# CHARACTERIZATION OF MICROSTRUCTURAL AND OPTICAL PROPERTIES OF $MgFe_2O_4$ NANOPARTICLES FOR PHOTOCATALYST OF MERCURY (Hg)

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**ABSTRACT**

The photodegradation activity of $MgFe_2O_4$ nanoparticles for Hg waste has been successfully performed. The $MgFe_2O_4$ nanoparticles have been synthesized by the coprecipitation method. The obtained sample was characterized for their microstructural properties by using X-ray diffraction. The results confirmed that the sample was polycrystalline and has a spinel crystal structure with space group Fd3m. The size of the crystallite is about $29.4 \pm 0.2$ nm. The gap energy of the sample was calculated from the UV-Vis results (4.17 eV). Furthermore, the $MgFe_2O_4$ nanoparticles can be used as functional photocatalyst material for degradation of mercury (Hg) ions in wastewater.

**Keywords:** Characterization; Mercury; Microstructural; Optical; Photocatalyst

## Introduction

Pollution by heavy metals and chemicals in industrial processes contained in the water has a dangerous impact on human health. Several heavy metals that frequently found in contaminated water are manganese (Mn), sulfur dioxide ($SO_2$), lead (Pb), arsenic (As), chromium (Cr), cadmium (Cd), and mercury (Hg).[1,2] One of those materials that very dangerous is mercury (Hg). The presence of mercury presumably comes from the waste disposals and various human activities, the gold mining carried out with the amalgamation method, for instance. In addition, other industrial activities like paint, electronic, pulp/paper, fungicidal herbicides, insecticide, and so on. In the aquatic environment, mercury can be mostly identified in the form of Hg ion, which may be turned into Methylmercury ($CH_3Hg^+$). The change is possible due to the microorganism activity is done by *Desulfovibrio desulfuricans ls*.[3] The $CH_3Hg^+$ is volatile. Thus, it can be widely dispersed in the environment. The Hg ion, either in the form of the metal ion, in $CH_3Hg^+$, or in vapor, can infiltrate the human body in several ways, including the food chain. It can contain Hg (II) ion and $CH_3Hg^+$, which are transmitted through the process of bioaccumulation and biomagnification contained in the fish and other aquatic biotas. As a result, the level of mercury can be high.

Meanwhile, the vaporized mercury is carried into the human body through breathing. The material is then taken to the brain over the bloodstream by penetrating the boundary between the blood and the brain. As a result, it may cause the damage to the system of central nerve. In addition, the existence of Hg (II) ion, and the compounds of $CH_3Hg^+$ in the human body can damage the kidney and it may lead to death.[3]

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Therefore, to overcome the problem, the waste purification can be taken as a solution. The methods carried out in the waste purification are varying include, adsorption, precipitation, evaporation, solvent extraction, ion exchange, membrane filter, coagulation-flocculation, flotation, reverse osmosis, and photocatalysis.[1]

The photocatalysis method, which is being developed, is a way of minimalizing the industrial waste pollution towards the environment. It utilizes the sunlight to degrade the waste of heavy metal impurities. Moreover, the photocatalysis is a substance that can accelerate the chemical reaction by exploiting photons as the trigger for an energy source.[4] Semiconductor material plays an important role in photocatalysis reactions. If the semiconductor materials irradiated by the sunlight, it will produce electrons and holes, which act as the oxidation and reduction agents during the reactions. Many of studies have applied $TiO_2$ as the photocatalysis material. Hence, by using other materials for photocatalysis reactions like particular nanoparticles elements can promote specific findings in order to activate the material of which.

The research on nanotechnology in the field of environment, specifically in the countermeasure research of heavy metal pollution, attracted big attention against absorption chemistry and nanoparticle-based materials of photocatalysis. The nanoparticle materials have cheaper costs, saving times, easiness of production, and proofs of overcoming the environmental pollution.

In this study, the $MgFe_2O_4$ was used as nanoparticle material since its magnetization and saturation values are high. Besides, the Curie temperature and electrical resistivity are also high. The $MgFe_2O_4$ is an *n*-typed of soft magnetic semiconductor material. This magnetic material has an inverse spinel crystal structure.[5] Additionally, the nanoparticle of $MgFe_2O_4$ possesses the affinity with the metal ion, which is large enough. The $MgFe_2O_4$ is more responsive to the external magnetic field since it has superparamagnetic properties.[5] This research is focussed on carrying out a photocatalysis process of $MgFe_2O_4$ nanoparticles for degradation of ion in the contaminated water by mercury ion. Hence, the photocatalysis activity of the $MgFe_2O_4$ nanoparticles is studied in accordance with their microstructural and optical properties.

## Methods

The nanoparticle of $MgFe_2O_4$ was synthesized by using a precursor of $MgCl_2.6H_2O$ and $FeCl_3.6H_2O$ via coprecipitation method with molar comparison of 1:2. This process was started by mixing of 1.01 gram $MgCl_2.6H_2O$; 2.70 gram $FeCl_3.6H_2O$ and 3.7 mL HCL into 50 mL solvent. The solution was stirred continuously for two minutes until it homogeneity. Next, the precursors' solution was titrated into 25 mL of NaOH solution while constantly stirring at 1000 rpm and a temperature of 120 $^oC$ for 60 minutes. The obtained solution was then placed on a magnet table for another 60 minutes to achieve a room temperature and the slurry being separated from the solvent. To reduce the undissolved metal ion, the slurry of $MgFe_2O_4$ was rinsed with double distilled water and re-precipitated over a magnet table ten times. This process was carried out five times. Finally, the slurry of nanoparticle was heated at a temperature of 100 $^oC$ for 5 hours. It produced a solid $MgFe_2O_4$ nanoparticle in the form of chips. These solid pieces were characterized for their microstructural and optical properties by using XRD Shimadzu XD and UV-VIS Spectrometer.

## Results and Discussion

The X-ray diffraction pattern of the $MgFe_2O_4$ nanoparticle is shown in Figure 1. The XRD pattern proved that the sample was in accordance with the standard data of $MgFe_2O_4$, i.e., JCPDS No. 88-1935. The diffraction pattern shows that the appearance of five diffraction peaks, which are the characteristics of $MgFe_2O_4$ and confirms that the samples are a polycrystalline material. The nanoparticle has a spinel crystal structure with space group Fd3m. The highest intensity of the diffraction peak was taken place in (311) with a diffraction angle $2\theta$ of 35.42 $^{o}$C.

Accordingly, the crystallite size of nanoparticle was successfully calculated by using Scherer's equation.[6] The size is 29.4 ± 0.2 nm and the derived lattice constant *a* is 8.4 ± 0.1 Å.

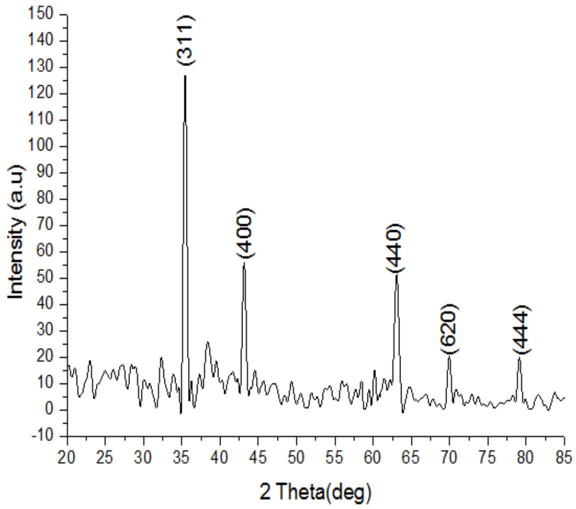


**Figure 1.** The XRD diffraction pattern of the $MgFe_2O_4$ nanoparticle

Moreover, the optical property of the sample was represented by using the UV spectrum of absorbance against photon wavelength (Figure 2).

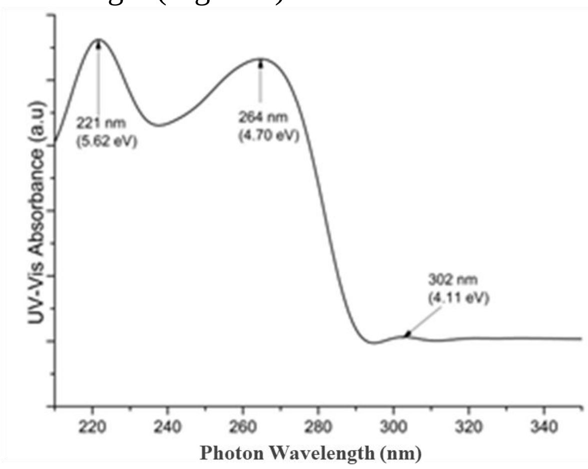


**Figure 2.** The absorbance against the wavelenght

Accordingly, Figure 2 shows that the appearance of three absorbance peaks at related wavelengths and photon energies of 221 nm (5.62 eV), 264 nm (4.70 eV), and 302 nm (4.11 eV). Based on the absorbance spectra, the highest elevation of the absorbance takes places between wavelengths of 302 nm and 264 nm. Physically, this phenomena confirms that the energy needed by an electron to excite from valence band to conduction band is in the range of 4.11 eV to 4.70 eV.

Regarding those phenomena, the calculated optical gap energy of $MgFe_2O_4$ nanoparticles by applying the *Kubelka-Munk* method[6,7] is about 4.17 eV such as figured out in Figure 3. This result is consistent with the absorbance spectra in Figure 3.

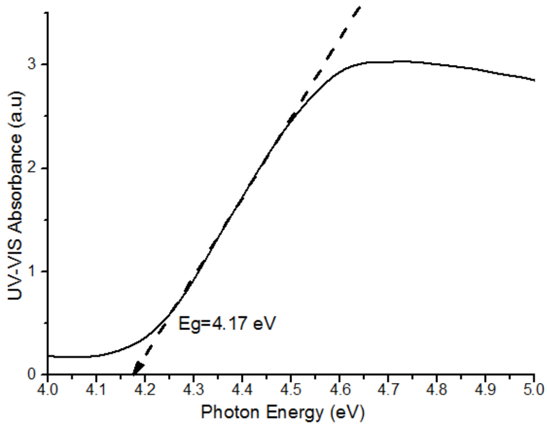


**Figure 3.** Tauc plot

This optical gap energy is higher than the result discovered by.[8] On this circumstance, the excitation type of inter-band could not be specifically determined into direct-gap or indirect-gap. It might be caused by the imperfect microstrain and lattice, which were shaped by the crystallite.[7] Presumably, the flawed crystal affected the number of the energy gap, which was resulted as the effect of the quantum confinement.

Furthermore, several UV radiations were carried out to test the photocatalytic activity of sample nanoparticle against Hg (II) ion (Figure 4). The duration of radiation were 30 minutes, 60 minutes, 90 minutes, 120 minutes, and 150 minutes.

The time duration of UV radiation during photocatalysis process represents the period of which photon trigger electron in the valence band of $MgFe_2O_4$ to excite into the conduction band across 4.17 eV of the optical gap energy. It also marks the length

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of interaction between the photocatalyst with the radiation and the mercury ion.

Consequently, the longer duration of the radiation occurred, the more effective contact between the photocatalyst and the ions. Therefore, some of the electrons were formed over the surface.[9] The reaction leaves holes having a strong oxidizing agent to form $OH^+$ radical which degrade Hg ion (II).

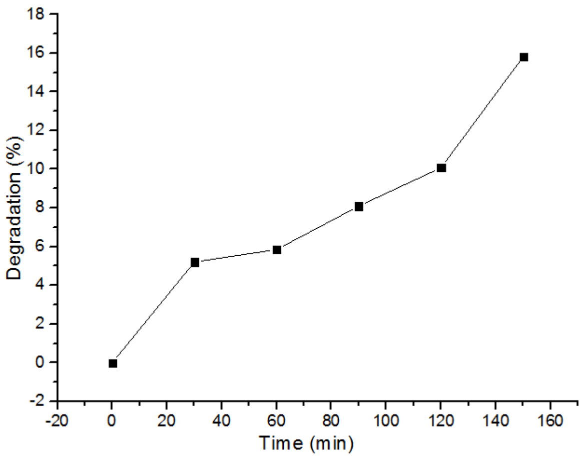


**Figure 4.** Percent degradation of Hg ion during specific reaction times

Figure 4 also informs that the increase in percentages degradation of Hg ion during 120 minutes of radiation time. However, at the time of 120 minutes to 150 minutes, the degradation decline by 3%. This was caused by three factors. First, the $MgFe_2O_4$ nanoparticle has reached the optimum time to degrade Hg ion. Thus, the point of saturation was detected at 120 minutes. Second, the numbers of Hg ion (II) of the process of photo-reduction, which were attached over the surface of the $MgFe_2O_4$ nanoparticle resulted from the interaction between the UV radiation and the sample. It affects the amount of the electrons which consequently declined the reaction of photo-reduction of Hg ion.

In accordance with those factors, it can be taken into account that the percentage of degradation against Hg ion (II) frequently tended to increase. Thus, it can be concluded that the $MgFe_2O_4$ nanoparticle may be referred to as a candidate for nano-photocatalyst under UV radiation. Further studies need to be done to improve the microstructural and optical gap energy of nanoparticles in other able to be used for photocatalysis process under visible light radiation.

## Conclusions

The $MgFe_2O_4$ nanoparticle had been successfully synthesized and characterized for their microstructural and optical properties. The size of crystallite nanoparticle is 29.4 ± 0.2 nm. The optical gap energy is 4.17 eV. Accordingly, the nanoparticles were found effective in degrading Hg ion in wastewater. The photodegradation increases as increasing radiation time for the photocatalysis process. Furthermore, the $MgFe_2O_4$ nanoparticle can be a candidate for functional photocatalyst material for reducing Hg ion in wastewater.

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