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Photodegradation of Crystal Violet Dyes Using Fe_2O_3/CuO with the Addition of MEA Additive

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Abstract. Synthetic dyes, including Crystal Violet (CV), are widely used in the textile and chemical industries and are increasingly released into the environment due to their toxic, mutagenic, and non-biodegradable nature. The purpose of this work is to examine the photocatalytic degradation of CV utilising Fe_2O_3/CuO heterojunction with monoethanolamine (MEA) as an additive. To maximise its optical and catalytic capabilities, the Fe_2O_3/CuO photocatalyst was synthesised using different CuO compositions and MEA volumes. Using UV-Visible Diffuse Reflectance Spectroscopy (UV-DRS) and the Kubelka-Munk technique, the band gap energy was determined. According to the findings, the incorporation of CuO did not show a pronounced effect, but it may contribute to the photocatalytic system through interfacial interactions and improved charge transfer within the Fe_2O_3/CuO structure. The lowest band gap value and optimal composition were found at 20% CuO, which was further reduced by adding 2 mL MEA. Tests of photocatalytic activity were conducted for 120 minutes when exposed to UV and sunshine. The results showed that CV had a high deterioration efficiency, reaching about 53.87% when exposed to sunshine. The creation of a Fe_2O_3/CuO heterojunction, which enhances charge separation and decreases electron-hole recombination, is responsible for the improved performance, whereas MEA enhances particle distribution and increases surface area. These results suggest that Fe_2O_3/CuO with MEA is a promising, economical, and eco-friendly photocatalyst for the treatment of wastewater contaminated with dyes.

Keywords: *photocatalysis, Fe_2O_3/CuO, monoethanolamine, Crystal Violet, band gap, wastewater treatment*

Abstrak. Zat warna sintetis, termasuk Kristal Violet (CV), banyak digunakan dalam industri tekstil dan kimia dan semakin banyak dilepaskan ke lingkungan karena sifatnya yang beracun, mutagenik, dan tidak dapat terurai secara hayati. Tujuan dari penelitian ini adalah untuk menguji degradasi fotokatalitik CV menggunakan heterojunction Fe_2O_3/CuO dengan monoetanolamina (MEA) sebagai aditif. Untuk memaksimalkan kemampuan optik dan katalitiknya, fotokatalis Fe_2O_3/CuO disintesis menggunakan komposisi CuO dan volume MEA yang berbeda. Dengan menggunakan *UV-Visible Diffuse Reflectance Spectroscopy* (UV-DRS) dan teknik Kubelka-Munk, energi celah pita ditentukan. Berdasarkan temuan tersebut, penambahan CuO tidak menunjukkan efek yang signifikan, tetapi dapat berkontribusi pada sistem fotokatalitik melalui interaksi antarmuka dan peningkatan transfer muatan dalam struktur Fe_2O_3/CuO. Nilai celah pita terendah dan komposisi optimal ditemukan pada 20% CuO, yang selanjutnya dikurangi dengan menambahkan 2 mL MEA. Uji aktivitas fotokatalitik dilakukan selama 120 menit saat terpapar sinar UV dan sinar matahari. Hasil penelitian menunjukkan bahwa CV memiliki efisiensi degradasi yang tinggi, mencapai sekitar 53,87% ketika terpapar sinar matahari. Pembentukan heterojunction Fe_2O_3/CuO, yang meningkatkan pemisahan muatan dan mengurangi rekombinasi elektron-lubang, bertanggung jawab atas peningkatan kinerja, sedangkan MEA meningkatkan distribusi partikel dan meningkatkan luas permukaan. Hasil ini menunjukkan bahwa Fe_2O_3/CuO dengan MEA merupakan fotokatalis yang menjanjikan, ekonomis, dan ramah lingkungan untuk pengolahan air limbah yang terkontaminasi zat warna.

Kata kunci: fotokatalisis, Fe_2O_3/CuO, monoethanolamin (MEA), Crystal Violet, energi celah pita, pengolahan limbah

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INTRODUCTION

The textile, printing, paper, and medical industries all employ Crystal Violet (CV), a cationic dye that is a member of the triphenylmethane dye group. This molecule is difficult to break down naturally due to its complex aromatic structure with dimethylamino groups, which contribute to its great chemical stability and photostability (Mancuso et al., 2023). The CV structure's large π-conjugated system allows visible light at wavelengths approximately 588–590 nm to be absorbed through π→π* electronic transitions, giving it its distinctive violet hue (Figure 1). The dimethylamino groups not only have remarkable optical characteristics but also function as electron donors that affect the HOMO–LUMO orbital energy and reduce the molecular band gap. Because of its persistent aromatic composition, CV is resistant to traditional wastewater treatment methods and tends to endure in the environment.

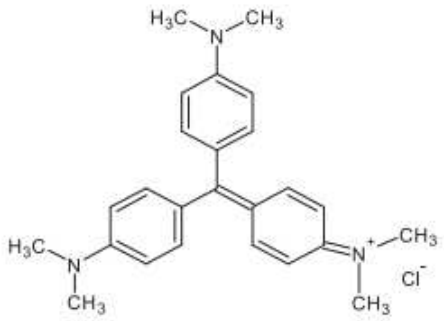

Figure 1. Chemical structure of Crystal Violet (Cheruiyot et al., 2019).

Because CV is toxic, non-biodegradable, and possibly mutagenic and carcinogenic to living things, its presence in aquatic settings has raised significant environmental concerns (Ahamad & Nasar, 2023). According to (Shamsudin et al., 2024), an estimated 10–20% of the world's total CV output is wasted during the dyeing process and dumped into water bodies, causing ecological imbalance and environmental damage. CV contamination can hinder aquatic species' ability to photosynthesise, decrease light penetration in water, and encourage the bioaccumulation of hazardous substances in the food chain (Sen et al., 2024). Therefore, reducing the environmental impact of Crystal Violet requires the development of efficient and eco-friendly treatment techniques to break it down into simpler and less dangerous molecules.

As an n-type semiconductor with a small band gap of around 1.9–2.2 eV, iron oxide (Fe_2O_3) can absorb visible light efficiently and is widely investigated as a photocatalyst among metal oxides. The most thermodynamically stable of its polymorphs is haematite (α-Fe_2O_3), which is frequently employed in composite materials to improve photocatalytic efficiency. Fe_2O_3 is a potential material for environmental remediation and sustainable energy applications since it is readily available, inexpensive, environmentally friendly, and has good chemical stability.

Despite these benefits, Fe_2O_3's photocatalytic efficacy is frequently constrained by quick electron-hole recombination and low charge carrier mobility, which lowers its catalytic efficiency. Fe_2O_3 is often mixed with other semiconductors to create heterojunction structures that enhance charge separation and allow interfacial charge transfer in order to get around these restrictions. In order to increase photocatalytic activity and expand the possible use of Fe_2O_3-based materials in the breakdown of organic contaminants in wastewater, these approaches have been extensively investigated (Zhang et al., 2023).

The inorganic compound copper(II) oxide (CuO), also referred to as cupric oxide, has garnered a lot of interest in photocatalytic applications because of its semiconductor characteristics. CuO is a p-type semiconductor that promotes redox reactions during photocatalytic processes and allows for effective absorption of visible light due to its comparatively narrow band gap of about 1.2 eV (Brasil, 2024). Because of their similar electrical structures and charge carrier properties, CuO and Fe_2O_3 are frequently coupled in photocatalytic systems. CuO has good electrical conductivity and redox behaviour, while Fe_2O_3 is an n-type semiconductor with strong oxidation capabilities and high chemical stability (Marquez et al., 2025). When these two semiconductors are integrated, they create a heterojunction interface that produces an internal electric field that improves photogenerated electron–hole pair separation and inhibits charge recombination (Belew & Assege, 2025).

CuO improves photon-to-chemical energy conversion efficiency by enhancing visible light absorption, which Fe_2O_3 alone is less capable of utilizing. Energy band theory states that CuO's lower conduction band level than Fe_2O_3 allows electrons to go from Fe_2O_3 to CuO while holes stay in Fe_2O_3. This leads to efficient charge separation and increased photocatalytic activity (Akinnawo, 2025). The Cu^{2+}/Cu^{+} redox pair in CuO enhances photocatalytic performance and interfacial electron transfer, indicating that the Fe_2O_3/CuO heterostructure provides synergistic effects for dye photodegradation in terms of stability, charge transfer efficiency, and visible-light responsiveness.

Through electron-donor interactions between its N and O atoms and metal centers, MEA can change the electrical characteristics of oxide material surfaces, improving particle dispersion and charge transfer efficiency. Additionally, polar-bound amine groups can be formed by MEA binding to oxide surfaces, increasing the surface's attraction for specific organic molecules or metal ions. According to quantum chemistry, MEA has a high electron density on nitrogen atoms (μ = 1.10 D, ΔE HOMO-LUMO = 4.20 eV), which makes it an efficient electron donor when coordination complexes with metal cations are formed. Because of these properties, MEA can be used as an organic dopant or structural modification to stabilise the catalytic surface and affect local charge distribution (Matyakubova et al., 2024).

MATERIAL AND METHODS

Materials

All chemicals used in this study were of analytical grade and used without further purification. Iron(III) chloride hexahydrate ($FeCl_3 \cdot 6H_2O$, ≥99%) and copper(II) chloride dihydrate ($CuCl_2 \cdot 2H_2O$, ≥99%) were purchased from Merck (Germany). Methanol (p.a, ≥99.8%) was obtained from Merck. Monoethanolamine (MEA, ≥99%) was supplied by Sigma-Aldrich. Crystal Violet dye (C.I. 42555) was also obtained from Sigma-Aldrich. Aquadest (distilled water) was used as the solvent throughout the experiment.

Instrumentation

The equipment used in this research included common laboratory glassware such as beakers, measuring cylinders, evaporating dishes, watch glasses, volumetric pipettes, a mortar and pestle, and a desiccator. The synthesis process was assisted using a

magnetic stirrer (Model C-MAG HS 7, IKA, Germany) with a stirrer bar, and a furnace (Type Nabertherm L3/11, Nabertherm, Germany) for calcination.

The optical properties of the samples were analyzed using a UV-Visible spectrophotometer (Shimadzu UV-1800, Shimadzu, Japan). The band gap energy was determined using a UV-Visible Diffuse Reflectance Spectrophotometer (UV-Vis DRS, Shimadzu UV-2600). Crystal structure and phase identification were characterized using X-Ray Diffraction (XRD, PANalytical X'Pert PRO, Malvern Panalytical, Netherlands). Morphological analysis was performed using Scanning Electron Microscopy (SEM, JEOL JSM-6510LV, JEOL, Japan). The photocatalytic activity test was carried out under UV irradiation using a UV lamp (Philips UV Lamp, Philips, Netherlands).

Procedure

Synthesis of Fe_2O_3 material

50 mL of methanol, covered with plastic wrap, was used to dissolve the $FeCl_3 \cdot 6H_2O$ precursor at a concentration of 0.5 M. The mixture was then homogenized for 40 minutes using a magnetic stirrer. After that, the solution was sonicated for 30 minutes at 45 W to produce a homogeneous solution (sol), which was then allowed to stabilize for a period of 24 hours. Additionally, the sample was dried for approximately one hour at 110 °C in an oven. To extract the Fe, the resulting gel was calcined in a furnace for approximately three hours at 400 °C. The sample was stored in a desiccator; after cooling, it was ground using a mortar and pestle so that the sample could be described.

Synthesis of CuO-doped Fe_2O_3 material

50 mL of methanol, covered with plastic wrap, was used to dissolve $FeCl_3 \cdot 6H_2O$ precursor at a concentration of 0.5 M and $CuCl_2 \cdot 2H_2O$ precursor at concentrations of 5%, 10%, 15%, 20%, and 25%. The mixture was then homogenized for approximately 1 hour and 30 minutes using a magnetic stirrer. Afterward, the solution was sonicated for 30 minutes at 45 W to create a homogeneous solution (sol), which was then allowed to stabilize for a 24-hour period. Additionally, the samples were dried for approximately one hour at 110 °C in an oven. To obtain Fe/CuO, the gel was calcined in a furnace for approximately three hours at 400 °C. To characterize the samples, they were stored in a desiccator and then ground with a mortar and pestle after cooling.

Synthesis of Fe_2O_3/CuO material with the addition of MEA

The $CuCl_2 \cdot 2H_2O$ precursor was dissolved at various concentrations, namely 5%, 10%, 15%, 20%, and 25%, while the $FeCl_3 \cdot 6H_2O$ precursor was dissolved at a concentration of 0.5 M. The solutions were dissolved in 50 mL of methanol, then tightly covered with plastic wrap to prevent evaporation. For forty minutes, a magnetic stirrer was used to homogenize the mixture. Next, 1 mL, 2 mL, and 3 mL of analytical-grade monoethanolamine (MEA) were added to each solution, and the mixture was stirred again for approximately 1.5 hours. To ensure the homogeneity of the sol, the solution was sonicated for 30 minutes at 45 W.

Photocatalytic activity of CuO-doped Fe_2O_3

Dissolve 0.1 grams of Crystal Violet in 100 mL of distilled water in a 100 mL volumetric flask, then pipette 10 mL of the Crystal Violet stock solution and dilute it in a 1000 mL volumetric flask. Add 0.1 grams of photocatalyst to 100 mL of the Crystal Violet solution. Before UV irradiation, the solution was

stirred in the dark for 30 minutes to achieve adsorption and desorption equilibrium. Next, the photolysis irradiation times were set to 0, 30, 60, 90, 120, 150, and 180 minutes. 5 mL of solution was taken at each interval and then centrifuged for 10 minutes to remove particles. To measure the absorbance of the supernatant solution, a UV-Vis spectrophotometer with a wavelength of 553 nm was used.

RESULT AND DISCUSSION

Band Gap Energy Analysis

UV-DRS was used to examine the optical characteristics of the Fe_2O_3/CuO photocatalysts in the 185–1100 nm instrument wavelength range (Lakhera et al., 2018). The Kubelka–Munk function was utilised to calculate the band gap energy : (Eg):

$$F(R) = \frac{(1-R)^2}{2R}$$

and calculated using:

$$E_g = h\nu = \frac{hc}{\lambda}$$

The band gap values were obtained from the plot of

$$h\nu\ (eV)\ versus\ (F(R)h\nu)^{1/2}$$

The calculated band gap values for Fe_2O_3/CuO with different CuO compositions are presented in Table 1.

Table 1. Band gap energy of Fe_2O_3/CuO photocatalysts

No.	Sample	Band Gap (eV)
1	Fe_2O_3 (pure)	1.49
2	Fe_2O_3/CuO 5%	1.6
3	Fe_2O_3/CuO 10%	1.52
4	Fe_2O_3/CuO 15%	1.58
5	Fe_2O_3/CuO 20%	1.5
6	Fe_2O_3/CuO 25%	1.55

Table 1 illustrates that when the CuO content increases, the band gap energy drops. The ideal composition was found at 20% CuO, which had the lowest band gap value (1.5 eV). Improved photocatalytic capability and increased absorption of visible light are suggested by this drop.

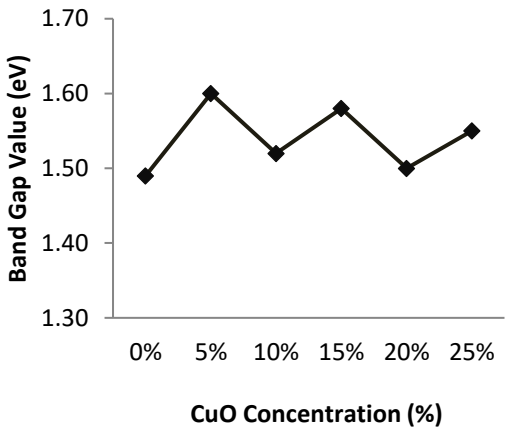

Figure 2. Band gap energy on the effect of CuO doping concentration

The formation of a heterojunction between Fe_2O_3 and CuO is responsible for the decrease in band gap energy (Figure 2). This heterojunction introduces new energy levels and promotes charge transfer, thereby reducing electron–hole recombination (Chandra & Darmayanti, 2019).

Effect of MEA Additive on Band Gap Energy

Monoethanolamine (MEA) was added in amounts of 1 mL, 2 mL, and 3 mL to further improve the optical characteristics. The results are displayed in Table 2.

Table 2. Band gap energy with MEA addition

No.	Sample	Band Gap (eV)
1	Fe_2O_3/CuO + 1 mL MEA	1.83
2	Fe_2O_3/CuO + 2 mL MEA	1.81
3	Fe_2O_3/CuO + 3 mL MEA	1.89

Table 2 shows that the addition of 2 mL MEA results in the lowest band gap value (1.81 eV), indicating the optimal condition.

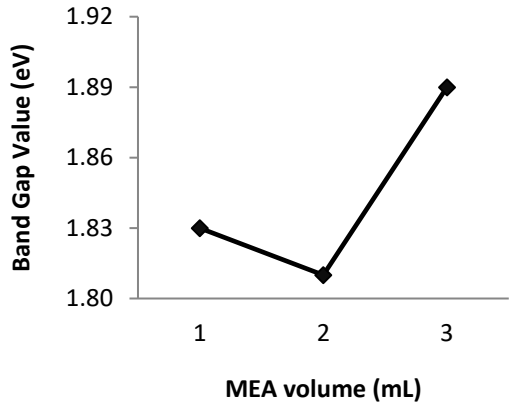

Figure 3. Effect of MEA on band gap energy

During synthesis, MEA functions as a complexing agent to stabilise Fe^{3+} and Cu^{2+} ions, resulting in a more uniform distribution of CuO inside the Fe_2O_3 matrix (Figure 3). MEA also enhances surface area, decreases particle size, and regulates crystal growth—all of which are advantageous for photocatalytic activity (Upadhyay et al., 2025).

Photocatalytic Activity

The degradation of Crystal Violet dye under UV and sunlight irradiation was used to assess the photocatalytic activity. Using a UV-Vis spectrophotometer, the maximum absorption wavelength (λ_{max}) of Crystal Violet was found to be roughly 590 nm.

The ideal sample (Fe_2O_3/CuO 20% with 2 mL MEA) was used in the photocatalytic studies, and the irradiation periods were 30, 60, 90, and 120 minutes. The degradation results are illustrated in Figure 4**.**

Photocatalytic degradation of Crystal Violet is influenced by both irradiation time and light source. Under UV irradiation, the efficiency remained low, reaching only 13.95% after 60 minutes. In contrast, exposure to sunlight resulted in a significantly higher degradation of about 53.87% within 30 minutes.

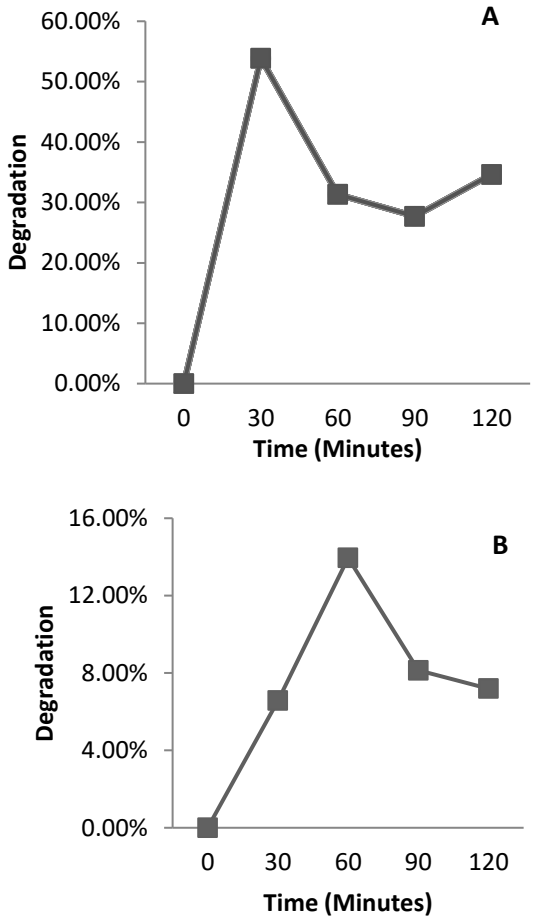

Figure 4. Photodegradation efficiency of Crystal Violet using Fe_2O_3/CuO (20%) with 2 mL MEA under (A) sunlight and (B) UV irradiation

The degradation efficiency often rises with radiation time, as seen in Figure 1, suggesting the ongoing production of reactive species such as hydroxyl radicals (•OH) and superoxide radicals (O_2•$^-$), which are essential to the degradation process. However, the creation of intermediate compounds that still absorb close to the characteristic wavelength of Crystal Violet may cause variations in degradation efficiency at intermediate irradiation durations.

Discussion

The results show that the photocatalytic performance is greatly improved by the creation of Fe_2O_3/CuO heterostructures and the addition of MEA. Improved absorption in the visible range is indicated by the band gap decreasing from 1.49 eV (pure Fe_2O_3) to 1.6 eV (Fe_2O_3/CuO) and then to 1.81 eV with MEA addition.

A number of synergistic effects can be used to explain the enhancing mechanism. Recombination of photogenerated electron-hole pairs is suppressed by the creation of a Fe_2O_3/CuO heterojunction, which is essential for increasing charge separation efficiency. CuO serves as an electron acceptor in this system, promoting electron transport and lowering recombination rates even more. The presence of MEA during the synthesis process improves structural homogeneity, leading to a more uniform particle dispersion. Particle size reduction also increases surface area and the number of active sites, both of which improve photocatalytic effectiveness (Ashu Abey et al., 2025).

Improved utilization of the light spectrum is reflected in the higher degradation efficiency under sunlight compared to UV irradiation. The broader photon energy range provided by sunlight enhances the generation of reactive radicals. During the photocatalytic process, photon absorption excites electrons from the valence band to the conduction band, producing electron–hole pairs. These charge carriers subsequently degrade Crystal Violet into simpler and less harmful compounds through reactions with water and oxygen, forming reactive species (•OH and $O_2^{\bullet -}$). CuO and MEA act synergistically to improve the optical properties and photocatalytic performance of Fe_2O_3-based materials, indicating strong potential for wastewater treatment applications (Gupta & Mandavgane, 2024).

CuO and MEA work in concert to improve the optical characteristics and photocatalytic activity of Fe_2O_3-based materials, suggesting that they have a great deal of promise for use in wastewater treatment (Irsyad & Sanjaya, 2025).

CONCLUSION

The research's results show that adding MEA to the Fe_2O_3/CuO photocatalyst results in optimum optical and photocatalytic characteristics for the breakdown of Crystal Violet dye. CuO was successfully incorporated into Fe_2O_3 to lower the band gap energy; 20% CuO was found to be the ideal composition. Additionally, the addition of 2 mL MEA enhanced the photocatalyst's structural properties and reduced the band gap, improving light absorption in the visible spectrum. Tests of photocatalytic activity showed that the best sample had a high rate of Crystal Violet degradation, reaching about 53.87% in 120 minutes when exposed to sunshine.

The development of a Fe_2O_3/CuO heterojunction, which improves charge separation and inhibits electron-hole recombination, as well as the function of MEA in regulating particle size and boosting surface area, is responsible for the enhanced performance.

For the treatment of dye-contaminated wastewater, especially when exposed to sun radiation, the Fe_2O_3/CuO photocatalyst with MEA addition can be regarded as a promising, economical, and eco-friendly material.

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