Synthesis and Characterization of Nickel and Nickel Hydroxide Nanopowders

Catherine Huang and Teresa D. Golden
Nickel, Ni, and nickel hydroxide, \( \text{Ni(OH)}_2 \), powders with nanosized particles were synthesized using a chemical method.

The polymer Poly(vinyl pyrrolidone) or PVP was added to solutions of Ni for purposes of particle protection.

X-Ray analysis revealed the size of \( \text{Ni(OH)}_2 \) particles to be approximately 19 nanometers in diameter and the sizes of Ni particles to be between 7 and 12 nm.
Metal nanopowders are highly useful because they possess unique chemical and physical properties and have possible applications in optical and magnetic devices.

Such powders are generally produced through the chemical reduction of metal ions using a reductant or are produced through electrochemically depositing metal particles as films.

Research on metal nanopowders focuses on the prevention of agglomeration and oxidization of the particles [1], synthesizing powders with small particle sizes [2, 3], and quantity synthesis with a focus on industrializing of the process [4].
Introduction – Nickel Hydroxide

- \( \text{Ni(OH)}_2 \) is used as the cathode of rechargeable Nickel/Metal Hydride (NiMH) Cell batteries and is also used to produce NiO, which is commonly used in glass and ceramic industries. [5]

- Powders of \( \text{Ni(OH)}_2 \) were synthesized using a simple and efficient chemical method.

- The powders were characterized using X-Ray analysis and the crystallite sizes determined using X-Ray peak broadening analysis, as X-Ray peaks become broader due to the effect of small crystallite sizes.

- A Williamson-Hall plot was used to estimate particle size (Fig. 2).
Nickel Hydroxide Powder Preparation

- Ni(OH)$_2$ powders were produced by the chemical reaction between nickel (II) chloride, NiCl$_2$ sodium hydroxide, NaOH, and the reductant hydrazine hydrate, N$_2$H$_4$.

- NiCl$_2$ was dissolved in distilled water was mixed with absolute ethanol and formed a green solution. In a separate beaker, N$_2$H$_4$ and NaOH were thoroughly combined.

- The solution of N$_2$H$_4$ and NaOH was then slowly added to the dissolved NiCl$_2$, resulting in a cloudy green solution. Precipitate formed as soon as the two solutions were mixed.

- The precipitate was filtered, washed, and dried using a standard funnel arrangement, then X-Rayed (Fig. 1).
X-Ray Diffraction Pattern of Nickel Hydroxide Powder

Figure 1. XRD pattern of Ni(OH)$_2$ powder

<table>
<thead>
<tr>
<th>Peak ($\theta$)</th>
<th>001 @ 19.47°</th>
<th>100 @ 33.62°</th>
<th>101 @ 38.85°</th>
<th>102 @ 52.29°</th>
</tr>
</thead>
<tbody>
<tr>
<td>FWHM (deg)</td>
<td>0.78</td>
<td>0.69</td>
<td>0.80</td>
<td>1.16</td>
</tr>
<tr>
<td>FWHM (rad)</td>
<td>0.014</td>
<td>0.012</td>
<td>0.014</td>
<td>0.020</td>
</tr>
<tr>
<td>$B_0\cos\theta$</td>
<td>0.014</td>
<td>0.0115</td>
<td>0.013</td>
<td>0.018</td>
</tr>
<tr>
<td>$\sin\theta$</td>
<td>0.169</td>
<td>0.289</td>
<td>0.333</td>
<td>0.441</td>
</tr>
</tbody>
</table>

Table 1. FWHM calculation of Ni(OH)$_2$ powder
Williamson-Hall Plot of Nickel Hydroxide Powder

Figure 2. Williamson-Hall plot of Ni(OH)$_2$ powder

<table>
<thead>
<tr>
<th></th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Y-intercept of W-H plot (rad)</td>
<td>0.0105</td>
</tr>
<tr>
<td>$k$</td>
<td>1.0</td>
</tr>
<tr>
<td>$\lambda$ (nm)</td>
<td>0.154056</td>
</tr>
<tr>
<td>$k\lambda/Y$-intercept = $L$ (particle size)</td>
<td>19 nm</td>
</tr>
</tbody>
</table>

**Table 2.** Particle size of Ni(OH)$_2$
Introduction - Nickel

- The many chemical and physical properties of nanosize nickel powder include an extremely high surface area per mass, conductive and magnetic properties, and control over the scattering of light.

- Nanosize nickel powders can be commercially utilized today as chemical catalysts, in fuel and solar cells, in paints and polymers, and in optical equipment.

- Stabilizers such as Poly(vinyl pyrrolidone) (PVP) are often used during synthesis for particle protection and are able to decrease particle size [6].
Ni powders were synthesized using a chemical method.

Different amounts of powder can be produced by varying the concentrations of the chemicals.

PVP was added to prevent agglomeration of the particles and to promote small particle sizes.

X-Ray analysis characterized the powders and the Williamson-Hall plot provided an estimate of the particle sizes (Fig 3, 4).
Nickel Nanopowder Preparation

- Preparation of the solution is as follows: 0.5 g of NiCl$_2$ was dissolved in 60 mL of distilled water for Ni$^{2+}$ ions. 0.1 g of PVP was dissolved in the Ni solution.

- In a separate beaker, approximately 1.0 g of a strong base, NaOH, was dissolved in 20 mL distilled water.

- 20 mL of the reductant, N$_2$H$_4$, was added to the beaker containing NaOH; the resulting solution was then added to the solution of NiCl$_2$.

- The solution turned a royal blue color and was allowed to sit overnight.
Nickel Nanopowder Preparation (cont’d)

- During post-processing using sonication or heating (Table 3), the solution became grey, a black precipitate began forming along the sides and bottom of the beaker, and a shiny silver substance coated the sides of the beaker.

- The solutions were left to precipitate overnight.

- The precipitate was filtered, washed, and dried using a standard funnel arrangement, and X-Rayed.
X-Ray Diffraction Pattern of Nickel Powder

Figure 3. XRD Pattern of Ni Powder stabilized by PVP

<table>
<thead>
<tr>
<th>Peak (θ)</th>
<th>111 @ 44.57°</th>
<th>200 @ 51.81°</th>
<th>220 @ 76.41°</th>
<th>311 @ 92.92°</th>
</tr>
</thead>
<tbody>
<tr>
<td>FWHM (deg)</td>
<td>1.12</td>
<td>2.81</td>
<td>3.90</td>
<td>5.73</td>
</tr>
<tr>
<td>FWHM (rad)</td>
<td>0.020</td>
<td>0.049</td>
<td>0.068</td>
<td>0.100</td>
</tr>
</tbody>
</table>

Table 3. FWHM of PVP-protected nickel particles

\[ B_{0} \cos\theta = 0.0185 \quad 0.0441 \quad 0.0534 \quad 0.0689 \]

\[ B_{1}^{†} = 0.0073 \quad 0.0073 \quad 0.0085 \quad 0.0103 \]

\[ B_{r} (B_{r}^2 = B_{0}^2 - B_{1}^2) = 0.019 \quad 0.048 \quad 0.067 \quad 0.11 \]

\[ B_{r}\cos\theta = 0.0176 \quad 0.0432 \quad 0.0526 \quad 0.0689 \]

\[ \sin\theta = 0.379 \quad 0.437 \quad 0.618 \quad 0.725 \]

\[ \dagger B_{1} \text{ is the correction for instrumental broadening} \]
Williamson-Hall Plot of Nickel Powder

Table 4. Particle size of PVP-protected Ni particles

<table>
<thead>
<tr>
<th>Y-intercept of W-H plot (kλ/L)</th>
<th>0.022</th>
</tr>
</thead>
<tbody>
<tr>
<td>k</td>
<td>1.0</td>
</tr>
<tr>
<td>λ (nm)</td>
<td>0.154056</td>
</tr>
<tr>
<td>kλ/Y-intercept = L (particle size)</td>
<td>7 nm</td>
</tr>
</tbody>
</table>

Figure 4. Williamson-Hall plot of PVP-protected Ni particles
## Production Methods

<table>
<thead>
<tr>
<th>Ni Procedures</th>
<th>Trial 1</th>
<th>Trial 2</th>
<th>Trial 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chemical Conditions</td>
<td>NiCl₂: 0.499 g H₂O: 60 mL N₂H₄: 20 mL NaOH: 20 mL</td>
<td>NiCl₂: 0.499 g H₂O: 60 mL N₂H₄: 20 mL NaOH: 20 mL</td>
<td>NiCl₂: 0.499 g H₂O: 60 mL N₂H₄: 20 mL NaOH: 20 mL PVP: 0.5 g</td>
</tr>
<tr>
<td>Post-processing method</td>
<td>Sonication in a bath sonicator</td>
<td>Low heat</td>
<td>Sonication in a bath sonicator</td>
</tr>
<tr>
<td>Resulting particle size</td>
<td>12 nm</td>
<td>19 nm</td>
<td>7 nm</td>
</tr>
</tbody>
</table>

**Table 5.** Comparison of three different methods of Ni powder synthesis
Discussion

- A Williamson-Hall plot indicated that PVP-protected particles had an estimated size of 7 nm, compared to an estimate of 12 nm for non-PVP protected Ni particles.

- A possible formula for the formation of Ni:
  \[ \text{NiCl}_2 \rightarrow \text{Ni}^{2+} + 2\text{Cl}^{-} \]
  \[ \text{Ni}^{2+} + 6\text{N}_2\text{H}_4 \rightarrow \text{Ni} (\text{N}_2\text{H}_4)_6 ^{2+} \]
  \[ \text{Ni} (\text{N}_2\text{H}_4)_6 ^{2+} + 2\text{OH}^- \rightarrow \text{Ni} + \text{N}_2 + 5\text{N}_2\text{H}_4 + 2\text{H}_2\text{O} \]

- The procedures for Ni(OH)$_2$ and Ni are nearly identical, except for the concentrations of the chemicals. One possible explanation for the different results is an insufficient amount of NaOH in the solution that produced Ni(OH)$_2$ (Fig. 5).
Discussion (cont’d)

Figure 5. The Pourbaix diagram for Ni
Conclusions

- This chemical method for synthesis of Ni nanopowders can be slightly modified to produce Ni(OH)$_2$ powders.

- The addition of PVP prevents agglomeration of particles and promotes smaller particle sizes.

- The most notable features of this method are the simple operation, high yield, and small particle sizes.

- Future research on this method include synthesizing nanopowders of various metals and investigating the applications of the metal nanopowders in optical equipment.
Acknowledgements

- TAMS Summer Research Scholarship
- Dr. Teresa D. Golden
- University of North Texas Chemistry Department
References