



## Effect of Surfactants on the Synthesis and Characteristics of Nickel Hydroxide Nanoparticle

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### ABSTRACT

Nickel hydroxide has a vital role in various applications, especially as a support material for energy storage materials. Nickel hydroxide can be synthesized through the hydroxide precipitation method. However, the product formed by this method may be large or more than 100 nm because the agglomeration step can occur easily. This present work aims to study the effect of surfactant types in the synthesis and characterization of nickel hydroxide nanoparticle. Nickel sulfate ( $\text{NiSO}_4$ ) solution was used as a precursor solution, while 5M sodium hydroxide (NaOH) solution was used as a precipitation agent. The surfactants studied were alkyl benzene sulfonate (ABS), sodium dodecyl sulfate (SDS), cetyltrimethylammonium bromide (CTAB), and polyvinylpyrrolidone (PVP). The nickel hydroxide synthesis process was carried out at 50 °C for 1 hour. The surfactant concentration used was at the critical micelle concentration (CMC), where the CMC for ABS, SDS, CTAB, and PVP were 0.01; 0.05; 3; and 0.5 %w/v, respectively. The synthesis of nickel hydroxide nanoparticle was carried out successfully precipitated almost 100% of  $\text{Ni}^{2+}$  ions. The product characterization that has been carried out shows that ABS surfactant produces the best nickel hydroxide nanoparticle product where the particle size is 3.12–4.47 nm.

**Keywords:** agglomeration; CMC; nickel hydroxide; nanoparticles; surfactant

### ABSTRAK

*Nikel hidroksida memiliki peran penting dalam berbagai aplikasi, terutama sebagai bahan pendukung untuk material penyimpanan energi. Nikel hidroksida dapat disintesis melalui metode*

*presipitasi hidroksida. Akan tetapi, produk yang terbentuk dengan metode ini dapat berukuran besar atau lebih dari 100 nm karena tahap aglomerasi dapat terjadi dengan mudah. Penelitian ini bertujuan untuk mempelajari pengaruh jenis surfaktan pada sintesis dan karakterisasi nanopartikel nikel hidroksida. Larutan nikel sulfat ( $\text{NiSO}_4$ ) digunakan sebagai larutan prekursor sedangkan larutan natrium hidroksida ( $\text{NaOH}$ ) 5M digunakan sebagai agen presipitasi. Jenis surfaktan yang dipelajari adalah alkyl benzene sulfonate (ABS), sodium dodecyl sulfate (SDS), cetyltrimethylammonium bromide (CTAB), and polyvinylpyrrolidone (PVP). Proses sintesis nikel hidroksida berlangsung pada suhu  $50^\circ\text{C}$  selama 1 jam. Konsentrasi surfaktan yang digunakan berada pada critical micelle concentration (CMC) dengan nilai CMC untuk ABS, SDS, CTAB, dan PVP adalah 0,0; 0,05; 3; dan 0,5 %b/v, secara berurutan. Sintesis nanopartikel nikel hidroksida yang dilakukan berhasil mengendapkan hampir 100% ion  $\text{Ni}^{2+}$ . Karakterisasi produk yang telah dilakukan menunjukkan bahwa penggunaan surfaktan ABS menghasilkan produk nanopartikel nikel hidroksida yang terbaik dengan ukuran partikel sebesar 3,12–4,47 nm.*

*Kata kunci: aglomerasi; CMC; nanopartikel; nikel hidroksida; surfaktan*

## 1. Introduction

Metal-based nanoparticles are essential and promising materials to be developed in the future. This condition is in line with the development of technology that requires nanoparticles as its constituent material. These materials have great potential and utilization to be applied in various fields such as drug delivery systems, catalysts, sensors, electronic equipment, biomaterials, and energy storage (Castro et al., 2014; Hafeez et al., 2020; Kobayashi et al., 2016; Ndolomingo et al., 2020; Slavin et al., 2017; Yarakı and Tan, 2020). The wide use of metal-based nanoparticles cannot be separated from the unique properties of these materials and can be engineered according to the application needs. Various studies related to this material have shown how the mechanical, thermal, magnetic, electrical, optical, and catalytic properties of metal-based nanoparticles are more attractive compared to the same material in bulk size (Li et al., 2007; Mahaleh et al., 2008; Xin et al., 2007).

In the process of synthesizing metal-based nanoparticles, one of the biggest challenges

is to obtain a material in nanometer and homogeneity. It is not easy because based on the synthesis mechanism, there is an agglomeration step that can make the material size large (more than 100 nm). In the process of material synthesis, especially the formation of metal crystals, agglomeration is a condition when two or more particles are in contact with each other and stick together for a long time (Lewis, 2017). As a result, the particles will grow and produce particles of a larger size. In addition, particle agglomeration will reduce the free energy surface with increasing size and reduce the particle surface area (Gosens et al., 2010). Therefore, the formation of agglomerates from the particles is undesirable and is strictly avoided.

To inhibit the agglomeration process, various methods can be used in the synthesis of nanoparticles, such as the use of sonication processes (Kaur et al., 2017; Mahbubul et al., 2015; Pradhan et al., 2016; Taurozzi et al., 2011) and the addition of surfactants (Kiani et al., 2010; Loosli and Stoll, 2017; Shah et al., 2019; Tientong et al., 2014). The addition of surfactants is the most common method and

is easy to apply in the nanoparticle synthesis. Surfactant is a molecule that has a polar group (hydrophilic, water-loving) in the head section and a non-polar group (hydrophobic, water-hating) in the tail section (Morsy, 2014). There are six roles of surfactants in nanoparticle synthesis, namely as (1) dispersing agent, (2) structural regulator, (3) covering agent, (4) weak reducing agent, (5) agent oxidation etching, and (6) ion exchange (Song et al., 2021). Consequently, the addition of this surfactant will affect the shape, size, and properties of the synthesized nanoparticles (Bakshi, 2016; Song et al., 2021).

In this study, the use of surfactants will be investigated more deeply in the nickel hydroxide nanoparticle synthesis process, especially its effect on the morphology (shape) and size of the precipitate formed. Nickel hydroxide is a nickel-based compound that plays a vital role in its application for energy storage, such as rechargeable batteries, fuel cells, and electrochemical capacitors (Hall et al., 2015; Nickel Institute, 2015; Song et al., 2002; Wanta et al., 2020a). Nickel hydroxide can be synthesized through several methods, such as precipitation, hydrolysis, electrochemical, sol-gel, hydrothermal, or chimie douce methods (Ramesh and Kamath, 2006; Rane et al., 2018; Wanta, 2020b). The main problem that generally occurs in the synthesis of nickel hydroxide is that the solids (crystals) formed, primarily through chemical precipitation methods, are generally "large" particles with more than 100 nm.

For example, in our previous study, Wanta et al., (2019) and Lyman (2020) studied and succeeded in synthesizing the nickel hydroxide from a Ni/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub> spent catalyst extract solution. However, the product

formed is agglomerated crystals. Based on the mechanism of the chemical precipitation method, the agglomeration step is unavoidable, especially for the spontaneous formation of particles such as nickel hydroxide. In addition, the agglomeration is caused by the high surface energy when the particles are dispersed, causing van der Waals forces where the dispersed particles tend to form agglomerates with larger sizes that are more stable. For this case, considering the application of nickel hydroxide for energy storage, the formation of "large-size" nickel hydroxide is an undesirable condition.

The solution to solve these problems is to utilize the addition of surfactants during the particle synthesis process. The addition of surfactants can control the reaction in the precipitation process because it has stable properties in the formation of nanoparticles (Singh and Sharma, 2010). The addition of surfactants in the precipitation process can reduce the surface energy to prevent agglomeration due to the van der Waals forces (Dahman et al., 2017). Therefore, this study will focus on the effect of adding surfactants in the synthesis and characterization of the nickel hydroxide formed.

## 2. Research Methodology

### 2.1 Materials

Nickel sulfate hexahydrate (NiSO<sub>4</sub>·6H<sub>2</sub>O, technical grade) was employed to prepare the precursor solution. As a precipitating agent, sodium hydroxide (NaOH, technical grade) was added in the form of a solution with a solution concentration of 5M. In addition, the surfactants used and varied for this study were alkyl benzene sulfonate (ABS, Subur Kimia Jaya, technical grade), sodium dodecyl

sulfate (SDS, Merck, >95%), cetyltrimethylammonium bromide (CTAB, Sigma–Aldrich, ≥98%), and polyvinylpyrrolidone (PVP, Merck). All materials were dissolved using demineralized water.

## 2.2 Procedures

### 2.2.1 Preparation of Precursor Solution

The precursor solution is a solution as a nickel source in the synthesis of nickel hydroxide nanoparticle. This solution was prepared by dissolving 30 grams of solid nickel sulfate hexahydrate ( $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ ) in 1 L of demineralized water (called as the mother liquor). From that solution, 500 ml of the solution was taken and poured into a 2 L glass reactor. Instead of the reactor, a series of tools used consisted of a waterbath, condenser, thermostat, thermometer, and stirrer. After the tools were assembled, the operating temperature was set at 50 °C. When the temperature was reached, 5M NaOH solution was dropped into the mother liquor until the pH of the solution reached 6. The solid phase formed was then separated from the liquid phase (supernatant) by filtration under vacuum conditions. This supernatant formed was the precursor solution for this study. The concentration of  $\text{Ni}^{2+}$  ions in the precursor solution (supernatant) was analyzed using a UV–vis spectrophotometer (Spectronic SP–2100 series) which dimethylglyoxime was used as a complexing agent.

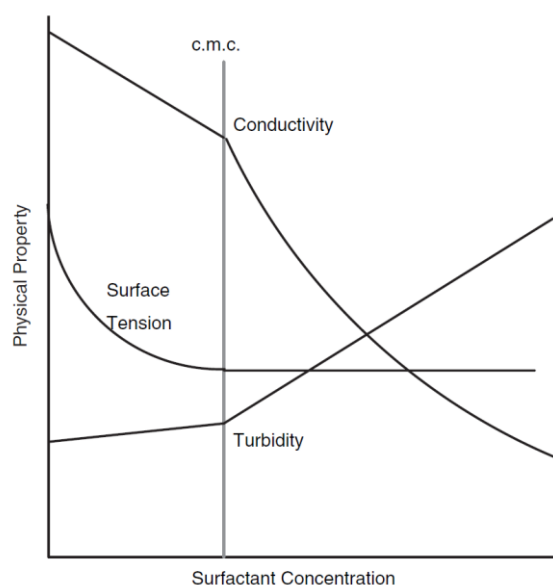
### 2.2.2 Determination of Critical Micelle Concentration (CMC)

This procedure was carried out to determine the surfactant concentration to be applied in the main process, i.e. the synthesis of nickel hydroxide nanoparticle. The determination of CMC was carried out in all

types of surfactants studied in this work. The prepared precursor solution was mixed with surfactant at a particular concentration to form a homogeneous mixture. The ranges of surfactant concentrations used for the determination of CMC are presented in Table 1.

**Table 1.** Variation of surfactant concentration for determination of CMC

Surfactant Type	Variation of surfactant concentration, %w/v
ABS	0 – 0.2
SDS	0 – 0.5
CTAB	0 – 10.0
PVP	0 – 5.0



**Figure 1.** Graphical form of the basis for determining critical micelle concentration (CMC) (Myers, 2006)

The tests on a mixture of precursor and surfactant solutions were conducted by observing two parameters, i.e. surface tension and turbidity. The surface tension measurement of the mixture was carried out using a Du–Nouy tensiometer (KRÜSS Model K6). Meanwhile, the turbidity was measured using a turbidity meter (EUTECH Instrument

TN-100). The measurement results of these two parameters were then plotted on a graph. The determination of CMC was carried out by comparing the form of the graph obtained in this study with the graph of the basis for determining the CMC presented in Figure 1.

### 2.2.3 Synthesis of Nickel Hydroxide Nanoparticle

The precursor solution that has been made was taken 500 mL and poured into a 2 L reactor. Then, the surfactant was mixed into the precursor solution according to the CMC that has been determined in the previous step. The precursor and surfactant solutions were mixed using a magnetic stirrer for 10 minutes. The equipment used to synthesize nickel hydroxide nanoparticle was the same as that used when preparing the precursor solution. This process was carried out at a constant temperature of 50 °C. After the operating temperature was reached, 5M NaOH solution was dripped into the reactor until the pH of the mixture became 10. When the pH of the mixture is reached, the precipitation process will take place at 50 °C for 1 hour. The solids formed during the precipitation process were separated from the liquid phase using the centrifugation method at 6,000 rpm for 10 minutes. The liquid phase was analyzed for the final Ni<sup>2+</sup> ions content using a UV-vis spectrophotometer. The separated solid was washed using water several times then dried at 100 °C to a constant mass. These dry solids were characterized using several instruments, such as X-ray Fluorescence (XRF, Epsilon XLE PANalytical, Netherlands), X-ray Diffraction (XRD, Bruker D8 Advance, Germany), Transmission Electron Microscope (TEM,

Hitachi HT7700, Japan), and the Particle Size Analyzer (PSA, Horiba SZ-100, Japan).

## 2.2 Data Analysis

The percentage decrease in the concentration of Ni<sup>2+</sup> ions in the liquid phase was calculated using Equation (1).

$$\% \text{Reduction} = \frac{C_o - C_i}{C_o} \times 100\% \quad (1)$$

where %Reduction is the percentage reduction of Ni<sup>2+</sup> ions in the liquid phase (%), C<sub>o</sub> is the initial Ni<sup>2+</sup> ions concentration in the liquid phase (ppm), C<sub>i</sub> is the final Ni<sup>2+</sup> ion concentration in the liquid phase (ppm).

## 3. Results and Discussion

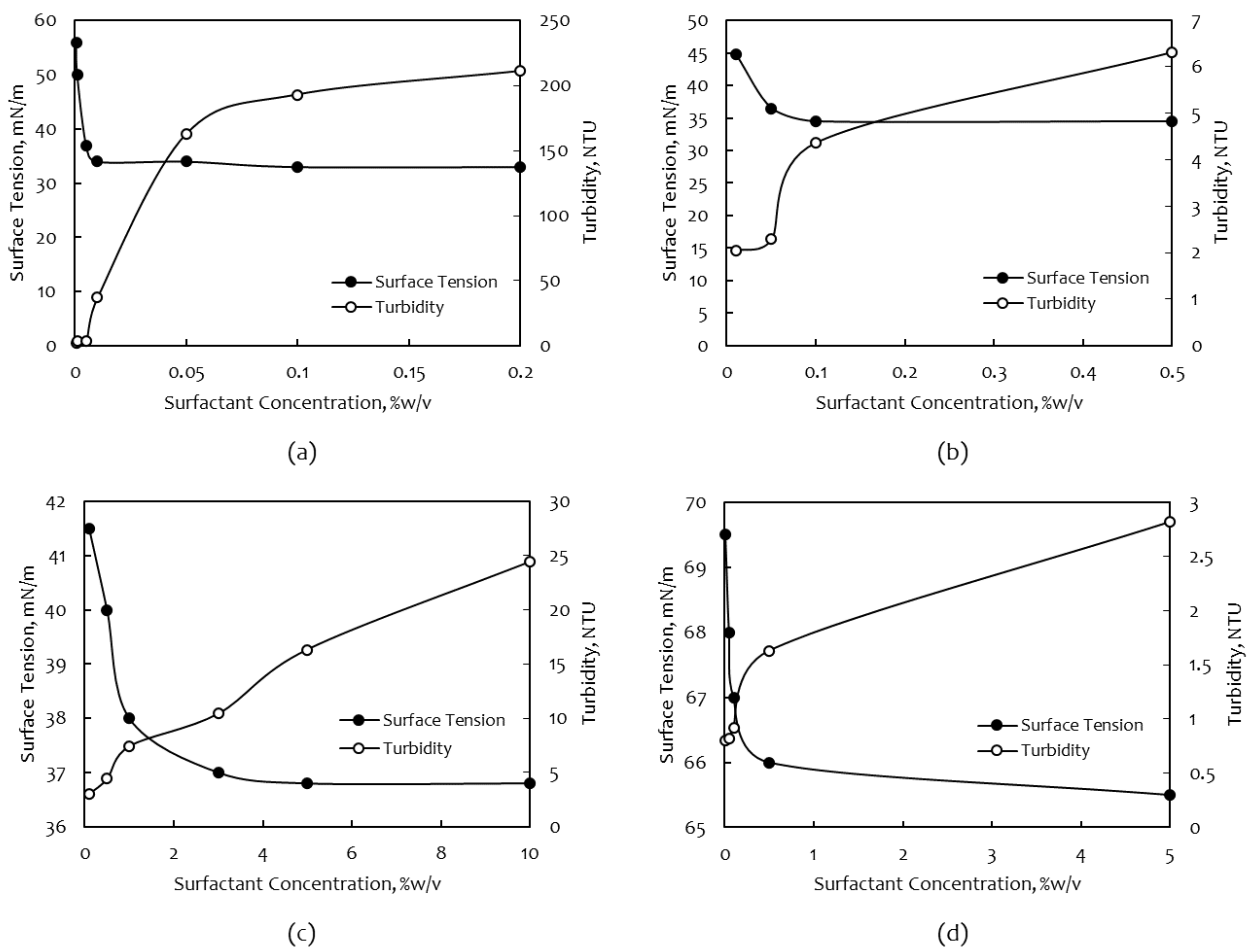
### 3.1. Determination of Critical Micelle Concentration (CMC)

When the surfactant is added to a precursor solution, the hydrophilic group will be adsorbed into the particles while the hydrophobic group will form an aggregate that covers solution's surface. The hydrophilic group with an electrostatic charge can cause repulsion between particles so that agglomeration does not occur. This aggregation process is known as micellization and occurs when the surfactant concentration is in minimum condition or commonly known as the critical micelle concentration (CMC). In the nickel hydroxide nanoparticle synthesis, the determination of CMC becomes a crucial thing to do. At that concentration, the agglomeration process between particles can be avoided or minimized so that the possibility of nickel hydroxide formed at the nanometer size will increase. In this study, the determination of CMC in a mixture of precursor solution and surfactant at various concentrations was conducted by observing

two parameters, namely surface tension and turbidity. The results of observations for the four types of surfactants are presented in Figure 2. Based on the observations as presented in Figure 2 and compared with Figure 1, it was found that the CMC for each surfactant is 0.01%w/v for ABS, 0.05%w/v for SDS, 3%w/v for CTAB, and 0.5%w/v for PVP.

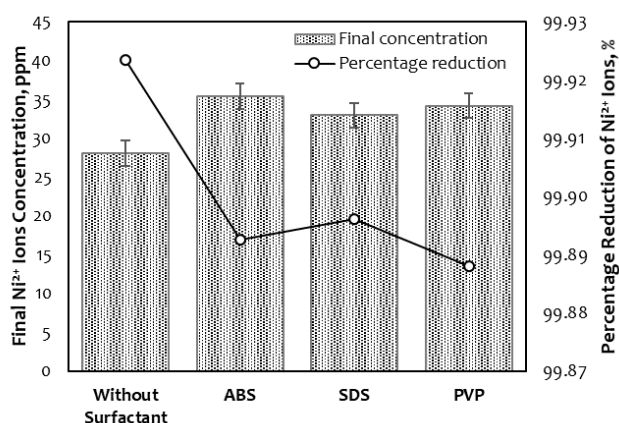
The phenomenon of agglomeration in the precipitation process is caused by the small (size) dispersed particles. That condition makes the particles unstable and have high surface energy so that the particles tend to agglomerate with other particles to obtain stability. The addition of surfactant at the

proper concentration (CMC) can reduce the surface energy. The surface tension on the particles will be constant when the surfactant concentration is added after passing through the CMC. However, if the addition of surfactant is below the CMC, the surfactant is not sufficient to protect the surface of the particles because the surface tension of the particles is still high. As a result, the particles will still agglomerate. Therefore, for the synthesis and characterization of nickel hydroxide nanoparticle, the surfactant used was at CMC conditions as obtained in Figure 2.



**Figure 2.** Determination of CMC using surfactants (a) ABS, (b) SDS, (c) CTAB, and (d) PVP

### 3.2. The Effect of Surfactant Type on Nickel Hydroxide Nanoparticle Synthesis



**Figure 3.** The effect of surfactant type on the reduction of Ni<sup>2+</sup> ions concentration in the liquid phase where the surfactant concentration used is 0.01%w/v for ABS, 0.05%w/v for SDS, and 0.5%w/v for PVP

Based on Figure 3, the addition of surfactant gives the percentage reduction of Ni<sup>2+</sup> ions which is not significantly different from without surfactant. The synthesis process was able to precipitate Ni<sup>2+</sup> ions almost 100% or more precisely around 99.90±0.02%. The phenomenon shows that the type of surfactant does not affect the precipitation process in general because the number of Ni<sup>2+</sup> ions that were precipitated does not differ either without or with the surfactants. However, the addition of this surfactant will affect on the quality of the solid formed. The explanation about this will be discussed in the next section.

### 3.3. Characterization of Nickel Hydroxide Nanoparticle

Nickel hydroxide formed from each variation of the experiment was analyzed with various instruments so that the characteristics of that product could be known. First, the product's composition was analyzed using X-

ray Fluorescence (XRF) instrument and the analysis results are presented in Table 2.

**Table 2.** The composition of nickel hydroxide formed

Element	Composition, %wt				
	Without surfactant	ABS*	SDS*	CTAB*	PVP*
Ni	96.41	94.85	95.11	90.29	95.20
Al	0.18	0.08	0.069	–	0.08
S	0.82	3.47	2.56	3.89	2.98
Si	1.26	1.03	1.20	0.98	1.15
P	0.29	0.24	0.24	0.24	0.23
Ca	0.78	0.16	0.17	0.16	0.13
Br	–	–	–	4.18	–

\*ABS, SDS, CTAB, and PVP are surfactant.

Based on Table 2, the purity of all precipitated products has reached more than 90%. Compared with our other studies (Wanta et al., 2019; 2020b), the purity obtained increased significantly or more than three times. The highest product purity is obtained when the precipitation process is carried out without surfactants, which is 96.41%. This increase is due to the product washing step. Product washing is an essential step because it can dissolve nickel sulfate (NiSO<sub>4</sub>) impurities that are trapped in the surfactant template and also precipitated.

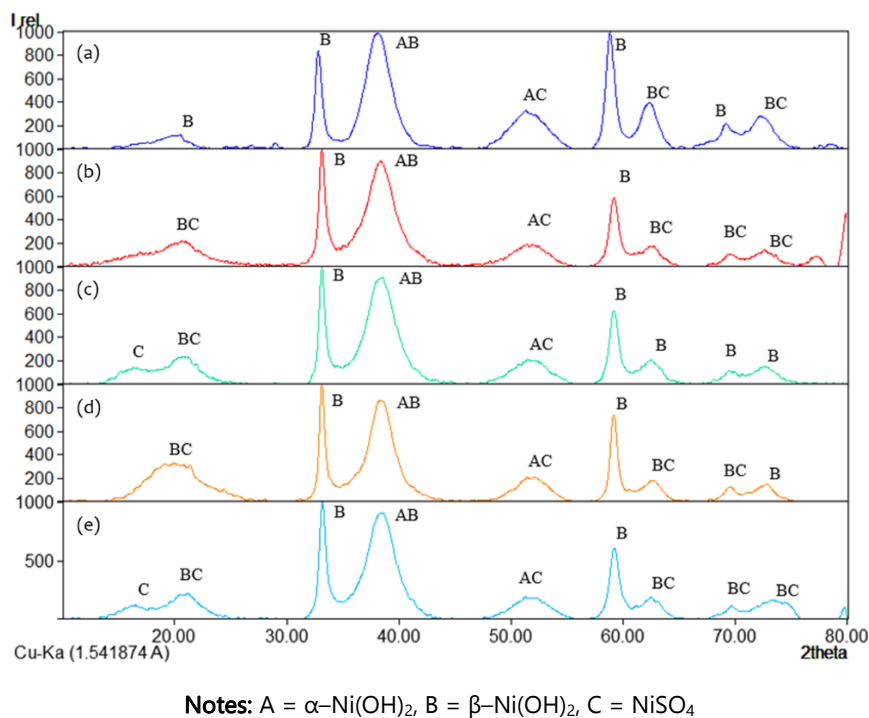
However, the experimental results show that the use of surfactants tends to reduce the purity of the product. It can be observed that all products which were produced using surfactants have lower purity than the product which were produced without surfactants. The biggest impurity in the product is sulfur (S) where is in the range of 2.56–3.89%wt. This element of sulfur indicates that the NiSO<sub>4</sub> compound is still attached to the nickel hydroxide product even though it has been washed several times. To strengthen these results, the crystalline phase of the product was also analyzed using an X-ray Diffraction (XRD) instrument. The results of

the analysis are presented in Figure 4 and the results have been processed using Match! software.

In general, the XRD of each experiment variations have a uniform pattern either without or with surfactant. The XRD pattern obtained shows that there are three crystalline phases formed, namely  $\alpha$ -Ni(OH)<sub>2</sub> (peak A),  $\beta$ -Ni(OH)<sub>2</sub> (peak B), and NiSO<sub>4</sub> (peak C). The presence of NiSO<sub>4</sub> crystals in the XRD pattern further confirms that there are still impurities in the product. When compared to the other two crystals, the peak intensity of NiSO<sub>4</sub> is lower than the peak intensity of the  $\alpha$ -Ni(OH)<sub>2</sub> and  $\beta$ -Ni(OH)<sub>2</sub> crystals. The low peak intensity of NiSO<sub>4</sub> indicates that the presence of impurities, especially elemental sulfur in the product is low and quantitatively, it can be expressed through the results of XRF analysis in Table 2.

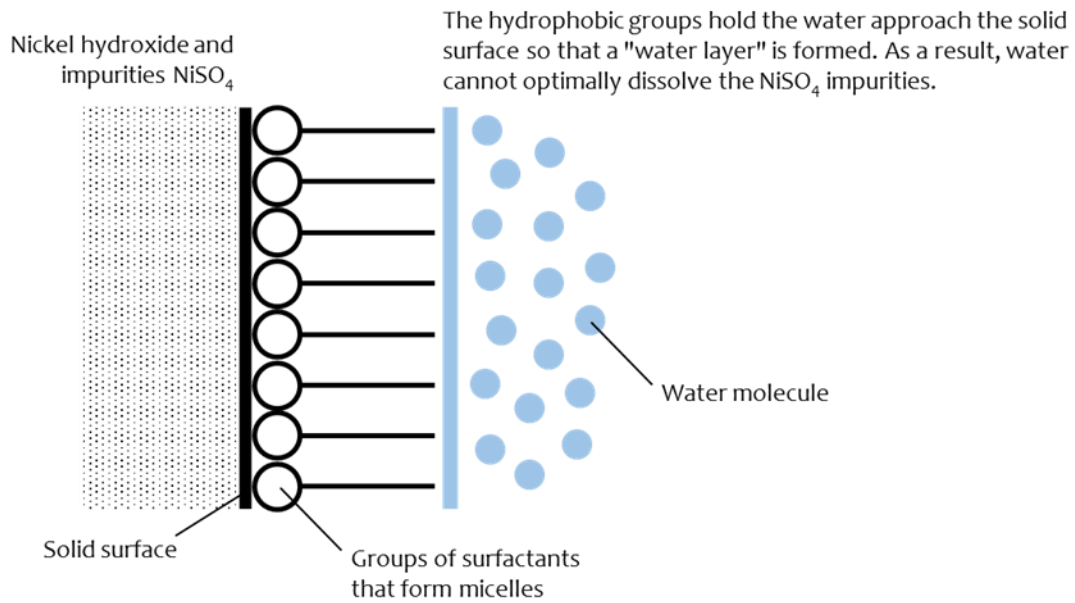
The presence of NiSO<sub>4</sub> impurities in the product occurs because NiSO<sub>4</sub> is still trapped

in the solid and cannot dissolve in water during washing. The template from micelle is formed by the surfactant enveloping the nickel hydroxide particles. The hydrophobic groups in the surfactants cause water to form a thin layer around the surface of the micelle template so that it is difficult for water to diffuse to the surface of the nickel hydroxide particles. The illustration can be seen in Figure 5. The indication of the formation of micelle templates on nickel hydroxide particles is further strengthened by the results of product analysis using Transmission Electron Microscope (TEM) instrument. The results of the analysis are presented in Figure 6. In Figure 6, there is a layer (indicated by arrows) indicating the formation of a micelle template. The removal of this template can be done by several methods, such as calcination, sonication, and ozonolysis (Hadden et al., 2019; Jabariyan and Zanjanchi, 2012).

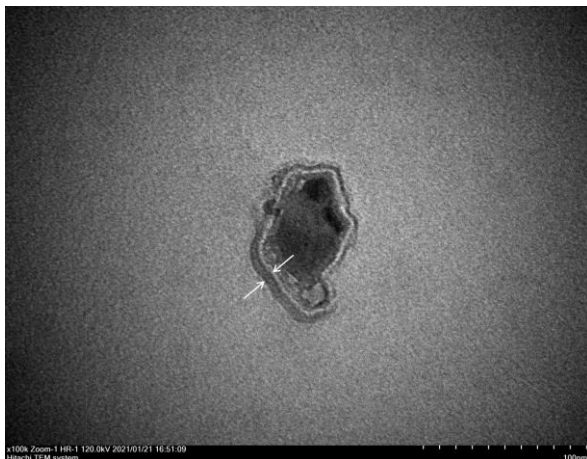


**Figure 4.** XRD pattern of nickel hydroxide nanoparticle (a) without surfactant and using (b) ABS, (c) SDS, (d) CTAB, and (e) PVP





**Figure 5.** Illustration of the phenomenon of  $\text{NiSO}_4$  impurity trapping in nickel hydroxide



**Figure 6.** Micelle template formed on nickel hydroxide nanoparticle

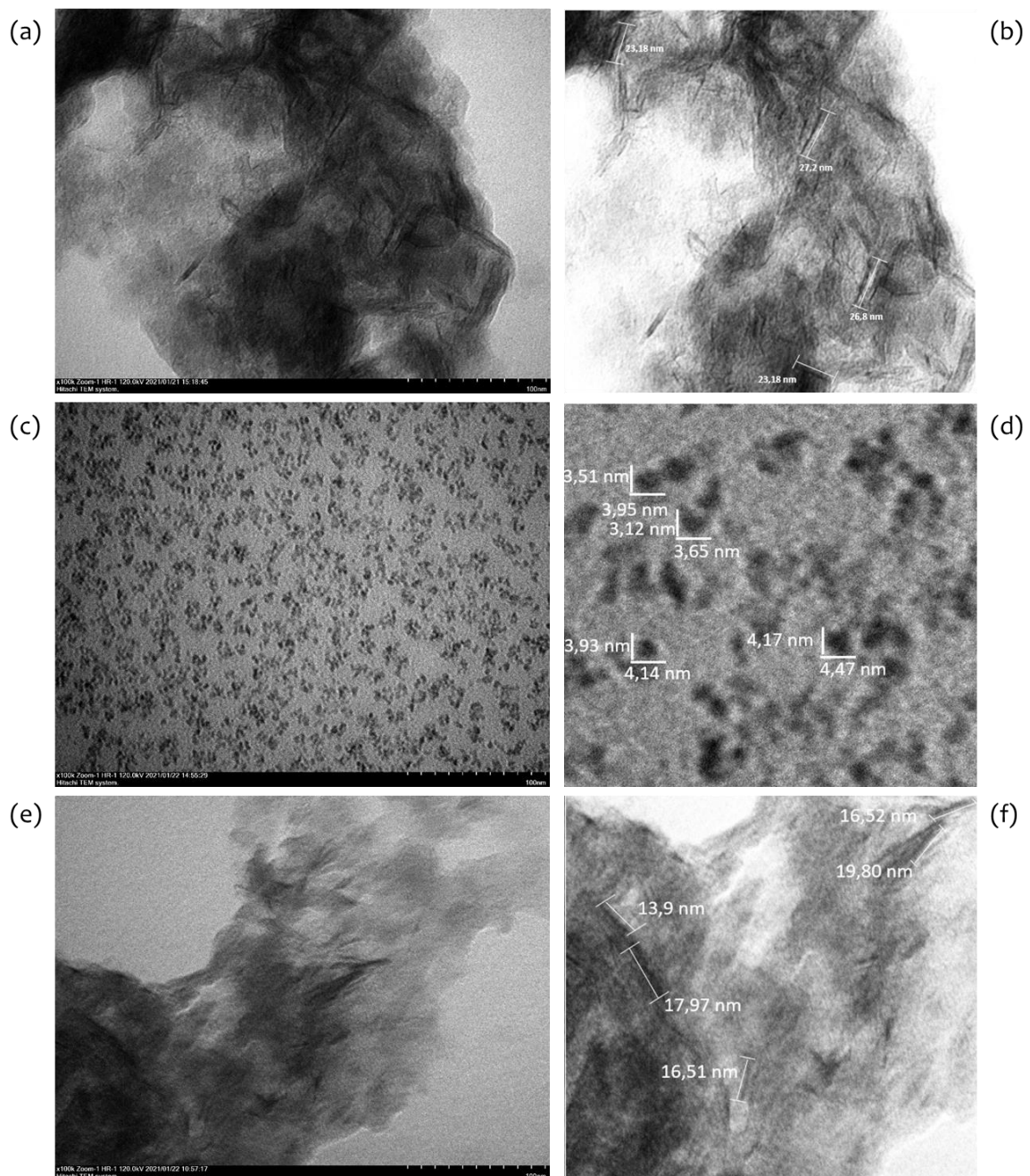
The formation of nickel hydroxide nanoparticle precipitates was further strengthened by the XRD measurement results (Figure 4). Figure 4 shows that the highest peaks are dominated by nickel hydroxide crystals, both in the  $\alpha\text{-Ni(OH)}_2$  and  $\beta\text{-Ni(OH)}_2$ . At  $2\theta$  angles around  $39.0^\circ$  and  $51.9^\circ$  nickel hydroxide is formed in the  $\alpha\text{-Ni(OH)}_2$  while  $\beta\text{-Ni(OH)}_2$  is found at  $2\theta$  angles around  $19.3^\circ$ ,  $33.1^\circ$ ,  $38.6^\circ$ ,  $59.1^\circ$ ,  $62.4^\circ$ ,  $69.5^\circ$ , and  $72.8^\circ$ . It indicates that the synthesis process carried out tends to form  $\beta\text{-Ni(OH)}_2$ . It is because the

precipitation process is carried out above room temperature (i.e.  $50^\circ\text{C}$ ) so that the formation of  $\beta\text{-Ni(OH)}_2$  will be more stable. The formation of  $\alpha\text{-Ni(OH)}_2$  crystals will be more formed when the operating conditions are set at room temperature or lower temperatures (Hall et al., 2015). The formation of  $\beta\text{-Ni(OH)}_2$  crystals is highly desirable because in this phase, nickel hydroxide is widely applied in various fields, especially energy storage. The shape and size of the nickel hydroxide particles produced in this study can be seen in Figure 7. The particle size was evaluated using ImageJ software.

Figure 7 a and b show nickel hydroxide particles were formed without surfactant dan have needle-like shape. The size of particles is in the range of  $23.18\text{--}27.2\text{ nm}$ . This form of nickel hydroxide particles was also found in the research conducted by (Jayashree et al., 2000), with darker spots on needle-shaped particles indicating the  $\beta\text{-Ni(OH)}_2$  phase. However, the product formed still undergoes agglomeration. The analysis results of nickel hydroxide particles with various surfactants

can be seen in Figure 7 c–j. In the variation of ABS surfactant (Figure 7 c dan d), the resulting particles are spherical with a size range of 3.12–4.47 nm which are relatively evenly dispersed. Figure 7 e–h show the variation of SDS and CTAB surfactants in the needle-like shape with a size range of 13.9–19.8 nm for SDS surfactants and 15.37–22.94 nm for CTAB surfactants. A similar form of  $\text{Ni}(\text{OH})_2$  was also

reported in a study (Pradhan et al., 2013) for the addition of SDS surfactant and (Li et al., 2012) for the addition of CTAB surfactant. Figure 7 i and j show the form of nickel hydroxide with PVP surfactant in a spherical shape with a size range of 8.41–21.05 nm. A similar form is also shown in another study (Couto et al., 2007).



**Figure 7.** Morphology and size of nickel hydroxide nanoparticle (a, b) without surfactant and using (c, d) ABS, (e, f) SDS

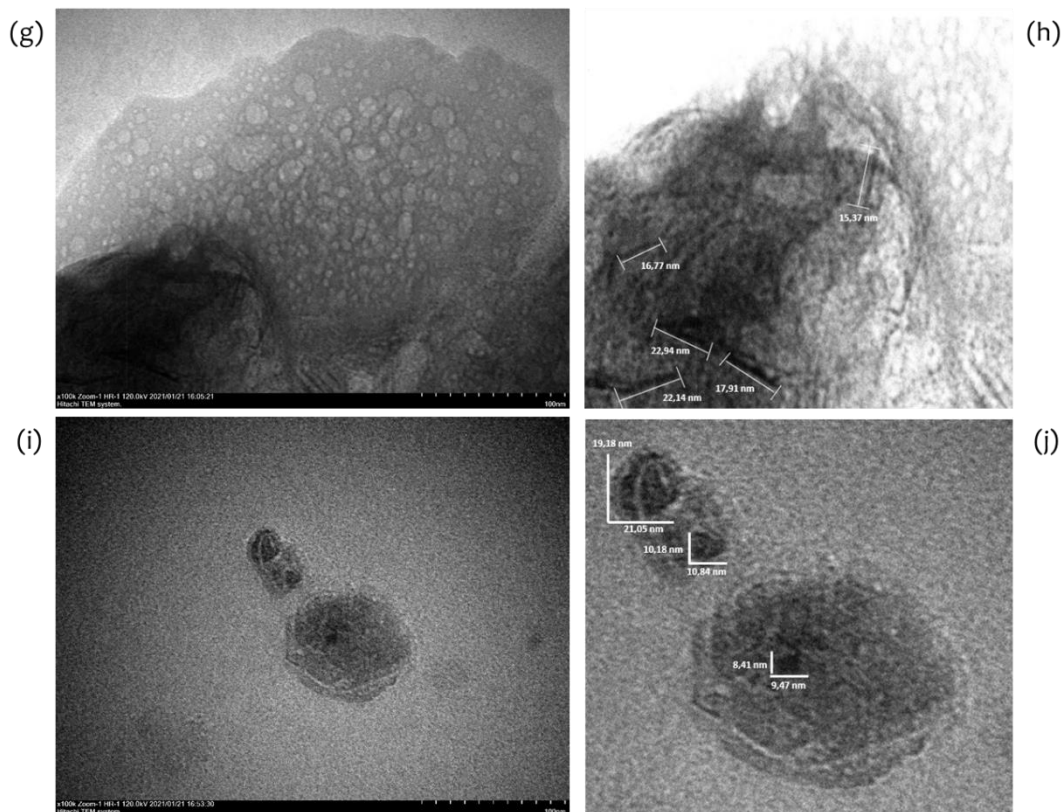


Figure 7. Morphology and size of nickel hydroxide nanoparticle (g, h) CTAB, and (i, j) PVP (cont.)

Based on the experimental data, the use of ABS surfactant produces the smallest particle size and no agglomeration occurs. It is characterized by the formation of spherical particles that are evenly distributed. ABS surfactant is a type of anionic surfactant that can provide a greater effect in reducing particle size. It happens because the negative charge on the hydrophilic group provides a better interaction in the formation of micelles with positively charged nickel hydroxide particles. In contrast, in cationic surfactants, the positive charge on the hydrophilic group cannot provide a strong bond in the formation of micelles with nickel hydroxide particles (Giarola et al., 2014).

#### 4. Conclusions

In this study, surfactants were applied in the synthesis of nickel hydroxide. It is done to

make particles with a nanometer size. The use of surfactants at critical micelle concentrations (CMC) can inhibit the formation of agglomerated products. The experimental results showed that micelle formation in each surfactant occurred when the surfactant concentration was 0.01%w/v for ABS, 0.05%w/v for SDS, 3%w/v for CTAB, and 0.5%w/v for PVP. Under these conditions, the nickel hydroxide nanoparticle synthesis process was able to precipitate  $99.90 \pm 0.02\%$   $\text{Ni}^{2+}$  ions. This result shows that the use of surfactants does not affect the amount of  $\text{Ni}^{2+}$  ions precipitated, but rather affects the characterization of nickel hydroxide formed. The characterization results showed that the use of surfactants decreased the purity of nickel hydroxide. This phenomenon is due to the presence of  $\text{NiSO}_4$  impurities that are still trapped in the micelle template. As a result,

these impurities cannot be removed during the washing process with water. The highest purity of the product was achieved when the synthesis was carried out without the use of surfactants, which was 96.41%. However, the use of surfactants can produce particles of smaller sizes. ABS surfactant is able to provide the best nickel hydroxide product where the product formed is homogeneous and has a size of 3.12–4.47 nm.

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