

# ONE STEP HYDROTHERMAL SYNTHESIS OF NICKEL DOPED TiO<sub>2</sub> NANOTUBE

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## Abstract

TiO<sub>2</sub> is one of the photocatalyst materials that is widely used and proven for environmental treatment. To increase the activity of TiO<sub>2</sub>, TiO<sub>2</sub> has been modified into a nanotube shape with nickel metal doping. The TiO<sub>2</sub> nanotubes with Ni doping have been synthesized with one step hydrothermal process. In this research, it shows that nickel doped with TiO<sub>2</sub> led to a reduction of crystal size and band gap energy. The smaller crystalline size and lower band gap energy enhanced its photocatalytic activity. The best results for photocatalytic activity were TiO<sub>2</sub>/Ni with a crystal size of 7.30 nm and a band gap energy value of 3.12 eV. The photocatalytic activity of TiO<sub>2</sub>/Ni was tested by degrading methylene blue under mercury lamp radiation, with the best result of 92.73 % degradation of methylene blue, within 240 minutes of photocatalytic activity.

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## Keywords:

TiO<sub>2</sub> nanotubes; Ni-doping; hydrothermal process; photocatalytic methylene blue degradation

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## 1. Introduction

Indonesian textile industry is growing rapidly to fulfill the demand from society. Indonesia Central Bureau of Statistic <sup>[1]</sup> reports that the growth of the Indonesian textile industry continues to increase by around 2.8% per year. The growth also increases the usage of textile dyes. Indonesian textile industry uses around 10,000 types of dyes each year. About 10-15% of textile dyes used in the dyeing process will be dumped along with wastewater. This wastewater can pollute the environment and can cause health problems because some of the dyes used can decompose into toxic and carcinogenic compounds <sup>[2]</sup>.

Several studies have been carried out to remove organic compounds and dyes contained in wastewater such as filtration, precipitation, coagulation, electrocoagulation, and adsorption using activated carbon. However, this method is considered inefficient due to high operational costs and can only transfer contaminants from one phase to another without any destruction <sup>[3]</sup>. Therefore, a better and more efficient way is needed to overcome this problem, such as photocatalysis process. The photocatalysis method is considered more effective in separating pollutants, decomposing wastewater and polluted air, and have ability to decompose of organic pollutants <sup>[4]</sup>.

One of the proven materials for a photocatalyst is TiO<sub>2</sub>. Titanium dioxide or TiO<sub>2</sub> is a semiconductor that has a wide band gap (3.2 eV-3.8 eV) which has photocatalytic efficiency around 5% of solar energy <sup>[5]</sup>. In addition, TiO<sub>2</sub> is relatively cheaper, stable, non-toxic, and can be used repeatedly <sup>[6, 26-28]</sup>. However, the wide band gap energy in TiO<sub>2</sub> limits its photocatalytic activity. The TiO<sub>2</sub> have higher activity in the UV region with a wavelength of 388 nm, while the desirable process will be in visible light region <sup>[7]</sup>.

The photocatalytic activity of TiO<sub>2</sub> can be increased in various ways, including by adding doping, making composites using other semiconductors, or increasing TiO<sub>2</sub> active sites <sup>[8]</sup>. Nanostructured TiO<sub>2</sub> claimed have higher active sites compared to nanoparticle shaped. The well proven nanostructured shape of TiO<sub>2</sub> that has higher active sites such as nanotubes and nanowires. Doping acts as electron trapping which can increase the photocatalytic activity of TiO<sub>2</sub> by minimizing recombination between holes and electrons <sup>[9]</sup>. The addition of doping in the structure of TiO<sub>2</sub> led to change the electron structure of the TiO<sub>2</sub> photocatalyst <sup>[10]</sup>. By changing the electron structure of TiO<sub>2</sub>, the responsiveness to light absorption are changes. Several types of metals that are often used as doping on TiO<sub>2</sub> are platinum (Pt), nickel (Ni), molybdenum (Mo), and palladium (Pd), all the metals have high activity during photocatalysis process. However, due to the high prices of platinum, molybdenum, and palladium, the usage of Ni metal is preferred because it is abundant and cheaper compared to noble metals <sup>[11]</sup>. The

use of Ni metal as doping on TiO<sub>2</sub> can increase its photocatalytic efficiency in the visible light region and reduce the energy band gap in TiO<sub>2</sub> [12].

The process to modify TiO<sub>2</sub> nanoparticles into TiO<sub>2</sub> nanotubes can be done by several methods such as anodization and hydrothermal methods. The nickel metal doping into TiO<sub>2</sub> can be done by various methods such as anodization, hydrothermal, and sol gel methods. In this research, we combine both the modification of TiO<sub>2</sub> nanoparticles into TiO<sub>2</sub> nanotubes and the Ni metal doping with one step hydrothermal process. The hydrothermal process is chosen since the process is simple, low cost and scalable. In this study, we fabricated Ni doped TiO<sub>2</sub> nanotubes catalysts via one step hydrothermal method. The effect of Ni doping has been investigated for photocatalytic reduction of methylene blue.

## 2. Experimental Section

### A. Ni doped TiO<sub>2</sub> nanotubes synthesis

TiO<sub>2</sub>/Ni was prepared by the hydrothermal process. The mixture of components TiO<sub>2</sub> (P25, Degussa) and Ni(CH<sub>3</sub>CO<sub>2</sub>)<sub>2</sub>·4H<sub>2</sub>O (Sigma Aldrich) was prepared according to Table 1. Then the mixture was placed into a 100 ml beaker then 60 ml of 10 M NaOH (Sigma Aldrich) was added. The mixtures were stirred for 1 hour on a hotplate. Then the mixture was put into an autoclave and then heated in an oven for 24 hours at a temperature of 120 °C. The results obtained in the form of solids were washed with 0.1 M HCl (Sigma Aldrich) until they reached a pH of 1-2. After pH 1-2 was reached, the samples were washed with distilled water by using a centrifuge until they reached a neutral pH. The solids from the centrifuge were dried in an oven at a temperature of 80 °C for 6 hours. The solids were then annealed in a furnace at a temperature of 450 °C for 2 hours.

Table 1. The molar ratio between TiO<sub>2</sub> and Ni precursors

Samples Name	Molar Ratio of TiO <sub>2</sub> (P25) and Ni(CH <sub>3</sub> CO <sub>2</sub> ) <sub>2</sub> ·4H <sub>2</sub> O
A	20 : 0
B	20 : 0.5
C	20 : 1
D	20 : 1.5
E	20 : 2

### B. Characterization

The synthesized of Ni doped TiO<sub>2</sub> nanotubes (TiO<sub>2</sub>/Ni) was characterized by using X-Ray Diffraction (XRD) to analyze the crystal phase and degree of crystallinity. The Diffuse Reflectance Spectroscopy (DRS) has been used to analyze the band gap value of the material. UV Vis spectroscopy has been used to analyze the absorbance of the sample during photocatalyst degradation of methylene blue.

### C. Photocatalyst activity on methylene blue degradation

A total of 0.1 grams of solid TiO<sub>2</sub>/Ni were weighed and put into a glass container whose surface was covered with aluminum foil. Then 100 ml of 20 ppm methylene blue solution was added and stirred for 1 hour. After stirring, 2 ml of the sample was taken, then put into a centrifuge tube and recorded as the 0<sup>th</sup> time. The hotplate and glass container containing the sample and stirrer were placed into the photocatalyst reactor.

For the methylene blue degradation, the experiment for arrange calibration curve has been done. UV-Vis Spectroscopy is used for the absorbance value of sample. Based on calibration curve, the concentration of methylene blue can be calculated from equation (1). The Y axis is absorbance value and X axis is concentration of methylene blue.

$$y = 0,1853x + 0,0052 \quad (1)$$

The photocatalyst test was carried out in the photocatalyst reactor for 4 hours (mercury lamp, blower, and hotplate were turned on). The 2 mL of samples were taken during time interval at 15, 30, 60, 120, 150, 180, and 240 minutes. All samples were centrifuged at 7000 rpm for 10 minutes. After centrifugation, 1 ml of liquid was pipetted and then 2 ml of distilled water was added for the measurement of methylene blue contents by using absorbance method on UV-Vis spectrophotometer.

### 3. Result and Discussion

The synthesized TiO<sub>2</sub>/Ni was characterized using XRD to determine the crystallinity of samples. The TiO<sub>2</sub>/Ni diffraction pattern for each sample that has been characterized using XRD is shown in Fig 1. Based on the TiO<sub>2</sub>/Ni diffraction pattern (Fig. 1), all samples were only in the anatase phase. The rutile phase was not detected in the TiO<sub>2</sub>/Ni diffraction pattern in each sample because the formation of rutile phase began at a calcination temperature of more than 800 °C, while in this study the calcination temperature is 450 °C. Nickel diffraction peaks that did not appear during XRD characterization indicated that nickel atoms had entered the TiO<sub>2</sub> lattice [13, 14]. From the Fig. 1, it was processed using the Scherrer equation to determine the crystallite size.

$$D = \frac{0,89 \lambda}{\beta \cos \theta} \quad (2)$$

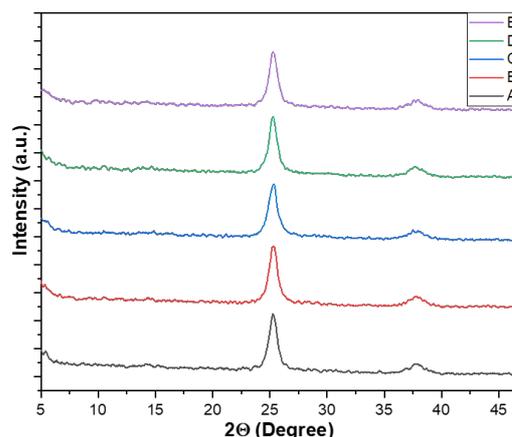


Figure 1. X-Ray diffractometer result of TiO<sub>2</sub>/Ni samples

Table 2. Crystallites size of TiO<sub>2</sub>/Ni sample

Samples	Molar Ratio of TiO <sub>2</sub> and Ni	2θ (°)	D (nm)
A	20 : 0	25.36	8.09
B	20 : 0.5	25.36	7.88
C	20 : 1	25.26	7.30
D	20 : 1.5	25.19	7.84
E	20 : 2	25.27	8.17

Table 2 shows the crystallite size decreased when nickel doping was added. From Table. II, sample A without nickel doping had a crystal size of 8.09nm, while samples B, C, and D with nickel doped addition has lower crystallite sizes. The addition of doping can affect the physical properties of TiO<sub>2</sub> nanotubes, one of which is to produce a small crystallite size [15]. Photocatalysts with small crystal sizes can induce a larger band gap due to the increased reduction-oxidation ability so as to increase the photocatalytic activity. Materials with nano size (1-100 nm) can provide high photocatalytic activity. The smaller crystal size led to the larger the crystal surface area [16]. The large surface area of the crystal enhances the rate of the photocatalytic reaction, due to the increased availability of active sites in the photocatalyst [15].

The characterization of the synthesized TiO<sub>2</sub>/Ni using a DRS spectrophotometer was carried out to determine the light absorption area to calculate the band gap energy value. The band gap energy values will affect during electron excitation process of TiO<sub>2</sub>/Ni [17]. In addition, the band gap energy value will also affect the absorption of the required light energy [18,19]. A small band gap energy value will only require a small amount of light energy. Fig. 2 shows the absorbance spectra of the synthesized material at a wavelength of 200-800 nm. From Fig.2, an increase in absorbance begins to occur at a wavelength of 400 nm for all samples. On the curve it can be seen that all samples absorb well in the UV light region where the wavelength is less than 380 nm.

From Fig.2, further analysis was carried out using the Kubelka-munk theory approach to determine the absorption properties of the synthesized material. The relationship between optical absorption coefficients is derived mathematically through the equation  $F(R) = (1-R)2/2R$  whose value is proportional to the value of the absorption coefficient per scattering or the value of  $k/s$ , where  $F(R)$  is the Kubelka-munk factor. The relationship between the Kubelka-munk factor and photon energy is shown in Fig. 3.

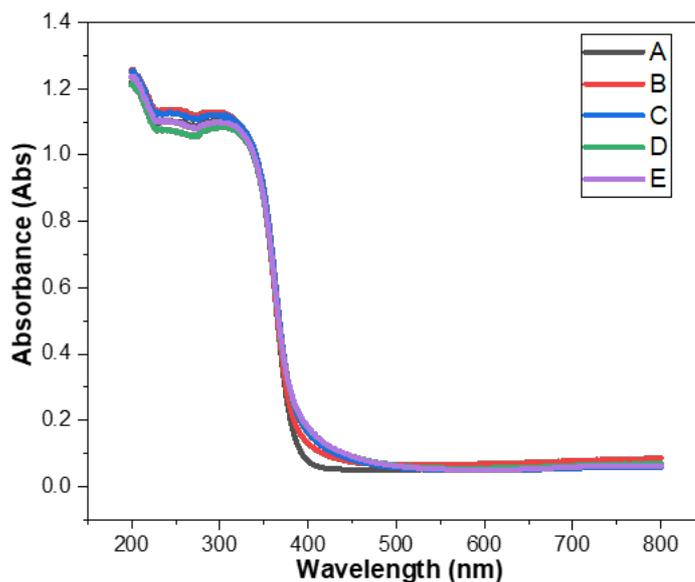


Figure 2. Diffuse reflectance spectroscopy (DRS) of TiO<sub>2</sub>/Ni samples

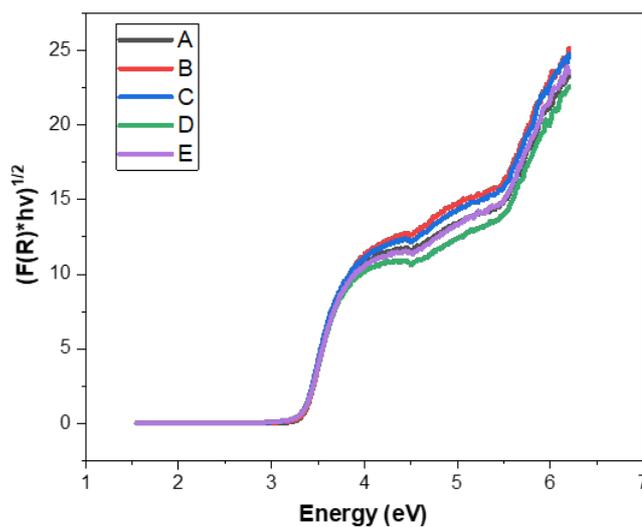


Figure 3. The relationship between Kubelka-munk factor and photon energy for TiO<sub>2</sub>/Ni Samples

Based on Fig. 3, the band gap energy value for each sample can be determined by linear extrapolation on the area of the graph that has the highest slope. The results of the linear extrapolation performed on each sample are shown in Table III.

Table 3. Band gap energy of TiO<sub>2</sub>/Ni samples

Samples	Band Gap Energy (eV)
A	3.16
B	3.15
C	3.12
D	3.14
E	3.20

Based on Table 3, sample A which was not added with nickel doped had a higher band gap energy value than samples B, C, and D were added with nickel doped. This evidence proves that the addition of nickel doping can

reduce the band gap energy value of the synthesized material. The decrease in the energy value of the band gap is due to the formation of new energy levels by nickel doping, so the distance between the valence band and the conduction band is reduced. The smaller the band gap energy value led to the increase of photocatalyst activity to capture the light with lower energy levels such as visible light. Angel et.al mentioned that lower band gap TiO<sub>2</sub> has higher photocatalytic activity for water treatment [29]. The band gap value is increasing again after sample C. This phenomenon happens since there is a possibility of agglomeration of doped metal that led to increasing band gap value.

The photocatalytic activity of TiO<sub>2</sub>/Ni was analyzed based on its ability to degrade the pollutant methylene blue. The degradation reaction of methylene blue is shown in Equation 3 below.



The solution of TiO<sub>2</sub>/Ni degradation in methylene blue was tested by using a UV-Vis spectrophotometer at a wavelength of 660 nm. This wavelength is the wavelength used to detect methylene blue.

Testing of the photocatalytic activity of TiO<sub>2</sub>/Ni was carried out with an initial concentration of 20 ppm methylene blue solution. The selection of this initial concentration is based on the content of dye in the waste produced in the textile industry, which is around 20 to 30 ppm [20]. In the photoreactor, each mixture of TiO<sub>2</sub>/Ni and methylene blue will be irradiated using a mercury lamp for a certain time. Prior to irradiation, the sample was dispersed for 1 hour in the dark (without light). It aims to achieve equilibrium conditions for the adsorption of reactants on the surface of the catalyst. During this process there was no degradation of methylene blue which was indicated by the unchanged concentration of methylene blue before and after the dispersion took place.



Figure 4. The photocatalytic activity results of TiO<sub>2</sub>/Ni samples on methylene blue degradation

Fig. 4 shows a solution of methylene blue that has been degraded by TiO<sub>2</sub>/Ni at test times of 0, 15, 30, 60, 120, 150, 180, and 240 minutes for all samples. The left side samples on Fig.5 are samples at 0 minute. From the visual analysis, there is a decrease in color intensity over time. After being examined using a UV-Vis spectrophotometer at a wavelength of 660 nm, the absorbance value of each sample decreased. The longer the time of irradiation by a mercury lamp to a mixture of TiO<sub>2</sub>/Ni with methylene blue, the smaller the absorbance value.

The results of the degradation of methylene blue at 240 minutes for each sample are shown in Table IV and Fig. 5. From Table IV, it shows that samples B, C, and D have a higher percentage of degradation compared to sample A. This indicates that nickel doped samples has higher photocatalytic activity on methylene blue degradation. The smaller crystallite size and lower band gap value had the important effect on photocatalytic activity. This conclusion has been proved by Tan<sup>[15]</sup>, where the addition of doping was able to increase the photocatalytic activity of the synthesized material. Tan<sup>[15]</sup> also study the relation between crystallite size and active surface area, smaller crystallite size led to larger active surface area. The large surface area of the photocatalyst will increase the rate of photocatalytic degradation of organic pollutants because the availability of active sites in the photocatalyst increases<sup>[15]</sup>.

From several research, TiO<sub>2</sub> with the addition of metal doping in the form of iron (Fe), fluorine (F), and boron (B) can degrade methylene blue in the UV light region by 72%, 92%, and 98%<sup>[21-25]</sup>. Meanwhile, in this study, the addition of nickel (Ni) doping on TiO<sub>2</sub> was able to degrade methylene blue under UV light by 92.73% (sample C). This shows that the use of nickel metal as doping in TiO<sub>2</sub> semiconductors is better than iron and fluorine metals<sup>[21-25]</sup>. The highest photocatalytic activity of sample C shows that low band gap has effect for photocatalytic activity, as it is proven also by several study<sup>[29-30]</sup>.

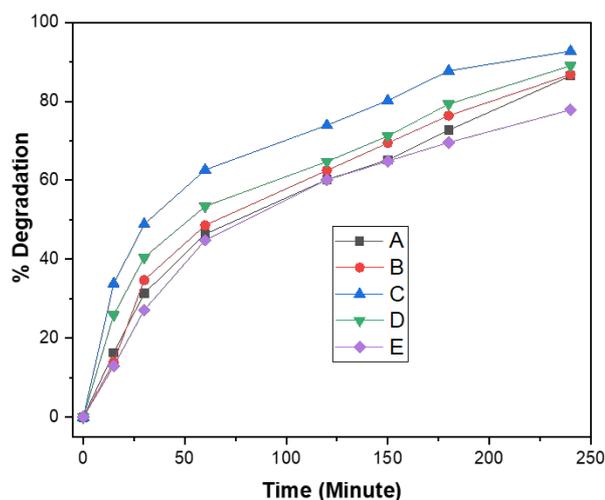


Figure 5. The photocatalytic activity of TiO<sub>2</sub>/Ni samples during methylene blue degradation

Table 4. The Degradation of methylene blue for TiO<sub>2</sub>/Ni samples after 240 minutes

Samples	Degradation of Methylene Blue (%)
A	86.47
B	86.81
C	92.73
D	88.99
E	77.83

#### 4. Conclusion

The TiO<sub>2</sub>/Ni photocatalyst can be synthesized from TiO<sub>2</sub> (P25) and Ni(CH<sub>3</sub>CO<sub>2</sub>)<sub>2</sub>·4H<sub>2</sub>O by using one step hydrothermal method in an autoclave at 120 °C for 24 hours. From the analysis using X-Ray Diffraction (XRD) the crystal sizes for samples A, B, C, D, and E were obtained, was 8.09, 7.88, 7.30, 7.83, and 8.16 nm. From the analysis using the DRS spectrophotometer, the band gap energy for samples A, B, C, D, and E was obtained, was

3.16, 3.15, 3.12, 3.14, and 3.20 eV. The results of XRD and DRS showed that the addition of nickel doping on the TiO<sub>2</sub> lattice has effect on reduction of crystallite size and the band gap energy value, that led to increasing of its photocatalytic activity. Sample C with a molar ratio of TiO<sub>2</sub> and Ni of 20:1 was able to degrade 92.73% of methylene blue in 240 minutes. While samples A, B, D, and E were able to degrade 86.47%, 86.81%, 88.99%, and 77.83% methylene blue.

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