Original Research Article

$CuO-CeO_2$ Photocatalysis for the Degradation of Remazol Textile Dyes

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Abstract

Water pollution, particularly from textile industrial waste, has become an increasingly significant environmental issue. Textile waste, especially synthetic dyes, contributes substantially to water pollution due to its complex structure and low biodegradability. Dyes such as Remazol Brilliant Red and Remazol Turquoise Blue, commonly used in the textile industry, are stable, toxic, and difficult to degrade biologically. One promising approach to address this issue is photocatalysis, using photocatalysts such as CuO and CeO_2, which possess high degradation capabilities for organic contaminants. This study explores the effectiveness of $CuO-CeO_2$-based photocatalysts in degrading Remazol Turquoise Blue and Remazol Brilliant Red dyes. The photocatalysts were synthesised with varying $CuO-CeO_2$ concentrations, and degradation tests were conducted using UV light to observe dye degradation. The results show that $CuO-CeO_2$ photocatalysts at a concentration of 25% achieved the highest degradation efficiency, with nearly 40% removal of Remazol Turquoise Blue and 28% removal of Remazol Brilliant Red after 120 minutes. Increasing the photocatalyst concentration indicates an increase in active sites, accelerating the degradation process. However, there is a limitation at very high concentrations due to the potential for catalyst particle agglomeration.

Keywords:

Photocatalysis, $CuO-CeO_2$, textile dye degradation, Remazol Turquoise Blue, Remazol Brilliant Red.

1. Introduction

Environmental problems, especially water pollution, have increased significantly in recent years. Lack of effective waste management and strict supervision of industrial waste disposal cause water pollution. Textile industry waste is the second largest contributor to waste in the world after plastic waste. (Candrastuti 2022)It is estimated that global dye production reaches about one million tons annually, with more than 15% of it going into the environment as pollutants in the form of industrial waste (Saeed et al., 2022). Textile industry waste has a strong colour characteristic that obstructs the entry of light into the water, and it is toxic and carcinogenic when consumed. (Kumari et al. 2023). Dyes can be grouped by their ionic properties into cationic, anionic, and non-ionic. In addition, dyes are also classified according to their functional groups, such as azo, indigo, phthalocyanine, anthraquinone, sulfur, and others.(Gondo and Mbaiwa 2022).

Some dyes often used in the textile industry are the anionic dyes Remazol Brilliant Red and Remazol Turquoise Blue G-133. Both dyes provide bright colours to textiles, but due to their complex structure, they are reactive and difficult to degrade biologically (Purnawan et al., 2021). Textile dyes result in various environmental problems in aquatic ecosystems due to their high stability, low biodegradability, and increased aromatisation (Aziz et al., 2023). Based on these problems, various biological and physicochemical approaches with different levels of effectiveness have been applied in textile dye effluent treatment, including membrane filtration, adsorption, and electrocoagulation techniques. Of these water pollutant mitigation and treatment methods, photocatalysis is superior in degrading organic

contaminants (Yusuff 2020). Photocatalysis involves oxidation reactions using specific photocatalysts such as titanium dioxide (TiO_2), zinc dioxide (ZnO), cadmium dioxide (CdO), copper oxide (CuO), stannic oxide (SnO_2), or cerium dioxide (CeO). $_2$(Chairungsri et al. 2022). Some photocatalysts have been used to degrade Methylene Blue, achieving 96.2% degradation efficiency within 180 min. (CHEN et al. 2024). Remazol red ultra RGB can be degraded using SrFe12O19-Fe3O4 photocatalyst, reaching 100% within 1 hour (Paulista et al. 2024). (Paulista et al. 2024a). Photocatalysts using Ag-impregnated ZnO can degrade Remazol brilliant violet 5R 95.7% within one hour (Aziz et al., 2023). (Aziz et al., 2023). The degradation of Remazol turquoise blue reached 99% using PM with 22 mM H2O2 (Sakarkar 2020). (Sakarkar 2020).

Various types of photocatalysts that have been developed show significant potential in textile waste treatment. One of them is a Copper oxide (CuO)- based photocatalyst combined with Cerium dioxide (CeO_2). Copper oxide (CuO), as a p-type semiconductor with a narrow band gap of 1.2 ~ 1.7 eV, which is applied to lithium-ion batteries, field emission displays, photodetectors, sensors, and supercapacitors, can be made p-n connections with CeO2. (Li, Liu, Huang, et al. 2019). Cerium dioxide (CeO_2) has been widely researched due to its photocatalytic solid redox ability, good chemical stability, and high oxygen storage ability.4 However, the rapid recombination of electron-hole pairs reduces its catalytic activity. Considering CeO2 as an n-type semiconductor with a wide bandgap, combining it with narrow bandgap semiconductors to fabricate CeO2-based p-n heterojunctions may be a good strategy. (Chen et al. 2024).

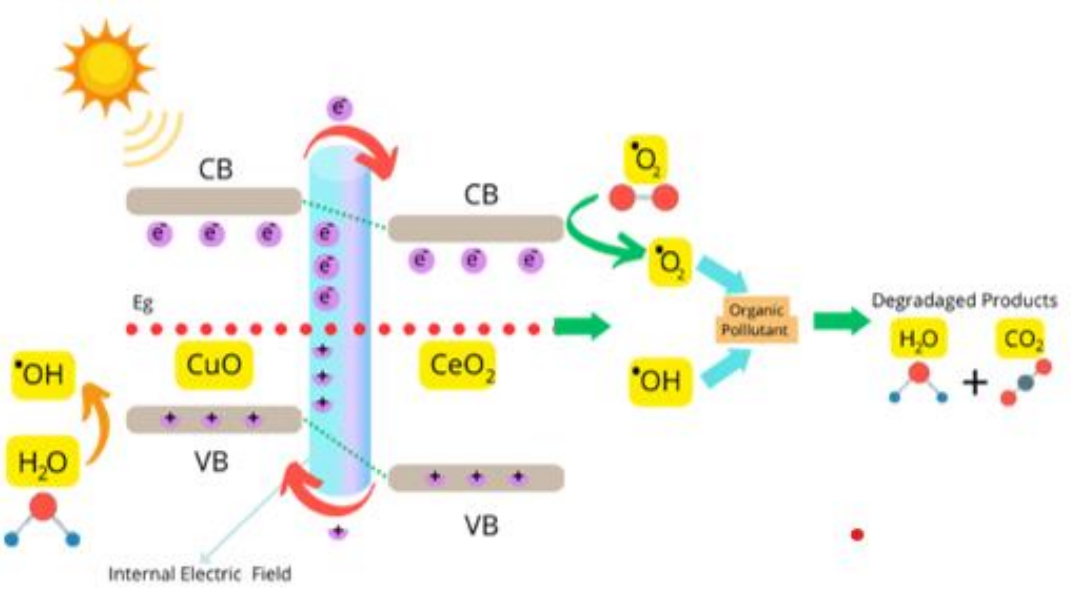


Figure 1 The photocatalytic mechanism through CuO@CeO2 photocatalyst**.**

Figure 1 shows photocatalytic degradation, for example, organic pollutants by CuO-CeO_2 incoming light creates an internal electric field that promotes the movement of electrons and holes on CuO and CeO_2.

$CuO\text{-}CeO_2 + h\nu \rightarrow e^- (VB) + h^+ (CB)$ .....(1)

$e^- + O_2 \rightarrow \cdot O_2^-$ .....(2)

$h^+ + H_2O \rightarrow \cdot OH + H^+$ .....(3)

Dye + ·OH + O2·· Degradation products .....(4)

The electrons on CuO interact with oxygen to produce superoxide radicals (O_2^-), while CeO_2 supports the formation of hydroxyl radicals (0H·) from water. This combination accelerates the degradation of organic pollutants to water and carbon dioxide, increasing photocatalytic efficiency. (Kusmierek 2020). The development of heterogeneous catalysts based on Cu with high stability, easy

recovery, and efficient reactivity, such as MnO_2, graphite, and ZSM5, has enhanced the catalytic activity and stability of CuO(Zhao, et al. 2019). The unique properties of Cerium Oxide (CeO_2), such as abundant oxygen vacancies and strong interactions with supporting metals, have been shown to promote CO_2 conversion reactions, making CeO_2 a crucial material for large-scale CO_2 utilization (Graciani, 2014). ith CeO_2 as the promoter, the interaction between raw material molecules (CO_2 and H_2O) is enhanced on the photocatalyst, accelerating the production of active intermediates (Nolan, 2018).

This study explores the effectiveness of CuO-CeO_2-based photocatalysts in degrading textile dyes, specifically Remazol Turquoise Blue and Remazol Brilliant Red, which are known to have complex structures and low levels of biodegradability. It is expected that the CuO-CeO_2 photocatalyst can be a sustainable solution in textile effluent treatment through a superior photocatalytic mechanism, as well as significantly contribute to reducing the impact of water pollution in aquatic environments.

2. Methods

2. Material

The ingredients are as follows: copper sulfate pentahydrate ($CuSO_4 \cdot 5H_20$) sodium hydroxide (NaOH), sodium citrate, cerium dioxide, and Remazol Turquoise Blue and Remazol Brilliant Red dyes.

2.2 Synthesis of CuO-CeO2

In this research, the Cu^{2+} ion composition in the synthesis of CuO@CeO_2 composite photocatalysts was quantified and represented as the percentage of Cu moles relative to the total moles of Cu and CeO_2. The composition percentage ranged from 1% to 25%, aiming to investigate the impact of increasing Cu concentration on the photocatalyst's performance. Initially, the masses of CeO_2 and $CuSO_4 \cdot 5H_2O$ were calculated to correspond to the target mole percentages of Cu. For molar ratios of 1%, 3%, 5%, 10%, 15%, and 25%, the respective masses of CeO_2 were 6.82 g, 6.68 g, 6.54 g, 6.20 g, 5.85 g, and 5.16 g, while the masses of $CuSO_4 \cdot 5H_2O$ added were 0.10 g, 0.30 g, 0.50 g, 1.00 g, 1.50 g, and 2.50 g. The NaOH mass used in each ratio was adjusted to exceed twice the number of moles of Cu, ensuring optimal reaction conditions.

The synthesis began by preparing a 500 ml beaker filled with 400 ml of deionised water. The mixture was stirred vigorously using a magnetic stirrer for one hour to ensure even solubility. After that, the mixture was heated to a temperature of 70°C. At this temperature, 1 gram of sodium citrate was added as a stabilising agent, along with the appropriate amount of $CuSO_4 \cdot 5H_20$ for each specified percentage. The mixture was then stirred intensively at a constant temperature of 70°C for two hours to ensure complexation between Cu^{2+} and the stabilising agent. Next, 50 ml of NaOH solution with excess concentration was gradually added to the mixture. The gradual addition of NaOH aims to control the pH level of the mixture to be at an optimal condition for the formation of CuO in the CeO_2 matrix. After the NaOH addition was complete, the mixture was transferred into a Teflon-lined autoclave, which was then sealed and heated in an oven at 90°C for four hours.

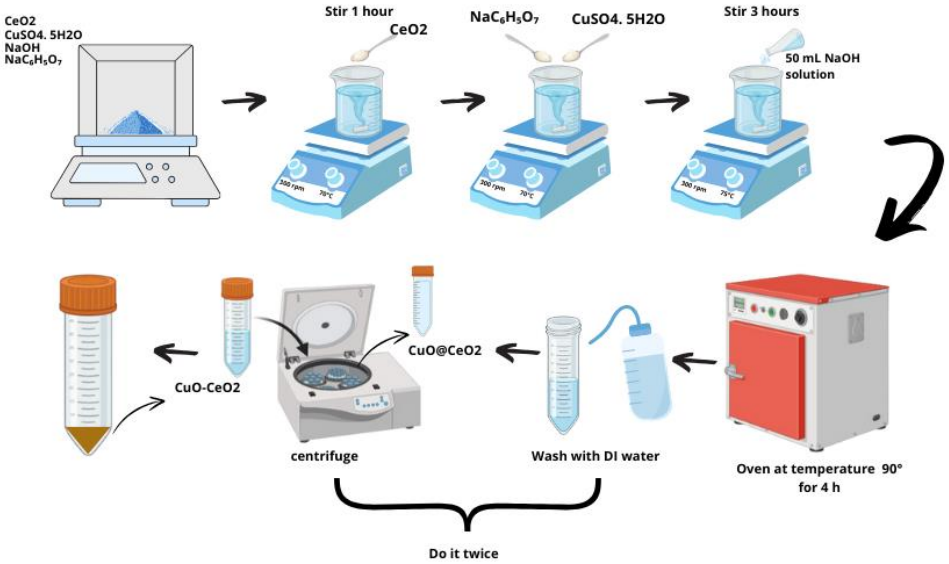


Figure 2. Preparation of CuO-CeO_2 Composite

This autoclaving stage aims to facilitate the hydrothermal reaction, which results in CuO crystals evenly distributed on the CeO_2 surface, thus forming a homogeneous composite. The final process involves filtering the CuO@CeO_2 solid product using centrifuge to ensure complete separation from the residual solution. The filtered product is washed repeatedly with water and ethanol to remove any impurities or unreacted reagents. After washing, the composite product was oven-dried at 70°C for 24 hours to evaporate the remaining solvent and obtain the CuO@CeO_2 photocatalyst in solid form ready for characterisation and photocatalytic activity testing.

2.2 Photodegradation of Remazol Turquoise Blue and Remazol Brilliant Red

The photodegradation of Remazol Turquoise Blue and Remazol Brilliant Red was conducted using a solution prepared by dissolving 150 ml of each dye at a concentration of 20 mg/L. To this solution, 0.05 g of the CuO-CeO_2 composite photocatalyst was added. The photocatalytic reaction was performed under UV light exposure (8 W × 2) at room temperature and atmospheric pressure. The UV light source was positioned ten centimetres above the solution surface in the beaker to ensure uniform illumination across the solution.

During the process, the solution was stirred at a constant speed of 300 rpm to maintain homogeneity and promote optimal interaction between the photocatalyst and the dye molecules. The reaction proceeded for two hours, with periodic sampling at time intervals of 0, 5, 10, 15, 30, 60, 90, and 120 minutes. At each interval, 5.0 ml aliquots were extracted to measure the remaining concentrations of Remazol Turquoise Blue and Remazol Brilliant Red.

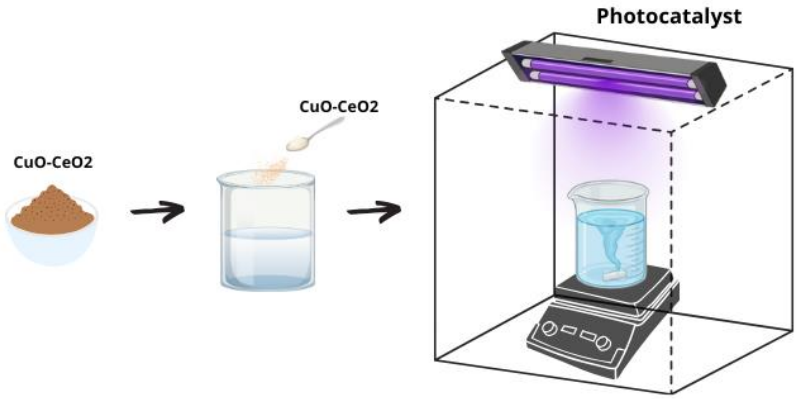


Figure 3 Photocatalys Test

After sampling, each sample was centrifuged for 10 minutes at 1500 rpm to separate the photocatalyst particles from the solution. The remaining concentration of Remazol Turquoise Blue and Remazol

Brilliant Red was then analyzed using UV-Vis spectroscopy at a wavelength of 630 nm. This analysis aims to assess the effectiveness of the photodegradation carried out and to evaluate the potential use of CuO-CeO_2 photocatalysts in the treatment of textile dye waste. The largest absorbance for Remazol Turquoise Blue and Remazol Brilliant Red was measured at wavelengths of 670 and 548 nm, respectively. To measure degradation, the maximum absorbance of Remazol Turquoise Blue and Remazol Brilliant Red was measured using a UV-Vis spectrophotometer.

The formula for calculating degradation efficiency is as follows:

$$\text{Degradation efficiency (\%)} = [(Co - C)/Co] \times 100 \quad \text{.....(1)}$$

Co = initial sample concentration

C = sample concentration after the degradation process

where Co and C are the initial intensity and the varying intensity of the dye molecules, respectively (Murugadoss et al. 2023).

3. Result and Discussion

a. Effect of CuO-CeO2 catalyst concentration on the degradation of remazol turquoise blue and remazol brilliant red

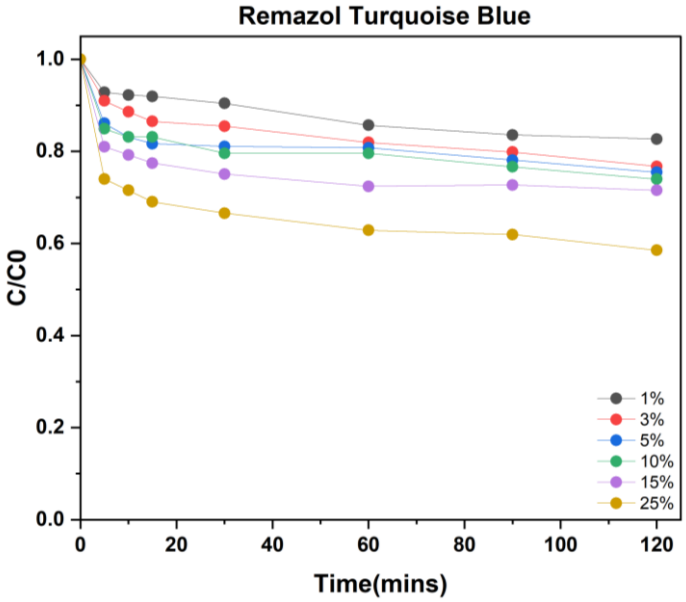


Figure 4 Effect of CuO-CeO2 catalyst concentration on the degradation of remazol turquoise blue

Figure 4 shows the degradation of Remazol Turquoise Blue (RTB) using the catalyst at various concentrations (1%, 3%, 5%, 10%, 15%, and 25%). The graph shows that RTB degradation occurs faster at higher catalyst concentrations, which is reflected by the sharper decrease in C/Co values, especially in the first 20 minutes, at 15% and 25% concentrations. The data in the table supports this result, where the decrease in C/Co at 25% concentration starts from 0.74 and ends at 0.59, showing the highest degradation efficiency compared to other concentrations. The effect of increasing catalyst concentration on degradation can be explained by the increasing number of active sites that accelerate photocatalytic reactions (Trung et al. 2024), allowing more intensive interactions between the RTB molecules and the catalyst surface. However, the rate of degradation slowed down after about 60 min, indicating the possible achievement of equilibrium conditions or the presence of degradation products that inhibit the continuation of the reaction on the catalyst surface (Murugadoss et al. 2024). (Murugadoss et al. 2023).. These overall results indicate that the optimal concentration for RTB degradation lies at 25% CuO-CeO2 concentration, which can maintain high efficiency considering economic and operational aspects. Copper-based catalysts are capable of converting CO_2 into C_2+ hydrocarbons through electrochemical

CO_2 reduction (Cheng et al. 2021). Among these, copper-based catalysts derived from oxides are particularly appealing due to their high selectivity towards C_2+ products at relatively low overpotentials. Previous studies have demonstrated that Cuδ+ surface species ($0 < \delta < 1$) and oxygen sites (O_2) in oxide-derived copper catalysts play a crucial role in activating CO_2 and enhancing the binding of CO intermediates (Zhang et al. 2020). However, achieving successful production of C_2+ hydrocarbons in photocatalytic systems remains a significant challenge due to the slow electron transfer and sluggish reaction kinetics of C–C bond formation. CO species tend to desorb from the photocatalyst surface before undergoing further conversion into C_2 products through subsequent multi-electron reduction processes (Albero, Peng, and García 2020). Therefore, it is crucial to accelerate the migration of photogenerated electrons and extend the residence time of CO at catalytic sites by rationally designing photocatalysts (Wang et al. 2021).

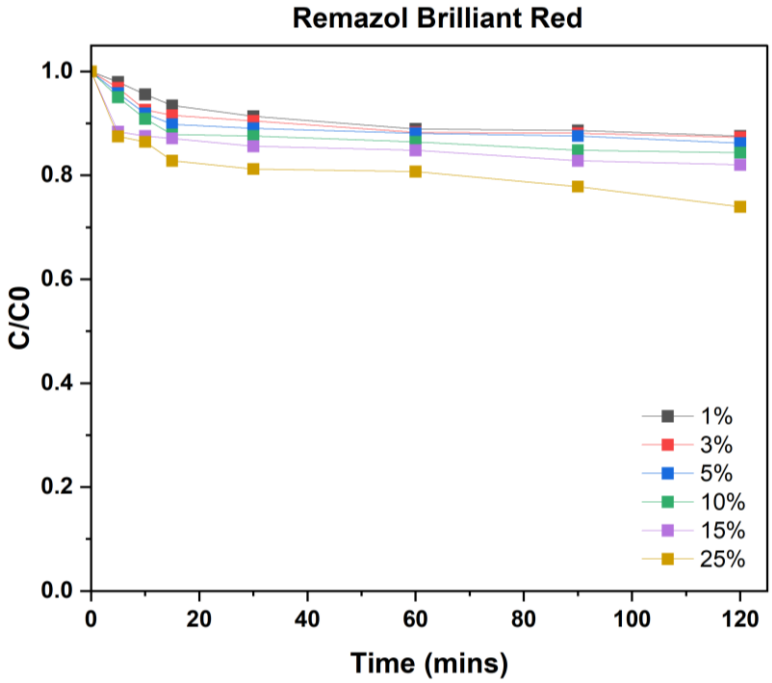


Figure 5 Effect of CuO-CeO2 catalyst concentration on the degradation of remazol brilliant red

Figure 5 shows the effect of CuO-CeO2 catalyst concentration on the degradation of Remazol Brilliant Red (RBR) at various catalyst concentrations (1%, 3%, 5%, 10%, 15%, and 25%) with reaction time. The graph shows that increasing the catalyst concentration has a positive impact on the degradation rate of RBR, as seen from the faster decrease in C/Co values at higher catalyst concentrations.

At 1% catalyst concentration, the C/Co value only decreased slightly from 0.980 to 0.876 over 120 minutes, indicating a relatively slow degradation rate. Higher catalyst concentrations, such as 15% and 25%, showed a more significant decrease in the C/Co value. At 25% concentration, the C/Co value decreased from 0.87 to 0.74 within the same period, indicating that an increase in catalyst concentration resulted in an increase in degradation efficiency. Dyes with simpler structures or weaker chemical bonds are generally considered more susceptible to degradation by free radicals generated during photocatalytic processes (Karlinda 2021). This increased susceptibility is attributed to the ease with which these dyes undergo bond cleavage, facilitated by the reactive species produced on the photocatalyst surface. Remazol Turquoise Blue is hypothesized to have a simpler chemical structure compared to Remazol Brilliant Red, resulting in a higher vulnerability to bond breaking and subsequent degradation. The structural

differences between these dyes are believed to play a significant role in determining their relative degradation efficiencies, as simpler molecular configurations typically require less energy to break chemical bonds (Jegatheesan 2020). As a result, the photocatalytic process may more effectively target dyes like Remazol Turquoise Blue, where the chemical bonds can be disrupted more readily, compared to dyes with more complex and robust molecular frameworks.

b. Effect of CuO-CeO2 catalyst concentration on removal remazol turquoise blue and remazol brilliant red

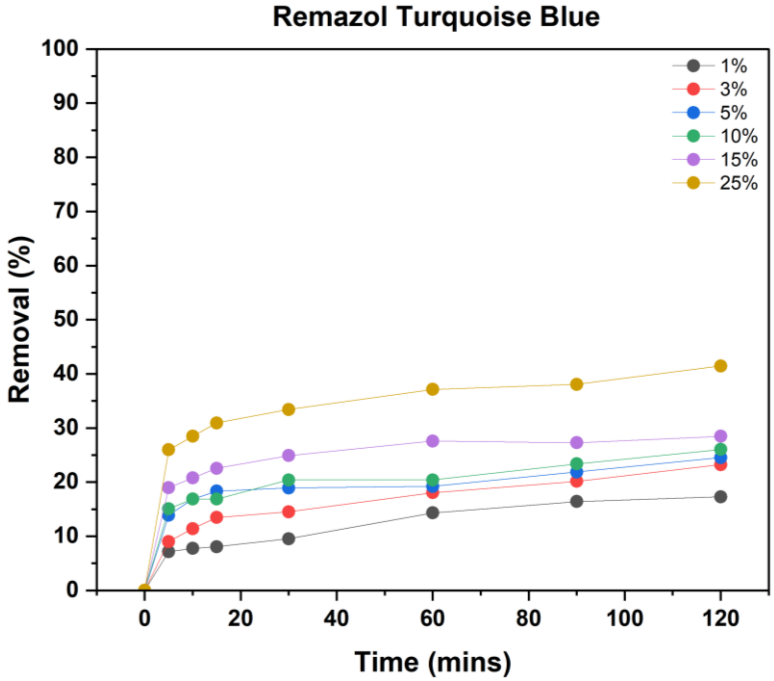


Figure 6 Effect of CuO-CeO2 catalyst concentration on removal **remazol turquoise blue**

Figure 6 shows the Remazol Turquoise Blue (RTB) removal percentage against time with variations in CuO-CeO2 catalyst concentration (1%, 3%, 5%, 10%, 15%, and 25%). The figure shows that the percentage *removal* tends to increase with time. Increasing the catalyst concentration appears to influence the RTB removal efficiency significantly. The removal rate was relatively slow at low catalyst concentrations (1%, 3%, and 5%), with the final percentage *removal* ranging from 10% to 20%. In contrast, at higher catalyst concentrations (10%, 15%, and 25%), the percentage *removal* experienced a more substantial increase. The 25% catalyst concentration showed the highest effectiveness, with a 40% *removal* percentage within 120 minutes. Based on **Figure 6**, it can be concluded that the use of a high concentration of catalyst causes an increase in the degradation rate initially, then the rate stabilizes at a certain scale. However, if the CuO-CeO2 concentration is more than 25%, the degradation rate shows no further change. This may be due to the clumping of free catalyst particles and the filtering effect (Alkaim 2015). This indicates that at high catalyst concentrations, the dye removal process can take place more effectively, even though the amount of dye in the solution is not very high. At high catalyst concentrations, more active sites are available on the surface of the catalyst to generate hydroxyl radicals (-OH) that can attack and destroy dye molecules (Suarsa 2021). CuO is recognized for its excellent photocatalytic activity, while CeO_2 enhances stability and UV light absorption capacity. This combination enables the more efficient generation of free radicals under UV irradiation, which is crucial for the degradation of dyes (Rahayu et al. 2022). When $CuO@CeO_2$ is irradiated with UV light, electrons in the valence band are excited to the conduction band, resulting in the generation of holes (h^+) and free electrons (e^-). The hydroxyl radicals (·OH) produced are highly reactive and are capable of attacking the

molecular structure of Remazol Turquoise Blue, breaking chemical bonds and converting it into simpler compounds (Osugi et al. 2006).

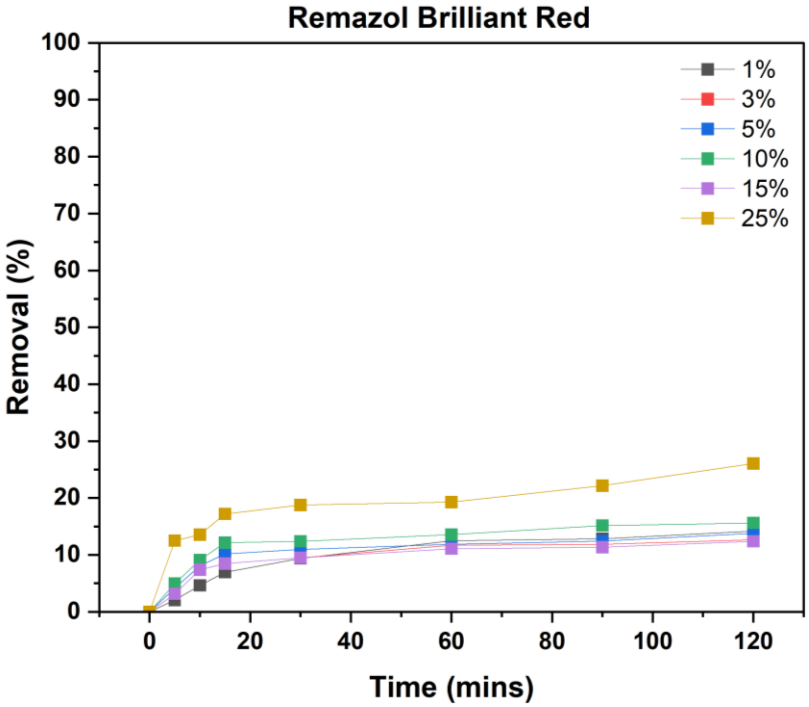


Figure 7 Effect of CuO-CeO2 catalyst concentration on removal **remazol brilliant red**

Figure 7 shows the percentage removal of Remazol Brilliant Red against time at various catalyst concentrations (1%, 3%, 5%, 10%, 15%, and 25%). In general, the percentage removal increases with time, but the impact of increasing concentration on removal efficiency is not very significant at low to medium concentrations. At low concentrations (1%, 3%, and 5%), the removal rate was relatively slow, with efficiencies only reaching about 5% to 12% after 120 minutes. At higher concentrations (10%, 15%, and 25%), there was a more obvious increase in the removal percentage. Especially at the highest concentration of 25% CuO-CeO2 catalyst, the removal percentage reached nearly 28% within 120 minutes, indicating that at higher concentrations, the removal efficiency of Remazol Brilliant Red can be improved, although the increase is still limited.

Remazol Brilliant Red is characterized by a complex chemical structure and strong bonds, making it more challenging to degrade compared to other dyes, such as Remazol Turquoise Blue. The azo bonds (-N=N-) within its molecules require higher energy to be cleaved, thereby reducing the degradation efficiency (Paulista et al. 2024). Prolonged UV exposure may facilitate higher removal efficiency by promoting additional interactions and radical attacks on the dye molecules (Sibarani 2019). The dye is generally negatively charged in solution, which may reduce electrostatic interactions with the photocatalyst surface. If the CuO@CeO_2 surface is not sufficiently positively charged at a specific pH, the adsorption of the dye is hindered, resulting in lower degradation efficiency. The reduced adsorption limits the availability of dye molecules at the active sites of the photocatalyst, thereby decreasing the generation of reactive radicals necessary for the degradation process. Consequently, optimizing the surface charge of CuO@CeO_2 under different pH conditions is essential to enhance the interaction between the photocatalyst and the dye, improving the overall degradation performance (Gunawan 2018).

4. Conclusions

At a CuO-CeO_2 catalyst concentration of 25%, the degradation and removal efficiency of Remazol Turquoise Blue (RTB) and Remazol Brilliant Red (RBR) reached the highest values compared to other concentrations. For RTB, the degradation showed a decrease in the C/Co value from 0.74 to 0.59 in 120 minutes, while for RBR, the C/Co value dropped from 0.87 to 0.74 in the same period. On the removal

aspect, the 25% concentration resulted in a removal percentage of about 40% for RTB and almost 28% for RBR in 120 minutes. This indicates that the high catalyst concentration provides more active sites that favor the formation of hydroxyl radicals (-OH), which accelerates the removal process of dye molecules. As a suggestion for future research, it is advisable to explore the CuO-CeO2 catalyst to see if there is a more significant increase in degradation efficiency, although be aware of potential negative effects such as particle agglomeration and screening effects that may inhibit the reaction. In addition, further research could consider variations in other parameters, such as solution pH and light intensity, which may also affect the degradation efficiency. Research into how to prevent or reduce the catalyst clumping effect may also help improve the effectiveness of the process at high catalyst concentrations.

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