Synthesis and characterization of nickel hydroxide nanoparticles obtained by chemical deposition method under different precipitation conditions

Y P Vinichenko¹ and E N Sidorova¹

¹Department of functional nanosystems and high-temperature materials, The National University of Science and Technology “MISiS”, Moscow 119991, Russia

Abstract. The aim of this investigation was to produce nickel hydroxide nanoparticles by chemical deposition method using different synthesis conditions such as temperature, pH and washing type. The phase composition was analyzed by X-ray diffractometer, which reveals that all the particles were β-Ni(OH)_2 phase with hexagonal lattice. XRD analysis also allowed determining the coherent scattering region size in order to compare it with particle size. Transmission electron microscopy showed that the Ni(OH)_2 particles have film forms with a thickness of about 1 nm. The surface area was determined using Brunauer-Emmett-Teller method and varies from 2 to 41 m²/gr.

1. Introduction
Nowadays the most promising and high developing field of research is production [1 – 3] and using nanomaterials [4 – 6] due to their unique properties caused by small size of microconstituents. Nano-sized structures with different morphologies (particles, films, tubes) are in demand in such important spheres of human activities as medicine, energy, microelectronics, etc. [7 – 9].

Nickel nanoparticles are widely used in various fields due to its good catalytic, magnetic and tribological properties [10 – 12]. For example using suspension with nano-nickel as oil additives increases enduring quality of engine parts. Also attractive is implementation nanosized nickel in producing recording and storage information systems [13], permanent magnets [11], magnetic cooling systems [11], for directional medicine transport [14], for treatment of cardiovascular disease [15] etc. There are a lot of reactions which are capable of catalyzing by nickel, one of them is water dissociation on hydrogen and oxygen at 300 °C [16]. Moreover, nickel based compounds exhibits high electrochemical performance and corrosion which allow them to find its application in various fields such as electrochemistry and battery technology (nickel - cadmium and nickel - metal hydride batteries) [17].

There are different methods available for the nickel nanoparticles [18] that can in different ways influence on morphology and grain size of final product. Nevertheless the problem of controlling the dispersion and particle shape has not been resolved. Thus, the main aim of this paper is to study the influence of precipitation conditions on the particle size and morphology of nickel hydroxide.

2. Experiment
The deposition process is represented on Figure 1. Nickel hydroxide powders were prepared using chemical precipitation, in which sodium hydroxide (NaOH) was added into nickel nitrate (Ni(NO_3)_2)
solution under different conditions listed in Table 1. There were 8 powders obtained under three different conditions such as four different temperature (15, 20, 30 and 45 °C), three pH (8, 9, 10) and different types of washing (decantation, centrifugation and ultrasonic sound). After the different condition the green precipitate was dried at 50 °C in oven. After the drying process the powder was characterized using different characterization techniques. Phase analysis and sizes of coherent scattering region were determined using X-ray diffractometer Difray 401 (Cr Kα source). Images of nickel hydroxide nanoparticles were obtained on transmission electron microscope LEO 912AB. The values of surface area were carried out using NOVA 1200e.

Figure 1. Schematic representation of precipitation reaction used for production of Ni(OH)₂ under different conditions.

### Table 1. Different approaches used in chemical precipitation.

| Sample | Temperature, °C | pH value | Washing type |
|--------|-----------------|----------|--------------|
| S1     | 15              | 9        | centrifugation |
| S2     | 20              | 9        | centrifugation |
| S3     | 30              | 9        | centrifugation |
| S4     | 45              | 9        | centrifugation |
| S5     | 20              | 8        | centrifugation |
| S6     | 20              | 10       | centrifugation |
| S7     | 20              | 9        | decantation   |
| S8     | 20              | 9        | ultrasonic    |

3. Results and discussion

The characteristic results reveal the information about the nanoparticles. XRD analysis confirms that the prepared nanoparticles are found to be β-Ni(OH)₂ phase with hexagonal lattice. From analyzing the broadening of reflections 20 °C was found to be the best temperature for obtaining the higher dispersed particles (Figure 2). Similarly the lowest grain sizes were found at pH 9 and centrifugation was found to be the best method of washing to obtain the nanoparticle.

The coherent scattering region sizes were determined by XRD analysis for lattice planes (100) and (101), and the obtained results are represented in Table 2.
Table 2. Sizes of CSR for different planes.

| Sample | S1   | S2   | S3   | S4   | S5   | S6   | S7   | S8   |
|--------|------|------|------|------|------|------|------|------|
| Size,  | (100)| 38.8 | 15.4 | 33.8 | 29.7 | 24.9 | 28.7 | 35.7 |
| nm     | (101)| 4.0  | 5.2  | 7.9  | 5.6  | 4.5  | 4.8  | 5.2  |

Morphology and particle size of powders were studied using transmission electron microscopy. Figure 3 shows TEM images of Ni(OH)$_2$ samples under different pH value. The TEM analysis illustrates that particles are like aggregates of thin films with the average particle size of 20 nm, with thickness of 1 nm.

![Figure 3. TEM image of Ni(OH)$_2$ nanoparticles at different pH](image)

**Figure 3.** TEM image of Ni(OH)$_2$ nanoparticles at different pH (a) pH 8, (b) pH 9 and (c) pH 10.

Table 3 introduces the surface area that was determined using Brunauer-Emmett-Teller method. Taking account of cylindrical form of particles the surface area can be related to the average particle size by equation (1).

\[
S = (2d + 4h) \cdot \frac{d}{1} \cdot h^{-1} \cdot \rho^{-1}
\]

Where 
- d – average diameter, m;
- h – average altitude, m;
- \(\rho\) – density of Ni(OH)$_2$, gr/m$^3$. 
Table 3. Surface area of the prepared samples

| Sample | S1 | S2 | S3 | S4 | S5 | S6 | S7 | S8 |
|--------|----|----|----|----|----|----|----|----|
| S, m²/gr | 6  | 2  | 9  | 41 | 4  | 27 | 39 | 3  |

BET analysis reveals that the highest area surface of 41 m²/gr was obtained for Ni(OH)₂ nanoparticles prepared at 45 °C with maintaining pH 9 for precipitation and collection of particles by centrifugation. Low values of surface area can be explained by the fact that particles form aggregates. So the Figure 4 shows the model of particle structure: thin films that consist of hexagonal lattices closely join to each other forming a bar structure.

![Figure 4. The model of particle structure](image)

Using TEM images the particle size distribution was plotted in Figure 5 for samples obtained under different pH value. All powders have quit narrow size distribution, which indicates that particles are seemed to be monodispersed.

![Figure 5. Particle size distribution of Ni(OH)₂ nanoparticles at different pH](image)

Comparing the average particle size obtained from TEM images and sizes of coherent scattering region determined by XRD analysis it can be assumed that one particle contain one CSR.
4. Conclusions
Synthesis of nickel hydroxide nanoparticles was obtained using different precipitation conditions. After characterizations of powders with different techniques we determined that all samples consist of $\beta$-Ni(OH)$_2$ phase with hexagonal lattice, and particles look like thin films. The similarity of sizes from TEM images and CSR leads to the fact that one particle consists of one coherent scattering region. The equation between cylindrical particles size $s$ and surface area $A$ was also deducted. Low values of surface area can be explained by the fact that particles form aggregates.

References
[1] Buesser B and Pratsinis S 2012 J. Rev. Chem. Biomol. Eng. 3 103 – 127
[2] Keshril A and Agarwal A 2012 J. Nanoscience and Nanotechnology Letters 4 228 – 250
[3] Wankhede P, Sharma P and Jha A 2013 J. of Engineering Research and Applications 3 1664 – 69
[4] Yang S and Dengteng G 2014 J. Materials Science and Engineering 3 1 – 7
[5] Froudakis G 2011 J. Materials Today 14 324 – 328
[6] Baati T, Bourasset F and Gharb N 2012 J. Biomaterials 33 4936 – 434
[7] Zhang Y, Liu X, Ru C, Zhang Y, Dong L and Sun Y 2011 J. of Microelectromechanical systems 20 959 – 967
[8] Bakry R, Vallant R, Najam-ul-Haq M, Rainer M, Szabo Z and Huck C 2007 J. Nanomedicine 2 639 – 649
[9] Zhenan B, Vosgueritchian M, Wang H, Koleilat G, Liu N and Kim M 2013 J. GCEP Report 5 1 – 16
[10] Singh S K and Xu Q 2010 J. R. Soc. 46 6545 – 47
[11] Wang Z, Kuok M, Ng S, Lockwood D, Cottam M, Nielsch K, Wehrspohn R and Gosele U 2002 J. Phys. Rev. Lett. 89 1
[12] Qiu S, Zhou Z, Dong J, Chen G 2001 J. of Tribology 123 441 – 443
[13] Nielsch K, Wehrspohn R, Barthel J, Kirschner J and Gosele U 2001 J. Applied Physics Letters 73 1360 – 62
[14] McHenry M and Laughlin D 2000 J. Acta Materialia 48 223 – 238
[15] Al-Qubaisi M, Rasedee A, Flaifel M, Ahmad S, Hussein-Al-Ali S, Hussein M, Eid E, Zainal Z, Saeed M, Ilowefah M, Fukurazi S, Isa N and Zowalaty M 2013 J. Nanomedicine 8 2597 – 508
[16] Kang G, Gillespie P, Gunnison a, Moreira A, Tchou-Wong K and Chen L 2010 J. Environmental Health Perspectives 119 176 – 181
[17] Fan D and White E R 1998 J. Electrochem. Soc. 145 34 – 39
[18] David S, Lockwood J, Bock C, Barry R 2015 J. R. Soc. A 471 1 – 65