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Photocatalytic Activity of MgO derived from Bangkalan Dolomite

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Abstract

In the industrial sector, water pollution due to dye waste needs special attention because it can harm humans and the environment. A waste-absorbing material with good photocatalytic properties is required to overcome this problem. The photocatalytic properties are owned by metal oxide materials, one of which is Magnesium Oxide (MgO) material. This study aims to analyze the activity level of MgO photocatalytic from Bangkalan dolomite. MgO synthesis was carried out by the leaching method, where UV-Vis characterized the results to determine the photocatalytic ability of MgO against methylene blue (MB). In this characterization, various treatments (sunlight, UV light, and darkroom conditions) were carried out with radiation times of 180, 240, and 300 minutes. The results of the UV-Vis spectrum analysis showed that MgO had a band gap of 4.01 eV. The degradation efficiency of photocatalytic obtained optimal under sunlight, UV light, and in a dark room with a radiation time of 300 minutes at 82%, 42%, and 34%. Thus, the synthesis of MgO in sunlight showed good photocatalytic activity in reducing MB dye to use for processing waste from the dye industry.

Introduction

The development of the textile industry is currently increasing, impacting the increasing amount of organic dye waste, resulting in water pollution. Water pollution due to organic dyes can threaten the human body, especially for people who live in aquatic environments, because they are carcinogenic [1]. One of the dyes used in the textile industry is methylene blue which is used as a basic dye for leather, mori cloth, cotton cloth and tannin [2]. Methylene blue dye has a benzene structure which is difficult to decompose, is toxic, carcinogenic and mutagenic so it is very dangerous when exposed to skin, eyes or even swallowed [3]. In addition, the more dye molecules in the water will also interfere with the photosynthesis process and damage aesthetics [4]. The various ways to overcome water pollution include using standard physicals that are effective, such as absorption, but still require additional steps to regenerate absorbents [5]. The biological methods are considered capable and environmentally friendly in overcoming the problem of water pollution due to organic dyes, but they can still not destroy organic dyes. Chemical processes such as photocatalytic degradation of organic dyes

using sunlight or UV are among the promising techniques and are regarded as efficient due to benefits such as cost-effectiveness and no secondary pollutant regeneration [6].

Photocatalytic is generally defined as a chemical reaction process that is assisted by light and a solid catalyst. The light will form electrons and holes *hole* ($e^-$and h^+). Electrons react with oxygen in water to form 0_2^- anions which then strongly oxidize hydroxyl radicals (•OH). While the holes oxidize the dissolved hydroxyl and convert it into a radical with great energy. Hydroxyl radicals with high energy will decompose organic pollutants in liquids into gases which then evaporate or become other harmless substances [7].

Photocatalytic use metal oxides to accelerate the process of destroying certain harmful compounds and pathogens [8]. The use of metal oxides as photocatalytic has also shown promising potential for various environmental applications, including mineralization of various organic pollutants, water disinfection, and gas sensing of wastewater [9]. TiO_2, ZnO, WO_3, Fe_2O_3, and CuO are metal oxides that have been extensively investigated and can be used as photocatalytic materials to decompose organic pollutants in the air, soil, and water [10–17]. Recently, metal oxides capable of acting as photocatalytic materials are MgO [18]. MgO has superb thermal conductivity, low refractive index, wide band gap and low dielectric constant to be widely applied in photocatalytic, semiconductors, and absorption [19].

MgO can be obtained from synthesizing commercial materials containing Mg^{2+} and natural materials such as dolomite. Dolomite is limestone with the chemical formula $CaMg(CO_3)_2$, which includes a clump of carbonate minerals, i.e. a combined mineral of magnesium carbonate ($MgCO_3$) and calcium carbonate ($CaCO_3$). Theoretically, dolomite minerals contain 45.6% $MgCO_3$ or 21.9% MgO and 54.3% $CaCO_3$ or 30.4% CaO [20]. Composition of Bangkalan dolomite is shown in **table 1.**

Table 1. The composition of Bangkalan dolomite minerals [21]

Compund	Grade (% weight)
CaO	63,42
MgO	26,39
Na_2O	5,93
SiO_2	1,20
$Al2O_3$	0,86
Fe_2O_3	0,74

Research conducted by Wulancahayani & Rohmawati, (2020) identified four phases of Bangkalan dolomite content, i.e. 44.8% $CaCO_3$, 38.1% MgO, 14.9% $Ca(OH)_2$, and 2.2% CaO. As catalyst, Khan et al, (2020) reported that photocatalytics using MgO under dark, sunlight, and UV light conditions were optimal with a contact time of 250 minutes and obtained degradation values respectively 25%, 58%, and 81%. Saputri & Rohmawati, (2021) reported the synthesis of MgO nanoparticles from Bangkalan dolomite extract using the leaching method. This study found that the MgO phase was completely formed with a volume fraction of 100% at a calcination holding time of 8 hours. However, Saputri & Rohmawati, (2021) have not reported photocatalytic activity of MgO from Bangkalan dolomite.

The studies above were conducted to analyze the level of photocatalytic activity of MgO from Bangkalan dolomite. The photocatalytic ability of MgO synthesis was investigated by detecting the photodegradation of MB dye under sunlight, UV light, and darkroom conditions.

This study of results can later be used for photocatalytic information for processing waste from the dye industry.

Experimental Method

Synthesis of MgO

Referring to Saputri & Rohmawati, (2021) research, the MgO synthesis process was carried out by dissolving 50 grams of dolomite powder into 210 ml of 5M HCl solution, stirring for 45 minutes at 75 ^{o}C, then filtering the solution using a filter paper. The filtered filtrate was added with 25% NH_3 to pH 12 and allowed to stand until a precipitate is formed, then washed with distilled water and heated at 90˚C for 6 hours. Furthermore, the resulting powder was calcined at a temperature of 800˚C for 8 hours.

Characterization

Data collection for the structural properties of MgO was characterized by XRD using the PAN analytical X'pert PRO system equipped with Cu-Kα_1 (λ = 1.540598) operated at 40 kV and 30 mA with an angle of 2θ range (20 - 90˚). Identify the phase composition formed in the sample using Qual-X software and perform analysis with the Origin software The calculation of crystal size (D) using the Debye-Scherrer formula.

$$\left(D = \frac{K\lambda}{\beta \cos \theta}\right) \quad (1)$$

The absorbance level of the synthesized sample was characterized using a UV-Vis spectrophotometer SPECORD 200 PLUS. Calculation of the band gap using the Tauc plot. The photocatalytic degradation of MgO against MB was marked by a UV-Vis spectrophotometer in the 300 to 700 nm wavelength range. Photocatalytic degradation is calculated using the formula:

$$\left(\%D = \frac{C_0 - C_t}{C_0} \times 100\%\right) \quad (2)$$

Photocatalytic Activity

The potential photocatalytic of MgO against MB dye was carried out using various treatments with visible light radiation time, as illustrated in **Figure 1**. Referring to Khan et al., (2020) study, 100 mL of a solution containing 20 ppm MB and 10 mg MgO was stirred constantly for 40 minutes. Then the effect of radiation level was observed, i.e. the reaction under UV light (10 W with 30 mW/cm^{-2}), sunlight, and conditions in a dark room. Each uses a time variation of 180, 240, and 300 minutes. Furthermore, 5 mL of suspension from each treatment was centrifuged for 5 minutes at 3000 rpm.

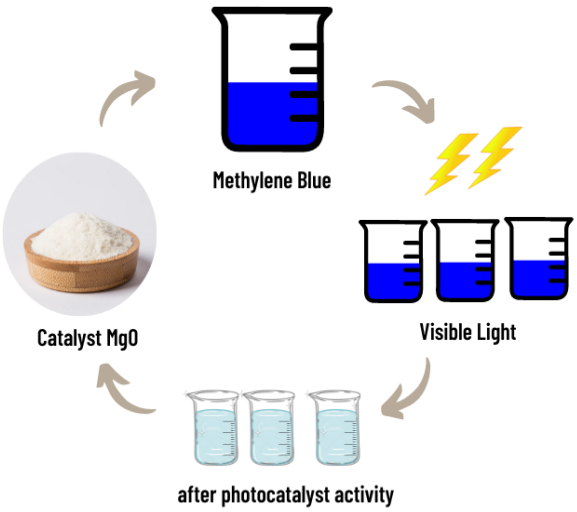

Figure 1. Photocatalytic mechanism

Result and Discussion

XRD analysis

Data from XRD performed analysis techniques to obtain preliminary information about the prepared samples' crystal structure, phase properties, and crystal grain size. **Figure 2** shows the XRD diffractogram pattern from the MgO synthesis. The observed peaks at 2θ = 36.964˚, 42.930˚, 62.303˚, 74.661˚ and 78.609˚ correspond to the hkl values [111], [200], [220], [311] and [222] with polycrystalline cubic structures, which correspond to with JCPDS standard (No: 78-0430) [24]. No other characteristic impurity peaks were observed, indicating that the synthesized MgO nanoparticles were high purity. The crystal size (D) of MgO was calculated using the Debye Scherrer **Equation 1**.

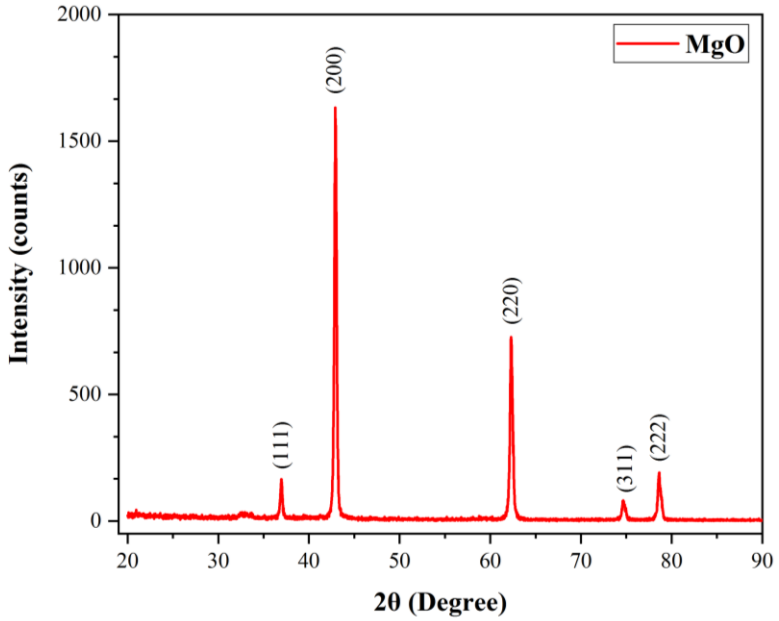

Figure 2. XRD Pattern of MgO

The result of the analysis from XRD that found the crystal size (D) calculated from the maximum diffraction peak (2θ = 42.93˚) in the lattice plane (200) was found to be 64.2 nm with an average crystal size of about 60.5 nm. Saputri & Rohmawati, (2021) prepared the synthesis of MgO by leaching method, showing a crystal size of 20.12 nm. Jannah & Rohmawati, (2018) also experimented on synthesizing nanocrystalline $CaCO_3$/MgO using the mixing method, where $CaCO_3$ has a crystal size of 21.07, and MgO powder has a crystal size of around 47.72 nm. Khan et al., (2019) stated that crystallites are defined as materials with a 1 - 100 nm size range. Thus the sample of MgO synthesized from dolomite includes nano-size [23].

UV-Visible spectroscopy analysis

The UV-Vis absorption spectrum of MgO was analyzed in the wavelength range of 200 to 700 nm and is shown in **Figure 3**. The UV-Vis absorption spectrum of the MgO sample showed that the absorption peak was strong at 260 nm. The absorption peak in this wavelength range follows research conducted by Rastogi et al, (2015) which stated that MgO has a broad absorption peak at 260 - 330 nm. If the surface characterization band appears at a wavelength of <300 nm, it indicates the presence of a small crystal size [28] which was confirmed by XRD.

Figure 4. shows the tauc plot of MgO synthesis which has an optical energy band gap of 4.01 eV, and this value is close to the results of research conducted by Khan et al. (2020), where MgO has a band gap value of 4.1 eV, and very effective in terms of photocatalytic degradation, maximum 81% against MB dye. Thus, the synthesized MgO has the potential to degrade MB, which in this case, is a dye used by industry.

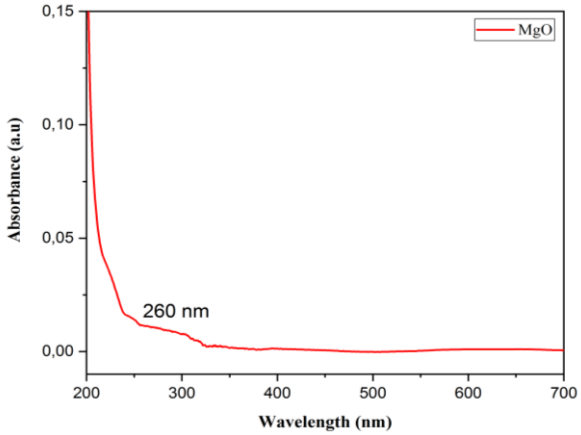

Figure 3. UV-Vis of MgO

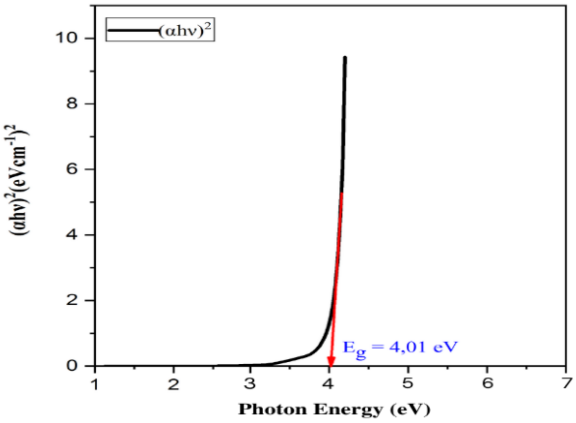

Figure 4. Tauc Plot of MgO

Photocatalytic degradation studies

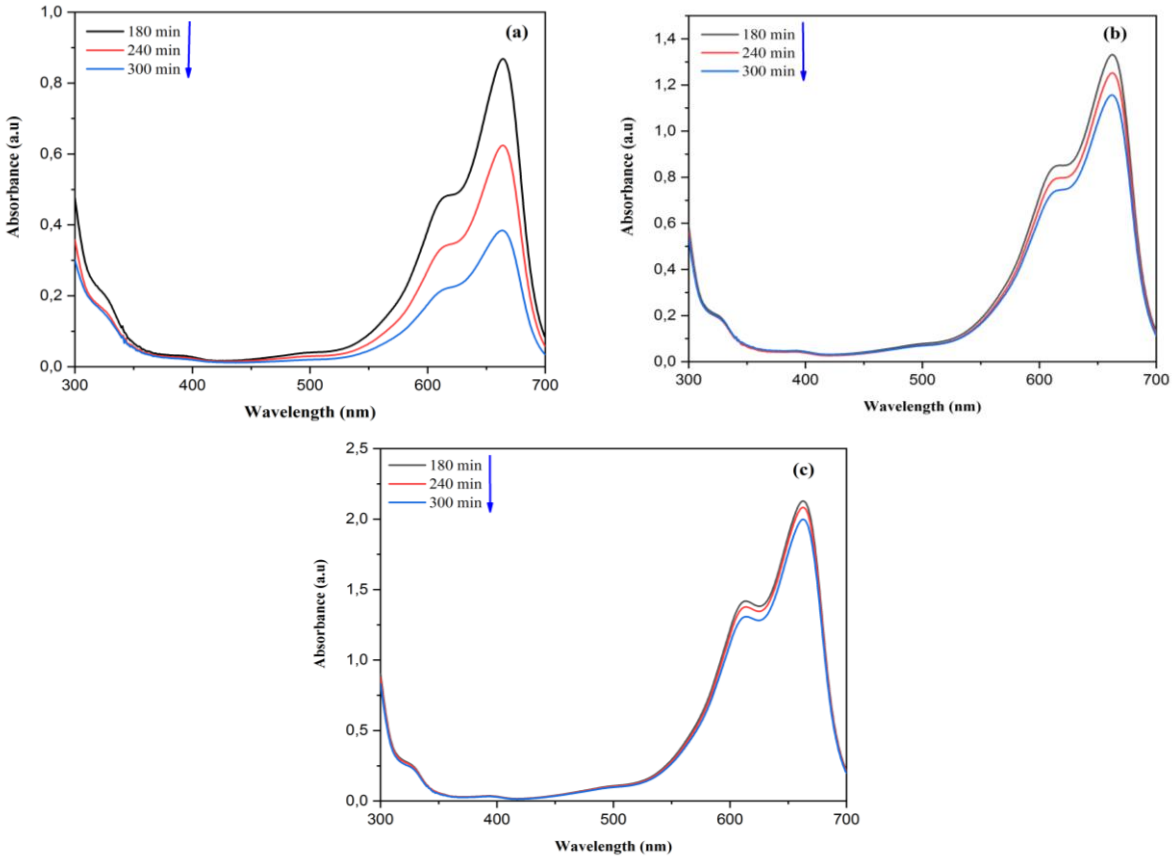

Figure 5. Absorption spectrum of MB dye at each time interval (180-300 minutes) (a) Sunlight (b) UV light (c) darkroom conditions

Table 2. Comparison of MgO photocatalytic degradation activity against some organic dyes

Catalyst	Energy Gap (eV)	Catalyst preparation method	Organic dyes	% of degradation	Time taken to degrade (min)	Ref.
MgO *Dalbergia Sissoo*	4,1	Green Synthesis	MB	81	250	[18]
MgO	2,9	Combustion	MB	75	120	[29]
MgO	-	Co-precipitation Hydrothermal	MB	- -	30	[30]
MgO	5,93	Microwave	MB CR	88 82	90 120	[31]
	5,85	Hydrotherma	MB CR	92 86	90 120	
MgO	5,67	Reflux condensation	MO MB	96,48 99,14	210 240	[19]
MgO Watermelon	4,97	Combustion	MB	97 92	180	[32]
MgO Dolomite	4,01	Leaching	MB	82	300	**Present work**

In this study, MB dye was used as a model contaminant to develop MgO photocatalytic activity under sunlight, UV light, and darkroom conditions. **Figure 5** shows the absorption spectrum of MB dye by MgO. On irradiation for 180 - 300 minutes, there was a significant increase in MB degradation. **Figure 5 (a) & (b)** MB solution which was initially blue, gradually faded to colourless until the maximum radiation time limit. So that by encouraging the development of MgO, there will be a decrease in the concentration of MB. As a result, The absorption value also decreases with the length of time irradiation with visible light [3].

The ability of photocatalytic degradation under conditions of sunlight exposure obtain higher yields than UV light is because sunlight has an intensity and wavelength between 310 - 2300 nm, which is greater than that of UV light (200 - 380 nm) [33]. Sunlight is a combination of visible light (45%) and UV light (5%), so it has relatively significant energy and can provide a lot of photon energy to form strong •OH radicals in photocatalytics [34–37]. This photon energy can cause the excitation of electrons from the valence band to the conduction band, the existence of this electron excitation process causes holes in the photocatalytic. This hole will oxidize methylene blue when it reacts with water, which will produce more hydroxyl radicals.

In the darkroom conditions, a low degradation value was obtained because there was no light source, the photocatalytic did not absorb photons to excite electrons to the conduction band so that no holes were produced in the photocatalytic system and the photocatalytic only acts as an absorber, the energy possessed by MgO was weak; as a result, the photon energy to produce •OH radicals was also low [38]. It can see from **Figure 5 (c)** that MB dye only absorbs on the surface of the catalyst and makes its degradation not optimal. To calculate the efficiency of photocatalytic degradation using **Equation 2**. The ability of MgO photocatalytic against MB dye in the treatment of sunlight, UV light and dark room conditions obtained degradation values of 82%, 42%, and 34%, respectively, within 300 minutes. The longer the irradiation, the better the photocatalytic effectiveness in degrading MB color, adsorption and desorption will continue to be in equilibrium, until they reach optimum conditions and degradation tends to be stable near 100%, as shown in Figure 5 for all conditions, i.e. in sunlight, UV light, and a dark room. In this photocatalytic study, it was shown that •OH radicals were generated more by photon energy from solar radiation than photon energy from UV light [38]. The reaction mechanism of MgO on the degradation of methylene blue dye is shown in **Figure 6**.

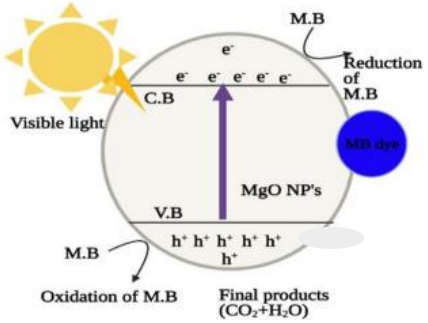

Figure 6. Mechanism degradation of MB dye [39]

When the semiconductor catalyst is exposed to light with a higher energy, the electrons (e^-) in the valence band are excited towards the conduction band and will leave holes (h^+) in the

valence band. Hole (h^+) will interact with H_2O and $0H^-$ on the surface of the catalyst to form OH radicals (•OH) which act as strong oxidizing agents. Electrons (e^-) will react with O_2 in the catalyst to form superoxide radicals ($\bullet\, 0_2{}^-$) which act as reducing agents. The oxidizing and reducing agents attack the methylene blue dye to produce CO_2 and H_2O and some acids with low concentrations [4]. Suppressing electron-hole pairs generates radicals and superoxide which degrade dye molecules and organic pollutants [40].

Based on the results of the radiation irradiation above, sunlight can be used as a very potential light source for photocatalytics in degrading MB dye to be used for processing waste from the dye industry. Many previous researchers have researched adsorption. Therefore, it can be used as a research reference, as shown in **Table 2**.

Conclusion

The results of the UV-Vis spectrum analysis showed that MgO had a band gap of 4.01 eV. The optimal irradiation time is 300 minutes in sunlight, UV light and dark room conditions with degradation results of 82%, 42% and 34%, respectively. The longer the irradiation, the more MB dye is degraded. The synthesis of MgO in sunlight showed a photocatalytic degradation activity greater than UV light in reducing MB dye, which can use to process waste from the dye industry. Based on the above results and discussion, MgO is suitable to be used as a material catalyst candidate to prevent air pollution caused by dyes, especially MB. For further work, SEM or TEM tests are needed to determine particle size, BET tests to estimate surface area and ensure pollutant mineralization, and monitor TOC (Total Organic Carbon) during the degradation reaction.

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