

# Optimization and Treatment Characteristics for Green Synthesis of NiO Nanomaterials from White Broccoli (*Brotrytis Cauliflower*) Flower Extract with Varying pH, Temperature, and Stirring Speed

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Received: 4 August 2024, Revised: 27 September 2024, Accepted: 4 October 2024, Published: 1 November 2024

## Abstract

This research aims to explore the effect of pH, temperature, and stirring speed on the characteristics of NiO nanomaterials synthesized using the *green synthesis method* from white broccoli flower extract (*Brassica oleracea var. botrytis*). Varying synthesis conditions were tested to determine the optimal conditions that produce nanoparticles with the desired size, distribution, and stability. Characterization was carried out using SEM-EDX, XRD, and TEM to observe the morphology, element composition, and crystal structure of the nanoparticles. The research results show that pH 11, temperature 60 °C, and stirring speed 750 rpm are optimal conditions for the synthesis of NiO nanoparticles with homogeneous size and distribution and good stability.

**Keywords:** Stability, Particle distribution, SEM-EDX, XRD-TEM, Morphology

## Introduction

Nanotechnology has shown great potential in various fields, including in the synthesis of materials with unique characteristics and wide applications. Nanomaterials, especially nanoparticles, offer significant advantages such as increased reactivity, larger surface areas, and unique optical, electronic, and magnetic properties compared to bulk materials. One of the most researched nanomaterials is nickel oxide (NiO), which has wide applications in catalysis [1], sensors [2], batteries [3], and biomedical fields [4,5]. Research on the synthesis of NiO nanomaterials has been carried out using various methods, both conventional and green synthesis approaches [6].

Conventional synthesis methods such as *sol-gel*, *coprecipitation*, and *thermal decomposition* have been

widely used in the synthesis of NiO nanoparticles. Although these methods have been proven to be effective in producing nanoparticles with good control over size and morphology, they have several disadvantages compared to green synthesis methods, such as using hazardous chemicals such as organic solvents, reducing reagents, and surfactants [7]. Certain situations necessitate very extreme reaction conditions, such as high temperatures and pressures, as well as special equipment and large amounts of energy [8]. Conventional synthesis produces chemical waste that needs to be handled carefully to avoid environmental pollution [9], because it has chemical residues that can affect biocompatibility and biomedical applications [10] and requires the use of large amounts of energy and

resources, such as in the drying and calcination processes. This increases production costs and the carbon footprint [11].

Green synthesis methods have become the focus of our research, because of their advantages in reducing environmental impacts and the use of hazardous chemicals. Green synthesis uses natural sources such as, plants as reducing and stabilizing agents, which is not only environmentally friendly but also economical [12]. Research uses green synthesis methods to synthesize NiO nanoparticles using various biological sources, such as neem (*Azadirachta indica*) leaf extract [13], moringa leaf extract (*Moringa oleifera*) [14], using fenugreek seed extract (*Trigonella foenum-graecum*), [15], banana peel extract (*Musa paradisiaca*) as a reducing and stabilizing agent for the synthesis of NiO nanoparticles [16], acacia (*Acacia nilotica*) leaf extract [17], and solanum leaf extract (*Solanum trilobatum*) [18]. White broccoli flowers (*Brassica oleracea var. botrytis*) are one of the biological sources that contain secondary metabolites such as flavonoids, polyphenols, and glucosinolates, which can function as reducing and stabilizing agents in the synthesis of nanoparticles [19].

White broccoli flower extract contains various bioactive compounds, including flavonoids, phenolic acids, glucosine, and vitamin C. These compounds act as reducing and stabilizing agents in the synthesis of nanoparticles [20]. Flavonoids have active hydroxyl groups (-OH), which can donate electrons. During the synthesis process, nickel ions ( $\text{Ni}^{2+}$ ) are reduced by electrons donated from the hydroxyl groups on flavonoids [21]. After reduction, flavonoids and other secondary metabolites help stabilize the formed NiO nanoparticles by forming a protective layer around the particles, preventing agglomeration [22].

In this green synthesis method, determining optimal synthesis conditions is essential to achieve the desired results. Temperature, pH, and stirring time are critical variables that significantly influence the size, morphology, and stability of the resulting nanoparticles. Research has shown that higher temperatures can speed up reaction rates and increase crystallinity, but they can also cause agglomeration if not balanced with proper control of stirring speed. The pH of the solution also plays an important role in determining the surface charge of the nanoparticles, which in turn influences the interactions between the particles and their colloidal

stability. The stirring time, on the other hand, ensures homogeneous distribution of the reducing agent and metal precursors in the solution, which is essential for uniform particle growth. Hessian *et al.* 2023, showed that variations in heating temperature from 60 to 100 °C significantly affected the size and catalytic activity of NiO nanoparticles synthesized using neem leaf extract [23] Messai *et al.* 2023, identified that a solution pH between 7 to 10 produces NiO nanoparticles with a smaller size and a more uniform size distribution [24] Therefore, in an effort to produce nanoparticles with optimal characteristics, it is necessary to carry out comprehensive experiments to systematically evaluate and optimize these variables. Higher stirring speeds result in particles with a more uniform size distribution, increasing the rate of ion reduction and producing particles with a smaller size and narrower distribution [25-29].

In this study, we explored the influence of pH, temperature, and stirring duration variables on the characteristics of NiO nanomaterials synthesized using the green synthesis method with white broccoli (*Brotrytis cauliflower*) flower extract. Through a comprehensive approach, this research aims to identify optimal conditions that produce nanoparticles with the desired size, distribution, and stability. The results of this research are expected to make a significant contribution to the development of nanomaterial synthesis technology that is more environmentally friendly and sustainable, as well as expanding the application of NiO nanoparticles in various industrial and technological fields.

## Materials and methods

### Materials and equipment

The research material used Nickel Precursor: Nickel(II) nitrate hexahydrate ( $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ ) obtained from Sigma-Aldrich. Natural Ingredients: Fresh white broccoli flowers (*Brotrytis cauliflower*) obtained from the local market. Solvents and Reagents: Deionized water is used in all processes. NaOH and HCl solutions are used to adjust the pH.

Laboratory Equipment: Blender, beaker glass, watch glass, Whatman No. filter paper. 41, hot plate with magnetic stirrer, pH meter, thermometer, centrifuge, oven, and furnace.

## Method

### Preparation of white broccoli flower extract

White broccoli flowers were washed thoroughly with deionized water and cut into small pieces.

### Making white broccoli flower powder extract (*Botrytis cauliflower*)

The prepared white broccoli (*Botrytis cauliflower*) was mashed using a blender and placed in the oven at 70 °C for 24 h. The drying method is used to remove water content from plants. Dried broccoli is mashed using a blender (*sharp*). Then it was sifted using a 100-mesh sieve [30].

### Making white broccoli plant extract (*Botrytis cauliflower*)

Put 10 g of white broccoli powder into a 250-mL Erlenmeyer flask, which was dissolved in 250-mL of distilled water. The solution was stirred using a *magnetic stirrer* and heated on a *hotplate* at a temperature of 60 - 70 °C for 3 h. After cooling, the solution was filtered using Whatman No. filter paper. 41 and vacuumed. The white broccoli extract solution was stored at 4 °C, to maintain the stability of bioactive compounds [30].

### Synthesis of NiO nanoparticles

The synthesis was carried out using nickel nitrate hexahydrate  $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  in crystal form with a concentration of 0.1 M (2.91 g) which was dissolved in 100-mL of deionized water (DI water), and stirred until the solution becomes green and homogeneous. Then 100-mL of the solution was added to 100-mL of white broccoli flower extract solution by dripping using a burette while stirring using a *magnetic stirrer* on a *hot plate* at the desired speed (500, 750 and 1,000 rpm) at

the desired temperature (40, 60 and 80 °C) until a color change occurs in the solution. The white broccoli flower extract solution to which Ni solution had been added was adjusted to the desired pH (9, 10 and 11) using sodium hydroxide (NaOH) and hydrochloric acid (HCl) and stirred for 30 min using a *magnetic stirrer*. Next, the Ni synthesis solution was centrifuged at a speed of 3,000 rpm for 25 min and rinsed using distilled water and ethanol. The washed sediment was placed in an oven for 24 h at 60 °C. The dried sediment was placed in a furnace at 500 °C and crushed until it passed through a 200-mesh sieve.

### Calculation of yield

The yield was calculated to determine the efficiency of the NiO nanoparticle synthesis process.

### Nanoparticle characterization

*Fourier Transform Infrared Spectroscopy* (FTIR): Used to identify functional groups present on NiO nanoparticles and to confirm the presence of organic compounds from white broccoli flower extract that may be adsorbed on the surface of the nanoparticles. SEM-EDX was used to observe the surface morphology of nanoparticles and analyze their elemental composition. *X-ray Diffraction* (XRD): To identify the crystallinity structure of NiO. *Transmission Electron Microscopy* (TEM): To observe particle morphology and size.

## Results and discussion

### Yield of NiO nanoparticles

The yield resulting from the synthesis of NiO nanoparticles using white broccoli flower extract showed significant variations depending on pH, temperature, and stirring speed. As seen in **Table 1**.

**Table 1** Rendering results.

pH	Rendement (%)	Temperature (°C)	Rendement (%)	Mixing speed (rpm)	Rendement (%)
9	84.13	40	82.0	500	81.84
10	90.83	60	90.8	750	90.87
11	93.23	80	88.8	1,000	85.11

In general, the highest yield was obtained at pH 11, temperature 60 °C, and stirring speed 750 rpm.

### Effect of pH on yield

Based on the data in **Table 1**, the yield of NiO nanoparticles increased with increasing pH. At pH 9, the yield was 84.13 %, increased to 90.83 % at pH 10, and peaked at 93.23 % at pH 11. This suggests that more alkaline conditions favor a more efficient synthesis process, which may be due to the increased ionization and reactivity of secondary metabolites involved in reduction reactions. Higher pH conditions tend to facilitate the formation of more stable complexes between nickel ions and organic compounds from white broccoli flower extract, thereby increasing the synthesis yield [31]. At alkaline pH, secondary metabolites such as flavonoids and polyphenols in white broccoli extract tend to be more ionized, increasing their ability to interact with metal ions. This produces a more stable complex and supports more the efficient formation of NiO nanoparticles [32].

Because more hydroxide ions are present, alkaline pH conditions facilitate reduction and complexation reactions more effectively due to the presence of more hydroxide ions. OH<sup>-</sup> ions help in the reduction of Ni<sup>2+</sup> ions to NiO and also in the precipitation of NiO from solution. This increases the efficiency of the synthesis process and the resulting yield [24]. Under alkaline conditions, OH<sup>-</sup> ions play a role in stabilizing the nanoparticles formed, thus preventing agglomeration and producing nanoparticles with a more uniform size and higher yield [33].

### Effect of temperature on yield

Temperature variations also showed a significant influence on the yield of NiO nanoparticles. At 40 °C, the yield was 82.0 %, which increased drastically to 90.8 % at 60 °C. However, the yield decreased slightly to 88.8 % at 80 °C. This suggests that there is an optimal temperature (around 60 °C) at which synthesis occurs with maximum efficiency. Too high a temperature may

cause partial decomposition of secondary metabolites or the formation of agglomerates, which reduces the efficiency of the process [23].

Research conducted by Anbuvaran *et al.* (2018) showed that the use of *Vitex negundo leaf extract* for the synthesis of zinc oxide (ZnO) nanoparticles at a temperature of 60 °C produced nanoparticles with optimal characteristics. [34]. Furthermore, Kumar *et al.* (2021) research supports this finding. They synthesized silver nanoparticles using mango leaf extract at a temperature of 60 °C and found that these conditions helped in the formation of nanoparticles of the desired size and increased the yield of the resulting nanoparticles. This research emphasizes the importance of temperature in controlling the size and morphology of the resulting nanoparticles [35].

### Effect of stirring speed on yield

The stirring speed also plays an important role in determining the yield of NiO nanoparticles. At a stirring speed of 500 rpm, the yield was 81.84 %, increasing to 90.87 % at 750 rpm. However, at a higher stirring speed of 1,000 rpm, the yield decreased to 85.11 %. This indicates that a stirring speed of around 750 rpm is optimal for increasing the yield, allowing homogeneous distribution and efficient interaction between the precursor and reducing agent, and that too high a stirring speed may cause excessive turbulence, disrupting particle formation and particle aggregate stability.

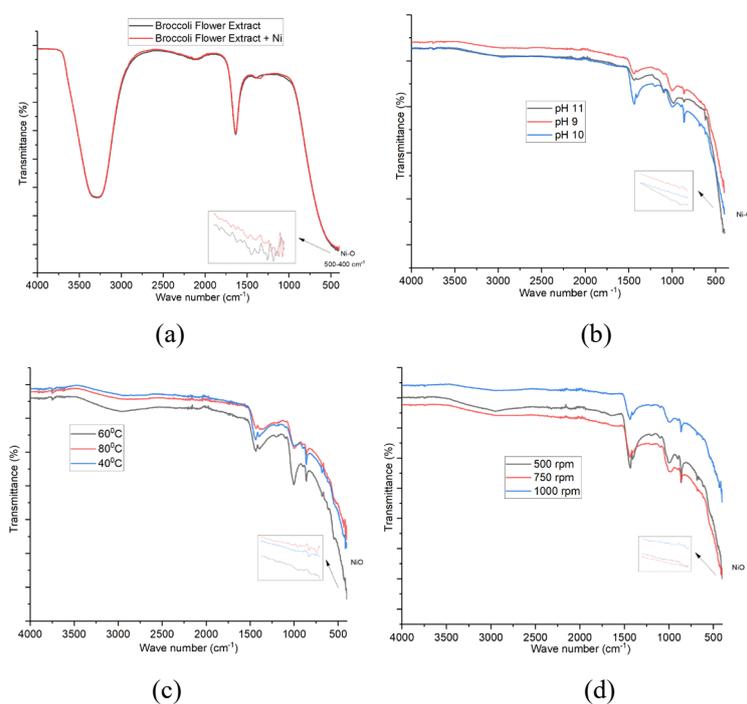
Iqbal research in 2021, using a stirring speed of 750 rpm during the synthesis of Ag-Co nanoparticles, showed an increase in catalytic efficiency and high product conversion [36]. This study confirms that high stirring speeds favor effective mixing and the formation of nanoparticles with desired characteristics.

## Characteristics of NiO nanoparticles

### Characteristics of fourier transform infrared spectroscopy (FTIR)

The FT-IR spectrum results show **Figure 1(a)**, that there is a significant peak in the  $500 - 400 \text{ cm}^{-1}$ , which indicates the presence of metal-oxide, in this case NiO. The FT-IR spectrum of white broccoli flower extract without the addition of Ni metal shows a peak with lower intensity in this area, indicating that there is no metal oxide formation. In contrast, the FT-IR spectrum of white broccoli flower extract added with Ni metal

showed a significant decrease in transmittance values in the  $500 - 400 \text{ cm}^{-1}$ . This decrease is caused by increased absorption of the IR signal by the formed Ni-O bonds, indicating that NiO nanoparticles are formed. Recent research by Singh *et al.* (2022) and Chen *et al.* (2023) supports these findings, where lower transmittance intensity in the  $500 - 400 \text{ cm}^{-1}$  is associated with better formation of NiO nanoparticles when using plant extracts. These findings confirm that the *green synthesis method* using white broccoli flower extract is effective in producing NiO nanoparticles [37-39].



**Figure 1** FT-IR characteristics: (a) White broccoli flower extract and white broccoli flower extract + Ni; (b) Variations in pH 9, pH 10, and pH 11; (c) Variations in temperature 40, 60, and 80 °C and (d) Variations in stirring speed 500, 750, and 1,000 rpm.

### Effect of pH on the formation of NiO nanoparticle clusters

pH greatly influences the formation and stability in green synthesis of NiO nanoparticles. At pH 11, the lower transmittance value indicates greater absorption at a wavelength of around  $400 \text{ cm}^{-1}$ . This means that NiO formation is more optimal, with more metal-oxide groups formed. Under conditions of pH 11, the alkaline environment helps in the process of reducing nickel ions to nickel oxide nanoparticles more efficiently. At this pH, conditions are more favorable for the formation of stable NiO, in accordance with the *Pourbaix diagram*,

where NiO is more dominant and  $\text{Ni}(\text{OH})_2$  begins to degrade into NiO with increasing pH [40,41].

At pH 10, the transmittance value was between pH 9 and pH 11, indicating that the formation of NiO nanoparticles was in moderate conditions. This shows that there is better absorption compared to pH 9 but not as good as at pH 11, where NiO formation can be further optimized. At pH 9, higher transmittance values indicate lower absorption at around  $400 \text{ cm}^{-1}$ . This may indicate that the pH 9 condition is less than optimal for the formation of metal-oxide groups. There may be less efficient settling or the formation of larger, less stable particles. This indicates that NiO formation is quite

good but not optimal. At pH 9, Ni(OH)<sub>2</sub> and NiO are in equilibrium, with a greater tendency towards the formation of Ni(OH)<sub>2</sub> which is partially soluble in water, so that the yield is relatively lower than at a more basic pH [42,43].

#### **Effect of temperature on the formation of NiO nanoparticle clusters**

Temperature affects the kinetics of chemical reactions in *green synthesis* of NiO nanoparticles. A temperature of 40 °C shows the highest transmittance intensity in the 500 - 400 cm<sup>-1</sup>. The high transmittance intensity indicates that NiO formation is less than optimal at this temperature, possibly due to insufficient thermal energy to support an efficient Ni-O bond formation reaction. Temperature 60 °C: Shows the lowest transmittance intensity in the 500 - 400 cm<sup>-1</sup>. The low transmittance intensity indicates that 60 °C is the optimal temperature for NiO formation, because the thermal energy at this temperature is sufficient to support the Ni-O bond formation reaction with high efficiency, while the temperature of 80 °C shows a transmittance intensity that is between temperatures of 60 and 40 °C, although these temperatures are high enough to support NiO formation, the possibility of particle redispersibility phenomena occurring which reduces the efficiency of Ni-O bond formation, resulting in transmittance intensity that is not as good as at 60 °C [44,45].

This is in accordance with the results of the combination of temperature 60 °C, pH 10, and stirring speed 750 rpm to synergistically maximize the yield of nanoparticles produced. The right temperature and pH support the optimal reaction rate, while a sufficiently high stirring speed ensures an even distribution of ions and reducing agents, producing nanoparticles in large quantities with controlled sizes.

#### **Effect of stirring speed on the formation of NiO nanoparticle clusters**

The stirring speed affects the reaction kinetics in the *green synthesis* of NiO nanoparticles. A stirring speed of 1,000 rpm has the highest transmittance intensity in the 500 - 400 cm<sup>-1</sup> range. The high transmittance intensity indicates that the stirring speed of 1,000 rpm is less than optimal for NiO formation. Although this agitation speed is high enough to support

NiO formation, excessive agitation can cause particle redistribution, which reduces the efficiency of Ni-O bond formation and results in lower transmittance intensity [46]. A stirring speed of 750 rpm shows the lowest transmittance intensity in the 500 - 400 cm<sup>-1</sup>. The low transmittance intensity indicates that a stirring speed of 750 rpm is the most optimal for NiO formation because agitation at this speed is sufficient to support the Ni-O bond formation reaction efficiently, while a stirring speed of 500 rpm shows a transmittance intensity that is between the stirring speed of 1,000 rpm. and 750 rpm, possibly due to insufficient agitation to support the Ni-O bond formation reaction efficiently.

#### **NiO characteristics in scanning electron microscope - energy dispersive analysis (SEM-EDX)**

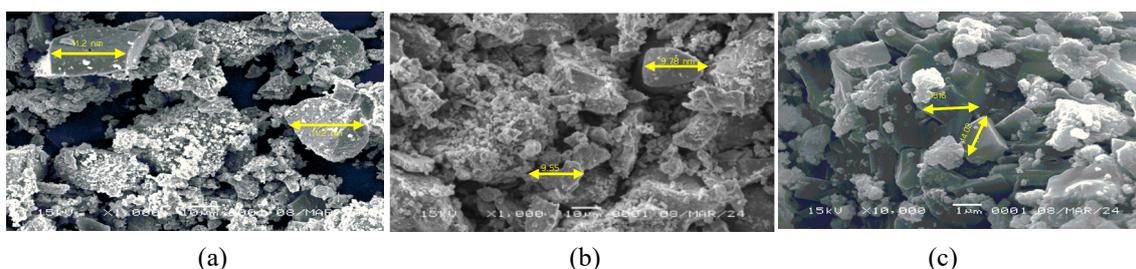
##### ***Effect of pH on morphology and homogeneity of NiO nanoparticles***

**Figure 2.1(a)** shows that nanoparticles at pH 9 tend to be irregularly shaped with varying particle sizes. The nanoparticle dispersion showed significant agglomeration, indicating that its homogeneity was poor. Nanoparticles tend to be amorphous with a high level of agglomeration. The nanoparticle dispersion was less homogeneous with significant agglomeration. This shows that at low pH, the nucleation process is not optimal, producing larger and non-uniform particles. Chu *et al.* (2014) found that increasing the pH in the hydrothermal process can change the morphology of NiO from a nanoflake structure to a thin nanobridge, indicating the important role of OH<sup>-</sup> in controlling product morphology [28].

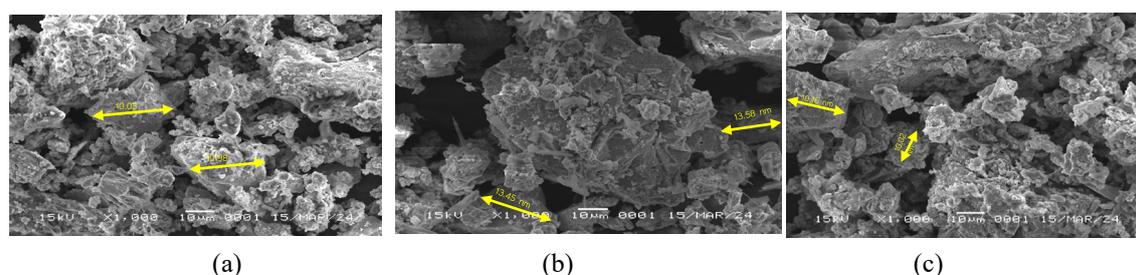
**Figure 2.1(b)**, for pH 10, nanoparticles begin to show a more defined shape, more particles are cubic. Homogeneity is better compared to pH 9, with more even particle distribution and less agglomeration. Morphology at pH 10, nanoparticles begin to show a more defined shape with many cubic particles. These particles are smaller and more uniform compared to pH 9. Homogeneity is better compared to pH 9, with more even particle distribution and reduced agglomeration. This indicates enhanced nucleation processes and more uniform particle growth. Singh *et al.* (2009) reported that at a higher pH the resulting Ag nanoparticles were smaller and had a narrower size distribution due to better stabilization by OH<sup>-</sup> [47].

**Figure 2.1(c)**, for pH 11 shows the most uniform morphology with a dominant cubic shape. The best homogeneity among the 3 pH 11 variations, with evenly distributed particles and little agglomeration. At pH 11, the nanoparticles showed the most uniform morphology with a dominant cubic shape. These particles are smaller and more well defined than at lower pH. At this pH,

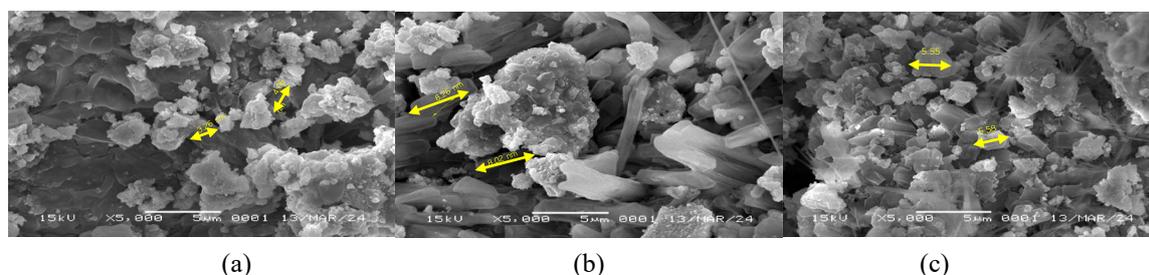
alkaline conditions promote rapid nucleation processes and more controlled particle growth. Zakaria & Osman (2021) showed that NiO nanoparticles synthesized at pH 11 had a cubic morphology with less agglomeration and a smaller average particle size, compared to lower pH [48].



**Figure 2.1** SEM characteristics with 1000x magnification at pH (a) 9, (b) 10, and (c) 11.



**Figure 2.2** SEM characteristics with 1000x magnification at temperatures (a) 40, (b) 60, and (c) 80 °C.



**Figure 2.3** SEM characteristics with 1000x magnification at Stirring Speed (a) 500, (b) 750, and (c) 1,000 rpm.

### Effect of temperature on morphology and homogeneity of NiO nanoparticles

**Figure 2.2(a)** Nanoparticles at 40 °C show varying particle sizes with irregular shapes. The particles tend to agglomerate, indicating uneven growth. The nanoparticle dispersion shows significant agglomeration, this shows that the homogeneity is not good at this temperature. **Figure 2.2(c)**, for a temperature of 80 °C, nanoparticles begin to show a more defined shape with a more uniform particle size, as well as better homogeneity compared to a temperature of 40 °C, with a more even particle

distribution and less agglomeration. Homogeneity decreases at this temperature with increased agglomeration and less uniform particle size distribution. **Figure 2.2(b)** a temperature of 60 °C produces the most uniform morphology with a dominant cubic shape. And the best homogeneity among the 3 temperature variations, with evenly distributed particles and little agglomeration. This indicates that a temperature of 60 °C provides optimal conditions for uniform nanoparticle growth.

According to research by Patra *et al.* (2021), increasing temperature in the nanoparticle synthesis

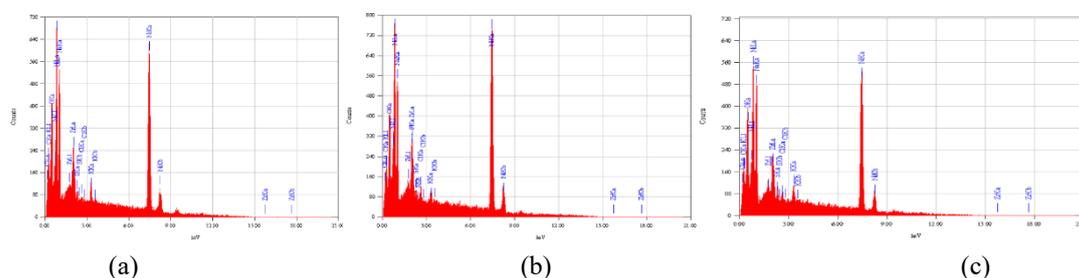
process can increase reaction kinetics and produce particles with a smaller size and narrower distribution. This study also found that the optimal temperature for uniform particle growth is around 60 °C [49]. Research by Mammadyarova *et al.* (2020) showed that higher temperatures can cause greater particle agglomeration, reducing homogeneity and increasing uneven particle size distribution [50].

### Effect of stirring speed on the morphology and homogeneity of NiO nanoparticles

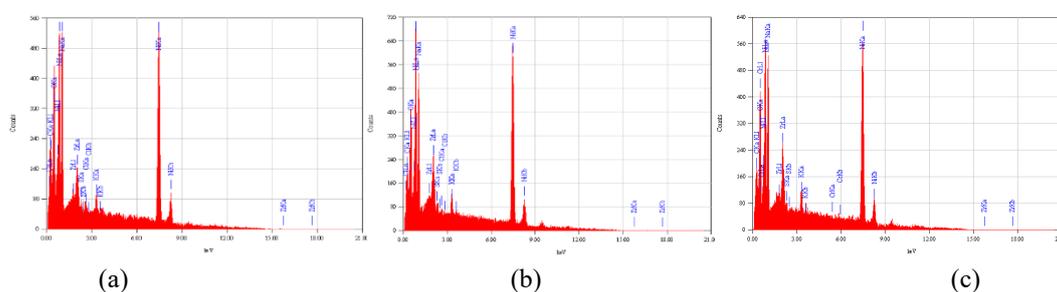
In **Figure 2.3(a)**, a stirring speed of 500 rpm shows varying particle sizes with irregular shapes. The particles tend to agglomerate. The nanoparticle dispersion showed significant agglomeration, indicating that its homogeneity was poor at this speed. **Figure 2.3(b)**, stirring speed 750 rpm, the nanoparticles show a more defined shape and a more uniform particle size. These particles have a more dominant cubic shape. Homogeneity is better compared to stirring speeds of 500 and 1,000 rpm, with more even particle distribution

and less agglomeration. This indicates that a stirring speed of 750 rpm provides optimal conditions for uniform nanoparticle growth. **Figure 2.3(c)**, stirring speed 1,000 rpm shows a less uniform morphology with some larger particles. At this rate, homogeneity decreases due to increased agglomeration and less uniform particle size distribution.

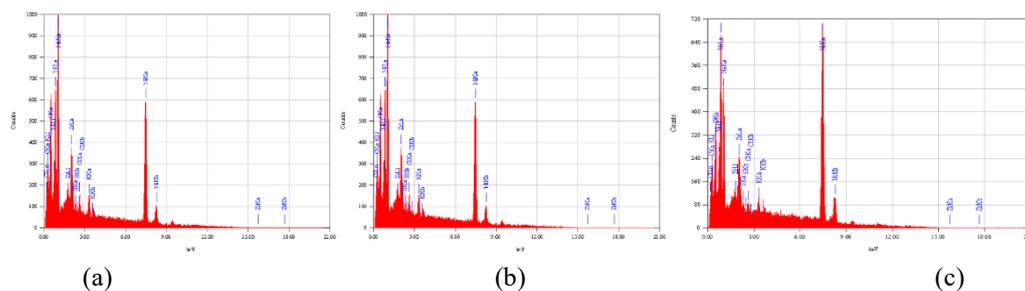
According to research by Fouad *et al.* (2019), higher stirring speeds can cause increased particle agglomeration, reduce homogeneity, and increase uneven particle size distribution. This study also found that the optimal stirring speed for uniform particle growth was around 750 rpm [51]. Research by Khan *et al.* (2016) also showed that increasing the stirring speed can increase the homogeneity of nanoparticles because the nucleation and particle growth processes are more uniform. However, too high a speed can cause greater agglomeration [28]. Research by Balavandy *et al.* (2014) found that increasing stirring time can increase stability and particle size, but excessive stirring can cause agglomeration and non-uniform particle size [29].



**Figure 2.4** EDX characteristics at pH (a) 9, (b) 10, and (c) 11.



**Figure 2.5** EDX characteristics at temperatures (a) 40, (b) 60, and (c) 80 °C.



**Figure 2.6** EDX characteristics at stirring speed (a) 500, (b) 750, and (c) 1,000 rpm

According to the EDX image results, white broccoli flower plant extract can bind Ni ions. Most Ni ions interact with organic compounds through coordination bonds or van der Waals interactions, which help form and stabilize nanoparticles. The release of Ni ions from nanoparticles is influenced by several factors, such as pH, the presence of complexation ligands, and the environment around the nanoparticles. According to research by Patra *et al.* (2021), increasing the pH in the nanoparticle synthesis process increases the oxidation of Ni elements to NiO, which is in line with the results found [49].

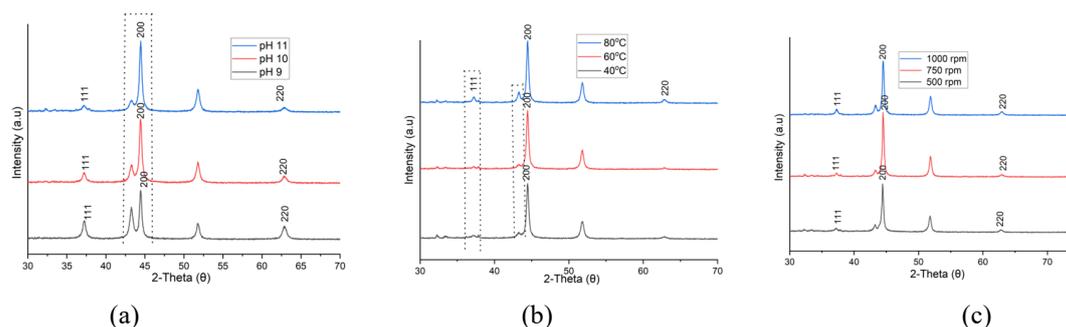
#### Characteristics of NiO in *x-ray diffraction* (XRD)

Overall, all variations in pH, temperature and stirring speed, result in the main diffraction peaks appearing at around the  $2\theta$  theta angles of 37.2, 43.3, and 62.9 °, which correspond to the (111), (200), and (220) crystal planes of the structure, cubic NiO. At pH 9 variations, the peak intensity is relatively lower compared to pH 10 and pH 11, this shows that the crystallinity at pH 10 and pH 11 is higher. The increase in peak intensity indicates that the NiO particles produced at pH 10 and pH 11 have a better crystalline structure and larger crystal size. pH 11 produces the highest intensity, this indicates the highest crystallinity. The significant increase in peak intensity at pH 11 indicates that this pH condition is the most optimal for the formation of NiO crystals with a highly defined structure [49]. Research by Manikandan *et al.* (2019)

also showed that higher pH conditions during hydrothermal synthesis can produce nanoparticles with better crystallinity and a more ordered crystal structure [52].

At varying temperatures, at 40 °C the peak intensity is relatively lower compared to temperatures of 60 and 80 °C, this situation indicates lower crystallinity. The increase in peak intensity indicates that the NiO particles produced at 80 °C have a better crystalline structure and larger crystal size than those at 40 °C. A temperature of 60 °C produces the highest intensity, this shows the highest crystallinity. The significant increase in peak intensity at 60 °C indicates that this temperature condition is the most optimal for the formation of NiO crystals with a highly defined structure. Research by Manikandan *et al.* (2019) also showed that higher temperature conditions during hydrothermal synthesis can produce nanoparticles with better crystallinity and a more ordered crystal structure [52,53].

Based on the XRD results, increasing the stirring speed from 500 to 750 rpm increases the crystallinity of NiO nanoparticles, but at 1,000 rpm the crystallinity decreases, so that the presence of the crystalline phase of NiO is less. The sharper and more intense diffraction peaks at a stirring speed of 750 rpm indicate that the crystallinity and crystal size of NiO are most optimal at this speed. These results are in line with previous research showing that higher stirring speeds during nanoparticle synthesis can improve crystal structure and crystallinity [52].



**Figure 3** XRD Characteristics at XRD; (a) pH (b) temperatures, and (c) stirring speed.

### Characteristics of NiO in transmission electron microscopy (TEM)

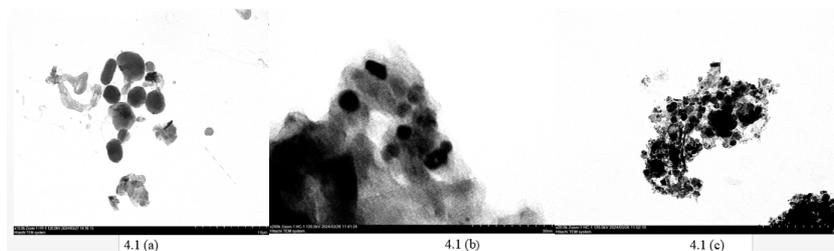
TEM images confirm observations from SEM and XRD, showing that NiO nanoparticles are cubic shape with varying size distribution depending on the synthesis conditions. Average particle size; 0.28 nm at pH 9, 0.66 nm at pH 10 and 24.76 nm at pH 11. TEM results show that the size of NiO nanoparticles is strongly influenced by pH during the synthesis process. Very small particle sizes at pH 9 and pH 10 indicate less than growth conditions, while pH 11 provides optimal conditions for the formation of particles with larger and more stable sizes. Research by Zakaria and Osman (2021) found that the size of NiO particles synthesized at pH 11 tended to be larger, but at higher pH, the particles tended to be smaller due to high agglomeration [48]. Higher pH in the nanoparticle synthesis process can increase particle size up to a certain point before causing a decrease in particle size due to changes in chemical conditions that inhibit the growth of larger particles [26].

Smaller particle sizes at temperatures of 40 °C (2.08 nm) and 80 °C (0.001 nm) indicate less growth conditions, while temperatures of 60 °C (4.78nm) provide more optimal conditions for the growth of larger particles. bigger and more stable. These results are in line with previous research showing that temperature can influence particle size and stability during nanoparticle synthesis. Research by Mammadyarova,

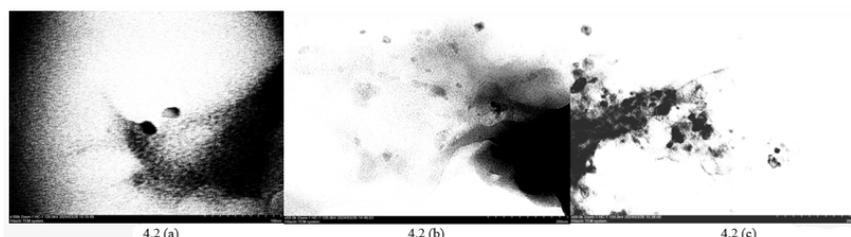
(2020) also shows that higher temperatures during synthesis can produce particles with larger sizes. However, agglomeration also increased at higher temperatures [50].

At a stirring speed of 1,000 rpm, the average particle size was obtained; 2.68 nm. High stirring speed affects the nanoparticle size by accelerating solution homogenization, increasing mass transfer, accelerating nucleation, and preventing particle aggregation. This results in more small particles.

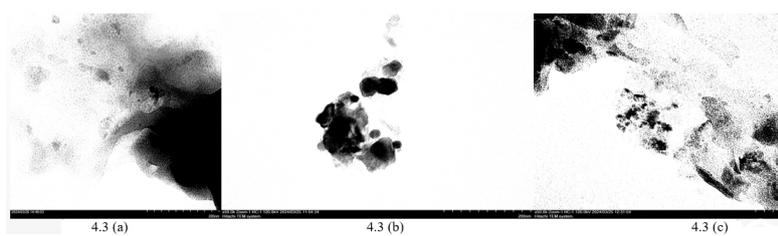
Smaller particle sizes at these velocities may indicate a very fast nucleation process but with limited particle growth. This could indicate that at very high stirring speeds, conditions become less than ideal for the formation of large and stable particles. Stirring speed 500 rpm average particle size; 3.63 nm. The particle size tends to be smaller due to slower reaction kinetics, allowing nucleation but limiting the growth of larger particles, whereas at a stirring speed of 750 rpm the average particle size is; 6.37 nm, more optimal conditions for the growth of larger particles than 500 rpm. Faster reaction kinetics allow for more balanced particle nucleation and growth, resulting in larger, more uniform particles. Previous research shows that increasing the stirring speed in the nanoparticle synthesis process can increase particle size. They found that at higher stirring speeds, particle size increased because faster reaction kinetics allowed the growth of larger particles [53,54].



**Figure 4.1** TEM characteristics of pH variations (a) 9, (b) 10, and (c) 11.



**Figure 4.2** TEM characteristics of temperature variations (a) 40, (b) 60, and (c) 80 °C.



**Figure 4.3** TEM characteristics of stirring speed variations (a) 500, (b) 750, and (c) 1,000 rpm.

## Conclusions

Higher pH increases the stability of Ni to NiO, with pH 11 showing the highest crystallinity. Smaller particle sizes at pH 9 and pH 10 indicate less ideal growth conditions, while pH 11 provides optimal conditions for the formation of larger, more stable particles. A temperature of 60 °C produces nanoparticles

with the best size and crystallinity. Temperatures higher or lower than 60 °C produce particles of smaller and less uniform size. A stirring speed of 750 rpm provides the best conditions for uniform growth of nanoparticles with larger sizes and homogeneous distribution. Stirring speeds that are too high or low produce particles with a smaller size and less uniform distribution.

## Acknowledgements

The author would like to thank the financial support from the National Research and Innovation Agency Number 37/II.7/Hk/2023, concerning Recipients of Research and Innovation Programs for Advanced Indonesia Wave 4 2024.

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