



SYNTHESIS OF CaO/C NANOCATALYST AS PHOTOCATALYST IN METHYLENE BLUE DEGRADATION PROCESS USING UV LIGHT

Rohmatullaili^{*1}, Jefri¹, Ayu Aulia Devi¹, Siti Rodiah¹

¹Program Studi Kimia, Fakultas Sains dan Teknologi, UIN Raden Fatah, Palembang, 30254.

DOI: 10.20414/spin.v6i1.8546

History Article

Accepted:

October 19, 2023

reviewed:

Feb 28, 2024

Published:

July 13, 2024

Kata Kunci:

fotodegradasi,
fotokatalis,
material, Metilen
biru, nanokatalis.

Keywords:

photodegradation,
photocatalyst,
material, Methylene
blue, nanocatalyst.

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ABSTRAK

Fotodegradasi metilen biru telah dilakukan dengan bantuan sinar UV dan material CaO/C hasil sintesis. Material awal berupa CaO cangkang kerang mas dan serbuk kulit jengkol, yang dikalsinasi pada suhu 900°C sehingga diperoleh nanokatalis CaO/C. Hasil karakterisasi gugus fungsi dengan spektrofotometer FT-IR menunjukkan gabungan antara spektra CaO dan serbuk kulit jengkol murni. Proses fotodegradasi dilakukan dengan optimasi massa katalis, pengaruh sinar, dan waktu degradasi. CaO/C menunjukkan massa optimum sebesar 50 g dalam larutan metilen biru 25 ppm dengan persentase penurunan konsentrasi metilen biru 5,2%. Waktu optimum CaO/C dicapai pada waktu 120 menit dengan persentase penurunan konsentrasi sebesar 20,968%. Kinetika reaksi CaO/C mengikuti model kinetika orde ke-1 dengan nilai konstanta 0,0018 min⁻¹.

ABSTRACT

Photodegradation of methylene blue has been carried out with the help of UV light and synthetic CaO/C material. The starting materials are CaO from golden snail shells and jengkol shell powder, which are calcined at a temperature of 900°C to obtain CaO/C nanocatalysts. The results of functional group characterization using an FT-IR spectrophotometer show a combination of the spectra of CaO and pure jengkol peel powder. The photodegradation process is carried out by optimizing the catalyst mass, the influence of light, and the degradation time. CaO/C shows an optimum mass of 50 g in a 25 ppm methylene blue solution with a percentage reduction in methylene blue concentration of 5.2%. The optimum time for CaO/C was achieved at 120 minutes with a concentration reduction percentage of 20.968%. The kinetics of the CaO/C reaction follows a 1st-order kinetic model with a constant value of 0.0018 min⁻¹.

How to Cite

Rohmatullaili., Jefri., Devi, A. A., & Rodiah, S., (2023) Synthesis of CaO/C Nanocatalyst as Photocatalyst in Methylene Blue Degradation Process Using UV Light. *SPIN-Jurnal Kimia & Pendidikan Kimia*. 6(1). 1-9.

*Correspondence Author:

Email: rohmatullaili_uin@radenfatah.ac.id

p-ISSN: 2580-2623

e-ISSN: 2745-6854

INTRODUCTION

The increasing development of the textile industry in 2022 reached 12.45%, compared to the previous year, namely 5.94% (Central Statistics Agency, 2021). This increase is also accompanied by increased waste produced, especially synthetic dye waste, which is difficult to degrade naturally, can accumulate, and is toxic (Jannah, 2020). The amount in certain concentrations in water will cause environmental pollution (Wijaya et al., 2019). One of the widely used synthetic dye wastes is methylene blue because it is a basic dye and is relatively cheap and easy to obtain. Methylene blue has a higher level of toxicity than other dyes (Jannah, 2020). Long-term exposure to methylene blue in humans can cause serious health problems such as tachycardia (increased heart rate), vomiting, shock, cyanosis, jaundice, paralysis of the legs, burns to the eyes, and problems with tissue necrosis (Fajarwati et al., 2016).

The heavy use of methylene blue in the textile dyeing process has caused around 17–20% of water pollution because the effluent tends to be discharged into seas, rivers, and lakes without or minimal treatment (Zhu et al., 2020). Various methods have been used by many previous researchers in the process of removing methylene blue from waters, including using adsorption and activated sludge methods. The adsorption method still leaves sludge, which is less effective (Riwayati et al., 2019). Meanwhile, adsorption using activated sludge is considered less than optimal because several types of dye waste have properties that are difficult to degrade (Juhra & Notodarmojo, 2019). An alternative method considered quite promising is photodegradation, where complex compounds from dye waste will be degraded into simple components that are safer for the environment, relatively cheap, and easy to apply (Al-Abror, 2021).

Several studies have reported that photodegradation methods can remove methylene blue in aquatic systems. Photodegradation of methylene blue using

carbon nanocatalysts from solid olive oil waste was carried out by Salwaha et al. (Sawalha et al., 2021) with a degradation percentage of 92%. The photodegradation process of dye waste will produce simple compounds such as CO_2 , H_2O , and mineral acids (Sugiyana & Notodarmojo, 2015). Novrian et al. reported that the methylene blue degradation process using sunlight and a ZnO-SnO catalyst could reach a degradation percentage of 88% (Novrian et al., 2018).

The photodegradation process is carried out using light and the help of semiconductor materials. Semiconductor materials will increase photocatalytic activity by absorbing light (Nguyen et al., 2021). Semiconductor materials that have been used in the photodegradation process include TiO_2 (Al-Abror, 2021), ZnO (Juhra & Notodarmojo, 2019), Al_2O_3 (Widihati et al., 2017) and CaO (Vanthana et al., 2020). One of the photocatalyst materials that is widely used is calcium oxide (CaO). Calcium Oxide (CaO) can be obtained from various materials containing CaCO_3 , which are then calcined to obtain CaO (Sugiyana & Notodarmojo, 2015). As done by Sibarani et al. (Sibarani et al., 2020), Oktavia (Oktavia, 2021), and Mohadi et al. (Mohadi et al., 2019) which use pigeon shells and crab shells as a source of CaO to be used as an adsorbent, egg shells as a catalyst and chicken bones as a catalyst. Apart from that, CaO was also found in golden snail shells, as in the research of Rodiah et al. (Rodiah et al., 2020) and Jefri and Rodiah (Jefri & Rodiah, 2022), who used CaO from golden snails shell as a catalyst for the transesterification reaction and as an adsorbent in the absorption of petroleum processing waste whose absorption capacity reached 100%.

CaO modification can increase its effectiveness or efficiency as a catalyst. Mohamed et al. modified CaO into CaO/C by utilizing egg shells and coffee grounds carbon. CaO/C material is used as a nanocatalyst in the methylene blue photodegradation process. The degradation percentage of methylene blue with

the CaO nanocatalyst reached 88.04%, while the CaO/C nanocatalyst resulted in a degradation percentage of 99.76% (Mohamed et al., 2021). Another natural material that has potential as a carbon source is jengkol bark (Pandia & Warman, 2016). The jengkol shell is the outermost part of jengkol, which can be used as a bio-adsorbent (Alfauzi et al., 2021). Jengkol skin contains high crude fiber (33.07-35.28%), dry matter (89.64-90.24), ash (3.15-3.48%), crude protein (7.9-8.41%), Neutral Detergent Fiber (NDF) (55.33-58.74%), Acid Detergent Fiber (ADF) (40.84-43.78%), hemicellulose (14.49-14.96%), and cellulose (26.99-28.23%) (Fildza et al., 2022; Pandia & Warman, 2016). Apart from that, there is a carbon element (C) of 44.02%, which is believed to be the most important element in making catalyst support (Jefri & Rodiah, 2022).

Based on the literature studies carried out in this research, CaO/C nanocatalysts were prepared. The CaO used resulted from previous research preparation by Rodiah et al., which comes from golden snail shells and is then modified with carbon from jengkol shells. The CaO/C nanocatalyst was characterized for its functional groups using an FT-IR spectrophotometer. The activity of the CaO/C nanocatalyst was observed in the methylene blue photodegradation process by varying the mass of the nanocatalyst, the influence of the light used, and the optimum time.

METHODS

Materials

The materials used in this research were methylene blue dye ($C_{16}H_{18}N_3SCl$) (Rafalab), calcium oxide (CaO) from golden snail shells, jengkol shells, and distilled water.

Tools

The equipment used in this research included standard glassware, Mettler Toledo analytical balance, Hammertz oven, magnetic stirrer, UV lamp (253nm), 100-mesh sieve, MDX-310 Tomy Centrifuge, Shaker Mill-Miller 1st High Energy Milling (HEM), Alpha Bruker FT-IR spectrophotometer, and Shimadzu UV-1900 double beam UV-Vis.

Preparation

Preparation and Characterization of CaO/C Nanocatalysts

CaO/C catalyst preparation refers to the procedure of Mohamed et al. (2021) with some modifications. The CaO/C catalyst was prepared by mixing CaO with jengkol bark powder (1:1) in 70 ml of distilled water. Then, the mixture is stirred using a magnetic stirrer until homogeneous. The mixture was then filtered and dried using an oven at 105°C until it reached constant weight. After drying, the mixture is calcined using a furnace at a temperature of 900°C for 4 hours to carbonize the jengkol skin. The calcined solids were ground and converted into nanoparticles using a ball mill at 1000 rpm for 4 hours. The nanoparticle powder obtained was called CaO/C nanocatalyst, which was further characterized using an FT-IR spectrophotometer.

Photodegradation Test

The methylene blue photodegradation test was carried out by mixing a certain amount of CaO/C nanocatalyst into a 50 ml 25 ppm methylene blue solution. The mixture was then stirred using a magnetic stirrer at 250 rpm. The photodegradation process begins when the mixture is exposed to sunlight for a certain time. Optimization of the photodegradation process was carried out by varying the mass of the CaO/C nanocatalyst used and determining the optimum time by varying the photodegradation time.

The photodegradation results were tested using a UV-Vis Spectrophotometer instrument to determine the remaining methylene blue concentration. Percent degradation (%D) of methylene blue is determined via the equation (1):

$$\%D = \frac{C_o - C_t}{C_o} \times 100\% \quad (1)$$

RESULT AND DISCUSSION

Preparation and Characterization of CaO/C Nanocatalysts

Mixing golden snail shell CaO with jengkol shell powder (*serbuk kulit jengkol* - SKJ) in water produces a thick, black solution with a pungent aroma, the amino acids cysteine and sulfur content in SKJ (Simbolon et al., 2017).

The material conversion from CaO-SKJ to CaO/C is carried out by heat treatment using a furnace. The resulting material is a fine white nano-sized powder called CaO/C nanocatalyst. The results of functional group characterization of the material successfully synthesized using the FT-IR Spectrophotometer are shown in Figure 1.

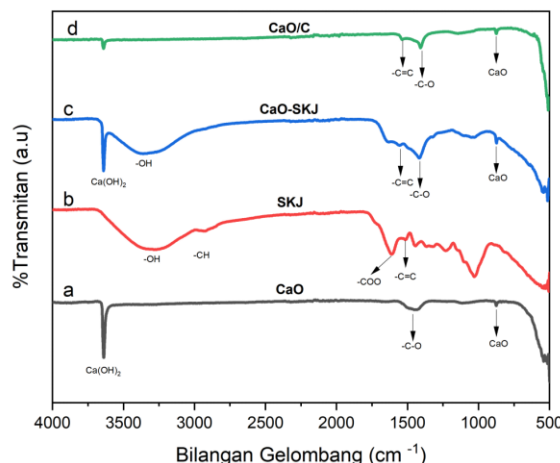


Figure 1. FT-IR spectra of a. CaO, b. SKJ, c. CaO-SKJ, and d. CaO/C

The FT-IR spectrum of CaO in Figure 1 (a) shows the stretching vibration of the O-H group (Ca(OH)_2) at a wave number of 3640.05 cm^{-1} , which is free O-H, the C-O group (CaCO_3) at a wave number of 1431.18 cm^{-1} , and Ca-O at wave number 873.91 cm^{-1} . The SKJ spectrum (b) shows the presence of an O-H group (bending mode) at wave number 3259 cm^{-1} , which is O-H bound by hydrogen bonds, a C-H group at wave number 2916.23 cm^{-1} , a -COO carboxyl group at wave number $1612, 76 \text{ cm}^{-1}$, and the C=C group (lignocellulose) at a wave number of 1540.57 cm^{-1} . The presence of lignocellulose makes the jengkol shell a carbon source (Hariati et al., 2017), which will be composited with CaO material.

The results of CaO-SKJ synthesis before calcination in Figure 1 (c) show the merging of the typical CaO peak with the jengkol shell. This is proven by the increase in peaks at wave numbers 3334.32 cm^{-1} and 1540.57 cm^{-1} , which are the functional groups of O-H and C=C (lignocellulose). Meanwhile, the CaO/C material resulting from calcination (Figure 1. d) shows a reduction in the intensity of the C=C (lignocellulose), O-H (Ca(OH)_2) groups and the

disappearance of the O-H groups (bending mode) bound to lignocellulose.

Peak changes in the spectra are caused by the calcination process (Budiarto, 2022). Previous research reported that the calcination process caused the disappearance of the -OH (bending mode) groups in the material, which was identified by the reduction of the peak at wave numbers $3500\text{--}3200 \text{ cm}^{-1}$ in the FT-IR spectra (Azalok et al., 2021; Oladipo, 2021; Ye et al., 2022). The calcination process also causes many -OH groups (Ca(OH)_2) to be converted into CaO, resulting in a decrease in intensity at the peak in the wave number area of 3600 (Hwidi et al., 2018).

The CaO/C material synthesized in this study has characteristics similar to those of the CaO/C material synthesized by Mohamed et al. (Mohamed et al., 2021). The CaO/C material that was successfully synthesized was made from CaO material (egg shells) with carbon from coffee grounds. The FT-IR spectra resulting from this research show the presence of the O-H (Ca(OH)_2) functional group at a wave number of 3643.69 cm^{-1} , the C=O (CaCO_3) group at a wave number of 1638.30 cm^{-1} , and the CaO in the fingerprint area. Based on

the FT-IR spectra, it can be assumed that CaO/C originating from golden snail shells and jengkol shells was successfully synthesized.

Photodegradation Test

Optimization of the photodegradation process was carried out by varying the nanocatalyst mass. The test results in mass optimization are shown in Figure 2.

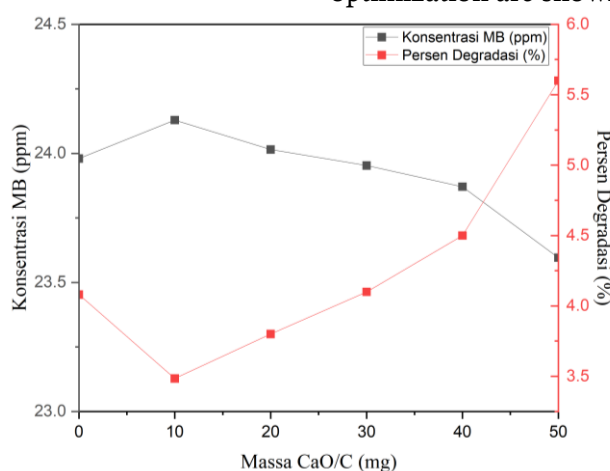


Figure 2. Variation of CaO/C mass on methylene blue concentration and percent degradation

The concentration of methylene blue decreased as the mass of the CaO/C nanocatalyst increased. Percentage decrease in methylene blue concentration within 30 minutes with mass variations of 0, 10, 20, 30, 40, and 50, respectively 4.08, 3.48, 3.8, 4.1, 4.5, and 5.6 %, and it is known that the optimum mass of CaO/C nanocatalyst is 50 mg. The test results in this study show that the greater the mass of CaO/C, the decrease in the percentage of methylene blue concentration. This is because the greater the mass of the catalyst, the more contact there is between the catalyst and methylene blue (Putri, 2022). Similar results were also reported by Zilfa et al. (2021) and Priatmoko et al. (2021) that the percent degradation of tartrazine and methylene blue increased with the addition of ZnO/zeolite and TiO₂-zeolite catalysts.

The test results also show that the concentration of methylene blue can be degraded without a catalyst with a concentration reduction percentage of 4.08%. This can be caused by UV rays, which are able to degrade methylene blue. Widihati et al. reported that the methylene blue photodegradation process with an initial concentration of 20 ppm without a catalyst resulted in a degradation percentage of 22.35% (Widihati et al., 2017). A similar thing was also

reported by Diantriani et al. (Diantriani et al., 2006) that methylene blue with an initial concentration of 50 ppm degraded using UV light without a catalyst produced a degradation percentage of 0.97%. However, the presence of a catalyst is needed to accelerate and increase the effectiveness of the reduction (Diantriani et al., 2006).

Photodegradation tests using UV light showed a better reduction in methylene blue concentration compared to those without UV light (Figure 3). Kakame & Wuntu (2019) reported that the light reaction for photodegradation of methylene blue with Ag-TiK showed better results than the dark reaction (Kakame & Wuntu, 2019). A similar thing was also reported (Ghofur et al. (2014) in the photodegradation process of methylene blue with TiO₂-Zeolite. UV light functions to stimulate electron jumps on the catalyst so that it can increase the effectiveness of the material (Adnan et al., 2022). Reducing the concentration of methylene blue, the dark reaction reached 13.8% at 120 minutes, which occurred due to the adsorption process by the CaO/C material (Ingrid et al., 2015). triggers the formation of OH and O²⁻ radicals (Ramamoorthy et al., 2020). These radicals will break certain chains of methylene blue, resulting in smaller molecular pieces, until complete

degradation produces H_2O and CO_2 , which are safe for the environment according to what has been proposed by (Yu et al., 2021), shown in Figure 4.

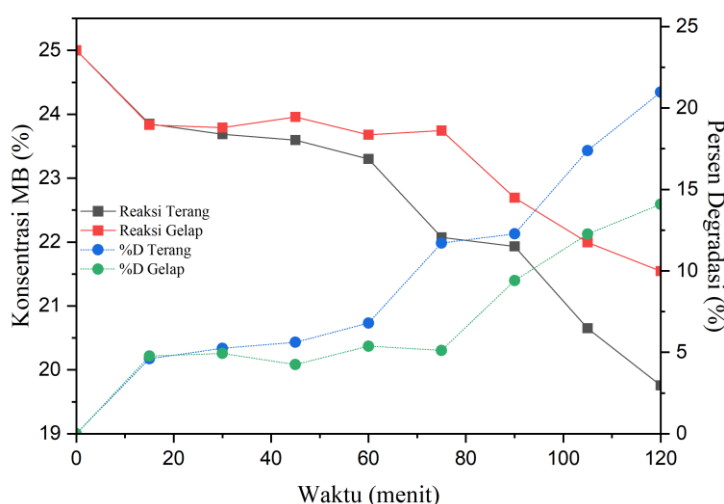


Figure 3. Comparison of the effectiveness of light and dark reactions

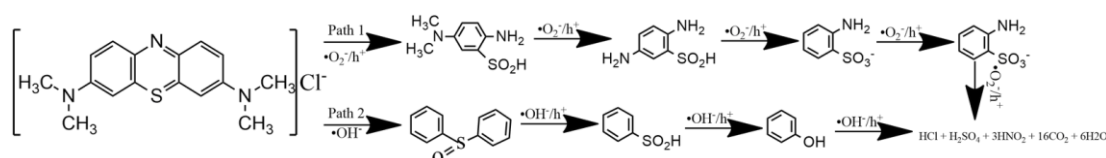


Figure 4. Mechanism of Methylene Blue Photodegradation Reaction (Yu et al., 2021)

The variation in exposure time (light reaction) shown in Figure 3 shows that the longer the exposure time, the percentage decrease in methylene blue concentration increases. The percentage reduction in methylene blue reached 20.968% at 120 minutes. This phenomenon occurs because the longer the exposure time, the more contact between the catalyst and the dye, and the more methylene blue molecules are absorbed (Risma, 2022). Previous research reported by Kakame & Wuntu (2019) on the methylene blue photodegradation process with a calcined fishbone Ag catalyst stated that the longer the radiation time, the more effective it is. The same thing was also reported by Dini & Wardhani (2014) and Pangajow et al. (2019) successively in the methylene blue photodegradation process with ZnO-Zeolite catalysts and $\text{Ag}_3\text{PO}_4/\text{Ag}/\text{HAp}$ Red Snapper (*Lutjanus* sp.) Bones. Analysis of degradation kinetics was carried out by processing data on variations in the time of methylene blue degradation to suit the 1st and 2nd order kinetic models. The analysis showed that the kinetics of methylene blue degradation by CaO/C tended to follow the 1st-

order kinetic model with a k value of 0.0018 min^{-1} (ppm/minute).

CONCLUSION

The CaO/C that was successfully synthesized was characterized using FT-IR, which showed the combined spectra of CaO and carbon from jengkol shells. CaO/C shows an optimum mass of 50 g in a 25 ppm methylene blue solution with a percentage reduction in methylene blue concentration of 5.2%. The optimum time for CaO/C was achieved at 120 minutes with a percentage reduction in methylene blue concentration of 20.968%. The kinetics of the CaO/C reaction follows a 1st-order kinetic model with a constant value of 0.0018 min^{-1} .

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