indonesia
3. Department of Chemistry and Research Institute for Convergence of Basic Science, Hanyang University, Seoul 04763, South Korea

*E-mail: ivandini.tri@sci.ui.ac.id

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Abstract

β-Nickel hydroxide (Ni(OH)₂) nanosheet–modified glassy carbon for pseudocapacitors was successfully prepared. The nanosheets were synthesized via a conventional growth seeding method combined with hydrothermal treatment. Transmission electron microscopy (TEM) characterization indicated the hexagonal structure of the nanosheets of around 15 nm in size, and X-ray diffraction (XRD) pattern confirmed the β-phase lattice crystal. These nanosheets used to modify the glassy carbon surface, increased the electroactive surface area by around 15 times. Furthermore, electrochemical investigation of the modified glassy carbon revealed an excellent performance and a specific capacitance of 628.01 F g⁻¹ at a 1 A g⁻¹ current density. Moreover, an outstanding stability was demonstrated, with 89.9% retention of the capacitance observed after 3000 cycles at a 1 A g⁻¹ current density.

Keywords: energy storage, glassy carbon, pseudocapacitor, surface modification, β-Ni(OH)₂

Introduction

The technology of energy conversion, namely, biofuel and solar cells, has been extensively investigated [1, 2]. Solar and fuel cells use the energy transformation derived from natural sources, such as, wind, solar, geothermal, and bioenergy into electricity, represent a major technological development for energy-harvesting devices. However, renewable energy sources are unreliable as a result of ecological and geographical limitations caused by natural movements and environmental mechanisms. Therefore, new strategies are required to mitigate the effect of inconsistent energy production using renewable sources. One promising solution is the usage of an energy storage system that can aid in saving electricity for future purposes.

Batteries by far show exceptionally prevalence in energy storage development. However, the related major safety and hazard issues have led to the reconsideration of batteries as storage devices. Overheated batteries can lead to severe damage, such as thermal runaway and cell rupture; they may burst into flames and cause widespread damage [3]. Battery usage as energy storage also features some drawbacks, namely, long charging time, overheating, and environmental issues [4, 5]. In contrast to batteries, supercapacitors are known for their relative lightness and environmental friendliness [6, 7]. In addition, supercapacitors require a notably shorter charging time compared with conventional batteries and capacitors but can be discharged similar to a normal battery.

Supercapacitors fall under two main types based on their charge storage mechanisms: electric double-layer capacitors (EDLCs) and pseudocapacitors [8, 9]. EDLCs store energy through ion adsorption mechanisms or double-layer capacitance, and pseudocapacitors (Faradaic capacitors) store energy through reversible charge–transfer reactions at the electrode–electrolyte interface [7, 8, 10]. Pseudocapacitors typically attain higher capacitances and energy densities than EDLCs due to their surface-activated redox reactions, which offer considerably faster discharge rates compared with batteries [11].

Nickel hydroxide [Ni(OH)₂] has garnered considerable attention for its potential in energy storage applications; such potential can be attributed to its excellent redox behavior and high theoretical capacity, which make it a
promising material for electrodes in supercapacitors and alkaline batteries [12]. The morphology of Ni(OH)₂ considerably influences its electrochemical performance and has prompted extensive efforts to controlling its specific surface area, ionic transport pathways, and conductivity [13]. Ni(OH)₂ exists in two primary crystalline forms: α and β. The α form exhibits a higher theoretical capacity but is unstable and susceptible to conversion to the more stable β form, which offers an improved stability and higher discharge voltage, which makes it suitable for electrode materials [14–17].

Carbon-based materials have been widely explored to improve the electrochemical performance of β-Ni(OH)₂, particularly its stability and conductivity. These materials offer wide potential windows, high power density, excellent cycling lifetime, and high conductivity. The combination of metal–carbon electrodes can considerably improve the conductivity of Ni(OH)₂. Examples include the Ni(OH)₂ deposited on carbon dots [18], nitrogen-doped multiwalled carbon nanotubes [19], biomass-derived carbon–Ni(OH)₂ composites [20, 21], and graphene-based electrode [22].

In this study, glassy carbon electrodes (GCEs) were selected due to their high electrical conductivity, chemical stability, and ease of modification. We used growth seeding method and hydrothermal treatment to synthesize β-Ni(OH)₂ nanoparticles deposited them on GCEs to serve as the main catalyst for pseudocapacitors. To enhance the performance, we incorporated activated carbon to increase the electroactive surface area (approximately 15 times) and provide numerous active sites. Notably, a specific capacitance of 628.01 F g⁻¹ was achieved at a current density of 1 A g⁻¹. The activated carbon also aided in the dispersion of Ni(OH)₂ catalyst ink onto the GCE surface. In addition, Nafion was used as a polymer binder to protect the catalyst layers and improve the durability [23]. The combined benefits of Ni(OH)₂, activated carbon, and Nafion on GCE resulted in enhanced electrocatalytic activity and durability, which make these materials suitable for high-performance pseudocapacitor applications [24, 25].

**Experimental Design**

**Materials and chemicals.** Nickel chloride (NiCl₂) and Nafion (5 wt%) were purchased from Sigma-Aldrich (St. Louis, USA), and trisodium citrate (C₃H₅Na₃O₇) was obtained from Wako Pure Chemical Industries, Ltd (Osaka, Japan). Potassium hydroxide (KOH), sodium hydroxide (NaOH), ammonium hydroxide (NH₄OH), acetonitrile (CH₃CN), ethanol (CH₃CH₂OH), 2-propanol (C₃H₇OH), hydrochloric acid 38% (HCl), and conductive carbon mesoporous were bought from Merck (Darmstadt, Germany). Double-distilled water was produced using Millipore Direct-Q® 5 UV. All chemicals were of analytical grade and used without further purification.

**Synthesis of β-Ni(OH)₂ nanoparticles.** Ni(OH)₂ nanoparticles were synthesized through a conventional seed-growth technique [26]. Initially, a precursor solution prepared from a mixture of 0.212 mM NiCl₂ and 1.210 mM C₆H₅Na₃O₇·2H₂O in 20 mL water was added with 10 mL 0.1 M NaBH₄ followed by vigorous stirring until the formation of black precipitate. Afterward, the solution was stirred at 35 °C for 9 h for the complete oxidation of nickel and formation of a stable nickel–citrate complex with a clear green color. Then, immediately 5 mL 4 M NaOH solution was added to the mixture to form β-Ni(OH)₂ nanoparticles. The mixture was continuously stirred for 10 min and then transferred to a Teflon-based autoclave and heated at 100 °C for 24 h to ensure the formation of β-Ni(OH)₂. The synthesized material was subsequently washed with water and ethanol before its final redispersion in water.

**Preparation and characterization of the electrodes.** We prepared the working electrodes by immobilizing β-Ni(OH)₂ on the GCE surface (55 mm diameter). Prior to use, the GCE was cleaned mechanically using alumina powder, washed in an ultrasonic water bath, and then left to naturally dry at room temperature. Next, ultrasonication was used in the preparation of a homogeneous suspension containing 5 mL β-Ni(OH)₂, 2 mL ethanol, 3 mL deionized water, and 0.5 mg activated carbon. Then, 10 µL of this suspension was dropped onto the GCE surface and allowed to dry naturally for the immobilization process. This step was repeated until the minimum measurable weight of the active material reached 75 µg cm⁻². The preparation process was completed through fixing the active material with a drop of 8 µL Nafion as a binder. Three additional electrodes, including an unmodified GCE, an electrode prepared without the conductive carbon and Nafion, and an electrode prepared without the Nafion, were also investigated for comparison.

The prepared material was analyzed via transmission electron microscopy (TEM, JEOL 2010), powder X-ray diffraction (XRD, Rigaku, Rint-2000) with Cu Kα, 45 kV, 200 mA, scanning electron microscopy (SEM, Hitachi SU3500), and X-ray photoelectron spectroscopy (XPS, Thermo Fisher XPS K-alpha system). Further, the electrochemical properties were determined at room temperature using an IVIUM COMPACTSTAT and EDAQ potentiostat (ER466). A three-electrode configuration cell, which contained a platinum wire as the counter and an Ag/AgCl (RE-1B, ALS) system as the reference electrodes, was utilized as the electrochemical cell, while 2.0 M KOH was used as the electrolyte.

**Results and Discussion**

**Structural and morphological properties.** SEM was performed to investigate the morphology of the GCE surface before and after the modification with β-Ni(OH)₂ na-
noparticles. Comparison with the unmodified GCE (Figure 1(a)) revealed the densely grown and well-distributed β-Ni(OH)₂ on the modified GCE surface (Figure 1(b)). The SEM images demonstrated the presence of β-Ni(OH)₂ nanosheet layers with a hexagonal structure, which is a characteristic feature of β-Ni(OH)₂ [13]. Figure 1(c) shows the β-Ni(OH)₂ nanosheets after their mixture with conductive carbon and subsequent immobilization onto the GCE surface. Figure 1(d) displays the final modification with the addition of conductive carbon and Nafion. The data compromised the clarity of β-Ni(OH)₂, possibly due to the presence of Nafion, which covered the immobilized β-Ni(OH)₂ on the electrode surface.

XRD characterization was conducted to analyze the synthesized β-Ni(OH)₂ nanoparticles and identify their polymorph structure. Figure 2 shows the XRD spectra of the modified GCE with the summary of diffraction peaks presented in Table 1. The findings indicate that the 20 values of the peaks matched the JCPDS No. 00-014-0117 reference card. The data, as shown by the alignment with the JCPDS reference card in Figure 2 (brown bars), revealed the successful formation of the β-Ni(OH)₂ nanostructure [13]. The graph revealed a noticeable abnormal broadening of certain diffraction lines, which indicates the presence of structural defects in the nanoparticles [27, 28]. In addition, the full width at half maximum measured using the Debye–Scherrer equation indicated an estimated single-crystal particle size of 4.6 nm.

Figure 1. SEM Images of GCE (a) Before and (b–d) after Modification with β-Ni(OH)₂, β-Ni(OH)₂ in the Presence of Activated Carbon, and β-Ni(OH)₂ in the Presence of Activated Carbon and Nafion

Table 1. XRD Parameters of β-Ni(OH)₂ Determined using the Lattice Parameters in JCPDS No. 00-014-0117

<table>
<thead>
<tr>
<th>Miller Indices (hkl)</th>
<th>2θ (°)</th>
<th>Broadened by Stacking Faults</th>
</tr>
</thead>
<tbody>
<tr>
<td>(001)</td>
<td>19.3</td>
<td>●</td>
</tr>
<tr>
<td>(100)</td>
<td>33.1</td>
<td></td>
</tr>
<tr>
<td>(101)</td>
<td>38.5</td>
<td></td>
</tr>
<tr>
<td>(102)</td>
<td>51.4</td>
<td>●</td>
</tr>
<tr>
<td>(110)</td>
<td>59.1</td>
<td></td>
</tr>
<tr>
<td>(111)</td>
<td>62.7</td>
<td></td>
</tr>
<tr>
<td>(200)</td>
<td>69.3</td>
<td>●</td>
</tr>
<tr>
<td>(201)</td>
<td>72.7</td>
<td>●</td>
</tr>
</tbody>
</table>

Figure 2. XRD Patterns of the Prepared β-Ni(OH)₂ (Brown Bar: JCPDS Reference Card)
TEM was performed to confirm the morphology of the synthesized β-Ni(OH)₂ nanoparticles. Consistent with the SEM image, the findings of TEM analysis verified the hexagonal structure of β-Ni(OH)₂ (Figure 3(a)). The TEM image did not distinctly reveal a hexagonal structure, but our methodology successfully produced thin hexagonal β-Ni(OH)₂ at 100 °C, consistent with the observations of Wang et al., 2015 [26] at the same temperature. Notably, hexagonal-like particles were detected in large-sized Ni(OH)₂ synthesized at high reaction temperatures (>80 °C). These morphological differences may be due to particle orientation, with the hexagonal morphology associated with the (100) facet and hexagonal-like morphology associated with the (001) facet of Ni(OH)₂. Consequently, our synthesized Ni(OH)₂ exhibited desired hexagonal shapes, as corroborated by our procedure and XRD analysis based on references.

ImageJ software (Figure 3(b)) was used to determine the size distribution ranging from 4 nm to 30 nm, with a predominant size of 15 nm. The less uniform size distribution observed possibly a result of the agglomeration caused by attractive forces between nanoparticles. As revealed by TEM, β-Ni(OH)₂ has a morphology revealing the presence of ultrathin nanosheets, as indicated by their transparency. The TEM inset in Figure 3(b) displays the randomly distributed and interconnected crumpled nanosheets, with an average thickness of approximately 2 nm. The 24 h hydrothermal reaction influence the observed nanosheet morphology, which is similar to that of a previous publication [26]. A previous report investigated the phase transition of β-Ni(OH)₂ via a hydrothermal reaction at 180 °C, in which the reaction up to 10 h resulted in the formation of flattened nanosheets and a compressed stacking structure [13]. This finding was also confirmed by a recent research conducted on the hydrothermal synthesis of β-Ni(OH)₂ at 200 °C for 12 h, which also led to the formation of a three-dimensional nanosheet structure [29].

The presence of β-Ni(OH)₂ and NiOOH was analyzed for further characterization via XPS. The XPS spectra of the GCE before modification (Figure 4(a), black line) showed the fluorine (F 1s) peak, which indicates the presence of an impurity introduced during XPS measurement. The spectra implied the presence of C and O before and after modification with β-Ni(OH)₂ (Figure 4(b), red line), whereas the composite spectra did not show the presence of Ni peak. The peaks of Ni 2p1/2 and Ni 2p3/2 were detected at the binding energies of approximately 890–850 eV (Figure 4(b), and the deconvolution peak of Ni 2p3/2 revealed two peaks attributed to β-Ni(OH)₂ and NiOOH at the binding energies of 855.4 and 856.6 eV, respectively. Moreover, the Ni 2p3/2 peak was observed at 855.4 eV, together with a satellite peak at approximately 861.3 eV. In addition, the peak of Ni 2p1/2 appeared at 873.3 eV with a satellite peak at around 879.5 eV. The spin–orbit splitting energy between Ni2p1/2 and Ni2p3/2 reached 17.9 eV, which indicates the presence of Ni²⁺ [30] (Chang et al., 2014). In addition, the O 1s spectrum showed the peak at 531.2 eV, which corresponded to the nickel–oxygen bonds (Figure 4(c)) was confirmed by previous findings [30].

**Electrochemical performances.** Cyclic voltammetry (CV) was applied in a 2.0 M KOH solution to investigated the properties of GCE before and after modification with β-Ni(OH)₂. The same process was applied to assess the effect of the modified β-Ni(OH)₂ in the absence and presence of carbon materials and Nafion. Figure 5(a) shows the absence of oxidation or reduction peak in the voltammogram of the unmodified GCE (black line), whereas the modified ones exhibited a typical oxidation and reduction peaks at around +0.40 V to +0.30 V (vs. Ag/AgCl), respectively, which indicates the pseudocapacitive behavior of nickel in the alkaline environment. This result is consistent with the oxidation and reduction peaks of β-Ni(OH)₂ in previous reports [29, 31, 32]. This characteristic was also likely due to the Faradaic reactions occurring on the Ni(OH)₂ surface, coupled with proton mobilization from β-Ni(OH)₂ toward the GCE current collector [33].

The presence of carbon as a supporting matrix in the modified GCE affected the lower current responses compared with the one without activated carbon, which is reasonable given the lower conductivity of activated carbon than β-Ni(OH)₂. Despite the decrease in the current response, the presence of carbon favored the achievement of stable capacitance for the β-Ni(OH)₂ compared with the absence of carbon. The activated carbon showed advantages in the control of the diffusion reaction on the electrical double-layer surface, which stabilizes the chemical reactions on the surface electrode [31, 34, 35]. In the presence of Nafion, despite the low capacitance, the onset of oxidation potential of nickel considerably shifted to around +0.45 V (vs. Ag/AgCl), and the reduction potential shifted from +0.29 V and +0.32 V to +0.35 V. This phenomenon can be attributed to less energy required to initiate redox reactions. These effects indicate that a more stable capacitance can be achieved with the use of Nafion membrane as the conductive binder beside activated carbon.

A remarkable electrochemical result was exposed in the presence of activated carbon. In addition, Nafion in the electrode exerted a notable effect on the electrochemical behavior, specifically in controlling Faradaic reactions and ion mobility on the electrode surface. In correlation with the result displayed in Figure 5(a), the β-Ni(OH)₂ exhibited a higher oxidation peak compared with the GCE with the presence of activated carbon and Nafion. This finding indicates that without the addition of activated carbon, the conductive binder beside activated carbon provides a greater stabilization of the electrode interface, which in turn decreases the onset of the oxidation reaction.
carbon and Nafion, the Faradaic reaction on the electrode surface can be also rapid and excessive. In the case of a supercapacitor, the reaction must be controlled not only for rapid charging time but also for efficient energy storage in the form of electrons. The application of supporting media on substrates contributes to the reduction of the polarization potential and facilitation of redox reactions on the electrode surface. Figure 5(a) also indicates that the addition of Nafion shifted the potential oxidation peak to a lower potential value, with Nafion controlling the ion mobility from the electrode surface to the diffusion layer.

Figure 3. (a) TEM Image of β-Ni(OH)₂ Nanoparticles with (b) Particle Distribution Obtained using ImageJ Software; The Inset of (b) Shows the Thickness of the Interconnected β-Ni(OH)₂ Stack.

Figure 4. (a) XPS Spectrum of the GCE before (Black Line) and after (Red Line) Modification with β-Ni(OH)₂ Together with the Related Deconvolutions Peaks of (b) Ni 2p and (c) O 1s.
Figure 5. Voltammograms of 2.0 M KOH at GCE (a) Before (Black Line) and after the GCE modification with β-Ni(OH) (Red Line), with β-Ni(OH) and Activated Carbon (Blue Line), and with β-Ni(OH)₂, Activated Carbon and Nafion (Green Line) with an Applied Scan Rate of 100 mV/s, and (b) GCE Modified with β-Ni(OH), Activated Carbon, and Nafion in Various Scan Rates

Table 2. Electrochemical Active Surface Area of the Prepared Electrodes

<table>
<thead>
<tr>
<th>Electrode</th>
<th>Linear equation</th>
<th>Electroactive Surface Area (mA cm⁻²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Unmodified GCE</td>
<td></td>
<td>0.34 × 10⁻⁷</td>
</tr>
<tr>
<td>β-Ni(OH)₂-modified GCE</td>
<td>y = 8.15 × 10⁻⁵</td>
<td>5.18 × 10⁻⁹</td>
</tr>
<tr>
<td>β-Ni(OH)₂-modified GCE in the presence of activated carbon</td>
<td>y = 1.85 × 10⁻⁵</td>
<td>7.79 × 10⁻⁹</td>
</tr>
<tr>
<td>β-Ni(OH)₂-modified GCE in the presence of activated carbon and Nafion</td>
<td>y = −1.29 × 10⁻⁵</td>
<td>5.45 × 10⁻⁹</td>
</tr>
</tbody>
</table>

The modification of the GCE surface also influenced the electrochemically active surface area, and this finding was directly correlated to the electron transfer kinetics between the reactant and electrode surface. Further, 2.0 M KOH was used to analyze the electroactive surface area at various scan rates. The current responses were used in the computation of the active surface area via Randle’s Sevcik equation below:

\[ ip = 2.687 \times 10^5 n^{\frac{3}{2}} A^{\frac{1}{2}} C^\frac{1}{2} v^\frac{1}{2} \]  

The peak current was proportional to the concentration (C) and square roots of the diffusion coefficient (D) and scan rate (v). In addition, the transferred electrons coefficient (n) of the redox substance affected the current. Figure 5(b) indicates consistent increase in the current responses of the modified GCE at the potentials of +0.45 and +0.35 V, proportional to the scan rates. Typical findings were also obtained during the use of unmodified and other modified GCEs. Accordingly, the electroactive surface area measurements were calculated (Table 2).

The Table 2 shows the high electroactive surface areas typically recorded after GCE modifications. Modification with β-Ni(OH)₂ caused the increase in the electroactive surface area from 3.43 × 10⁻¹⁰ mA cm⁻² to 5.18 × 10⁻⁹ mA cm⁻². Meanwhile, the addition of activated carbon to the β-Ni(OH)₂-modified GCE considerably increased the electroactive surface area to values 30% higher than 7.79 × 10⁻⁹ mA cm⁻². However, further addition of Nafion decreased the electroactive surface area to 5.45 × 10⁻⁹ mA cm⁻². Regardless, this value is still slightly higher than that of the modification without carbon. The increase in the electroactive surface area of a pseudocapacitor material leads to the decrease in its electrochemical performance as a result of the thickened diffusion layer on the electrode surface [36, 37]. This limitation can be overcome by the modification of Ni(OH)₂ with a stable and conductive material, such as activated carbon, for the effective control of ion mobility and improved conductivity. The modification was further enhanced by the presence of Nafion, which increased the selectivity toward specific ions (such as oxygen and hydrogen) and facilitated their passage through the permeable membrane layer formed by mixing the prepared modified Ni(OH)₂ with Nafion [38, 39]. The Nafion binder caused a back diffusion effect at the diffusion layer on the β-Ni(OH)₂ surface, which resulted in continuously suppressed ionic diffusion. This mechanism required ionic species (H⁺, K⁺, and OH⁻) to pass through a diffusion layer of finite thickness to reach or leave the electrode surface. In addition, this modification minimized the self-poisoning process of β-Ni(OH)₂, which prevented it from becoming an inactive substrate, similar to NiO₂, due to the overpotential reactions that decreased during the charging and discharging processes of supercapacitors, as described in Reactions (1) and (2).
Oxidation: $\beta$-Ni(OH) + OH$^-$ $\rightarrow$ NiOOH + H$_2$O + e$^-$ (charging) 
Reduction: NiOOH + H$_2$O + e$^-$ $\rightarrow$ $\beta$-Ni(OH) + OH$^-$ (discharging)

Analysis of GCE modified with $\beta$-Ni(OH) as pseudocapacitor. Analysis of the supercapacitor characteristic was attained through the use galvanostatic charge–discharge (GCD) and specific capacitance, energy density, and power density at various current densities during GCD measurements. Calculations revealed the highest charge–discharge performance observed at a 1 Ag$^{-1}$ current density with a discharge time of 630 s. As shown in Figures 6(a and b), a 1 Ag$^{-1}$ current density of the GCE modified with $\beta$-Ni(OH)$_2$ in the presence of activated carbon and Nafion required 260 s discharge time to complete the measurement. A specific capacitance of 628.01 F g$^{-1}$ was achieved, and this value is higher than other applied current densities of 2, 5, and 10 A g$^{-1}$, with specific capacitance of 579.71, 362.31, and 241.54 F g$^{-1}$, respectively.

Moreover, longer charging time result was observed compared with the discharging time. This finding suggests that the developed $\beta$-Ni(OH)$_2$ required a long period to accommodate the oxidation reaction that affected the energy density the performance. Furthermore, the energy density of the prepared electrode showed a linear decrease at 113.04, 104.35, 65.22, and 43.48 Wh kg$^{-1}$ to the applied current of 1, 2, 5, and 10 A g$^{-1}$. As a result, a density of 4.348 kW kg$^{-1}$ was reached at an applied current of 10 A g$^{-1}$ (Figure 6(c)). These findings suggest the potential of the modified electrode to be used as a supercapacitor. It also offers a balance between energy storage and power delivery.

Assessing electrode stability is vital for determining the effectiveness of electrodes in supercapacitor applications. Accordingly, a CV for 3000 cycles was performed to evaluate the electrode stability. CV was applied in the potential range between 0 and 0.6 V using a 2 M KOH electrolyte at a 100 mV s$^{-1}$ scan rate. The charge–discharge data were obtained for every 100 cycles. A remarkable retention of 89.9% was observed for the GCE modified with $\beta$-Ni(OH)$_2$ in the presence of activated carbon and Nafion after 3000 cycles. This finding confirms that the modification of $\beta$-Ni(OH)$_2$ with activated carbon and Nafion remarkably improved its stability performance as a pseudocapacitor material. Figure 7(a) reveals the slight decrease in the current responses of the oxidation and reduction peaks, which confirmed the excellent stability of the proposed electrode. In summary (Figure 7b), the prepared electrode exhibited an impressive long-term stability, which verified its exceptional condition and stability and thus makes it a promising material for supercapacitor applications.

![Figure 6. GCD Plots (a) of GCE Modified with $\beta$-Ni(OH) in the Presence of Activated Carbon and Nafion using a 2 M KOH Solution Together with the Related Plots of (b) Energy Density and Power Density, (c) Specific Capacitance at Various Currents](image-url)
Figure 7. Stability Tests of GCE Modified with β-Ni(OH)₂ in the Presence of Activated Carbon and Nafion using (a) 3000 Cycles in CV in a Solution of 2.0 M KOH with a 100 mV s⁻¹ Scan Rate and (b) 3000 Cycles Charge–Discharge Retention Counted by Every 100 Cycles

Table 3. Comparison of the Specific Capacitances of Some Reported Supercapacitors based on Ni(OH)₂ and Carbon

<table>
<thead>
<tr>
<th>Electrode</th>
<th>Specific Capcitance (F·g⁻¹)</th>
<th>Electrolyte</th>
<th>Cycle Stability (%)</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>β-Ni(OH)₂/ɤ-NiOOH</td>
<td>1420 at 2 A·g⁻¹</td>
<td>1 M KOH</td>
<td></td>
<td>[40]</td>
</tr>
<tr>
<td>β-Ni(OH)₂/GO/CNTs</td>
<td>1815 at 2 A·g⁻¹</td>
<td></td>
<td>97% after 2000 cycles</td>
<td>[13]</td>
</tr>
<tr>
<td>Ni(OH)₂@N-MWCNT</td>
<td>350 at 0.5 A·g⁻¹</td>
<td>6 M KOH</td>
<td></td>
<td>[19]</td>
</tr>
<tr>
<td>β-Ni(OH)₂ from NiAl-LDHs precursor</td>
<td>829 at 1 A·g⁻¹</td>
<td>10 M KOH</td>
<td>57.3%</td>
<td>[41]</td>
</tr>
<tr>
<td>β-Ni(OH)₂ nanoflakes/CC</td>
<td>815.67 at 1 A·g⁻¹</td>
<td></td>
<td>98.1% after 4000 cycles</td>
<td>[42]</td>
</tr>
<tr>
<td>Graphene-Ni(OH)₂/CC</td>
<td>1825 at 1 A·g⁻¹</td>
<td>2 M KOH</td>
<td>83.5%</td>
<td>[43]</td>
</tr>
<tr>
<td>RH-C/Ni(OH)₂</td>
<td>952 at 1 A·g⁻¹</td>
<td>6 M KOH</td>
<td></td>
<td>[21]</td>
</tr>
<tr>
<td>Ni(OH)₂/Activated Carbon</td>
<td>198 at 1 A·g⁻¹</td>
<td>1 M KOH</td>
<td>84% after 1500 cycles</td>
<td>[44]</td>
</tr>
<tr>
<td>Ni(OH)₂-3DOMC</td>
<td>552.50 at 1 A·g⁻¹</td>
<td>6 M KOH</td>
<td></td>
<td>[45]</td>
</tr>
<tr>
<td>GCE modified with β-Ni(OH)₂ and activated carbon and Nafion</td>
<td>628.01 at 1 A·g⁻¹</td>
<td>2 M KOH</td>
<td>89.9% after 3000 cycles</td>
<td>This work</td>
</tr>
</tbody>
</table>

The unique structure of β-Ni(OH)₂ demonstrated excellent performance compared with previously reported Ni(OH)₂-based materials. Table 3A displays the comparison of the stability test results and the evidence showing that the proposed material outperformed the others. After 3000 cycles, the GCE modified with β-Ni(OH)₂ in the presence of activated carbon and Nafion maintained an impressive retention of 89.9% of the charge–discharge...
performance. Furthermore, a 100% performance was retained after 1000 cycles, and it only slightly decreased to 98% after 2000 cycles while still exhibiting favorable specific capacitance. For further optimization of the proposed material, the size of the unique nanostructure of β-Ni(OH)₂ and activated carbon must be controlled to improve the specific capacitance. This goal can be achieved through the utilization of a stable capping agent and exploration of potential hydrothermal synthesis pathways that incorporate hard templates to improve particle homogeneity. In addition, the improvement of the current collector is an important consideration for considerably increasing the specific capacitance and overall stability of the pseudocapacitive material. In addition, different ratios of the catalyst ink must be explored to determine the optimal composition, which ensures that the presence of Nafion does not excessively compromise the pseudocapacitive performance.

**Conclusion**

The β-phase Ni(OH)₂-modified activated carbon was synthesized through the combined seeding growth method and hydrothermal treatment. The resulting β-Ni(OH)₂ exhibited a distinct nanostructure characterized by hexagonal layer sheets atop the beta phase lattice crystal and with particle sizes ranging from 4 nm to 30 nm and being predominantly around 15 nm. The incorporation of activated carbon into the β-Ni(OH)₂ considerably improved the electrochemical active surface area compared with that of the unmodified material. Subsequent modification with Nafion resulted in a remarkable stability, which retained 89.9% of the initial performance of the studied material after 3000 cycles. The prepared material showcased a specific capacitance of 628.01 Fg⁻¹, an energy density of 113.04 Wh kg⁻¹, and a power density of 0.44 kW kg⁻¹ at an applied current density of 1 A g⁻¹, which underscores its potential as an electrode for the advancement of energy storage technologies.

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**Author Contribution**

M.B.S.P.: data curation, formal analysis, investigation, and writing; A.A.: investigation, writing-review, and editing; A.R.S.: investigation, writing-review, and editing; R.K.P.: investigation, writing-review, and editing; H.C.: conceptualization, supervision, validation, writing-review, and editing; T.A.I.: conceptualization, supervision, validation, funding acquisition, writing-review, and editing. All authors are equally contributing and have given approval to the final version of the manuscript.

**Declaration of Competing Interest**

There are no conflicts to declare.

**References**


Preparation of β-Ni (OH)₂ Nanosheet–Modified Glassy Carbon


