Photocatalytic degradation of rhodamine B using Cu_2O prepared via *Terminalia catappa* leaf-extract route

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ABSTRACT

A green and sustainable method for synthesizing a cuprous oxide (Cu_2O) photocatalyst was established using the extract from Terminalia catappa leaves as a natural reducing and stabilizing agent. The resulting Cu_2O displayed a well-defined cubic morphology, high crystallinity, and phase purity, as confirmed by SEM, XRD, FTIR, and EDX analyses. UV-Vis diffuse reflectance spectroscopy indicated strong visible-light absorption, with characteristic bands at 449 and 475 nm and a narrow band gap of 2.12 eV, supporting its suitability for visible-light-driven photocatalysis. Photocatalytic activity was assessed via degradation of Rhodamine B (RhB) under visible-light irradiation, achieving over 90% degradation within 120 minutes. Kinetic analysis showed that the photodegradation data were best fitted by a pseudo-second-order model, reflecting an apparent kinetic behavior in which surface reactions and adsorption-related processes involving photogenerated charge carriers play a dominant role. Reusability tests demonstrated satisfactory stability, with the catalyst maintaining more than 80% degradation efficiency after five cycles. These findings demonstrate the potential of Terminalia catappa leaf-extract-mediated Cu_2O as an eco-friendly, low-cost, and efficient photocatalyst for wastewater treatment.

Keywords: Green synthesis; Cu_2O photocatalyst; Terminalia catappa leaf extract; Rhodamine B; photodegradation.

1. INTRODUCTION

Synthetic dyes are widely used in the textile, printing, paper, and cosmetic industries, resulting in substantial volumes of colored wastewater that pose significant environmental and health risks [1]. Rhodamine B (RhB), a representative xanthene dye, is characterized by high stability, vivid coloration, and resistance to conventional biological treatment methods [2]. The release of RhB-containing effluents can suppress photosynthetic processes in aquatic environments and induce toxic, mutagenic, and carcinogenic effects in various organisms. Consequently, the development of effective, sustainable technologies for removing RhB from water is critically important. Advanced oxidation processes, especially semiconductor-based photocatalysis, have shown promise due to their capacity to mineralize organic pollutants into non-toxic end products under mild conditions [3-6]. Nevertheless, the widespread implementation of photocatalysis remains constrained by factors such as catalyst costs, synthesis complexity, and environmental sustainability, underscoring the importance of green, efficient photocatalytic materials.

Cuprous oxide (Cu_2O) has attracted significant attention as a visible-light-responsive photocatalyst due to its narrow band gap, strong light absorption, abundance on Earth, and low toxicity [7, 8]. Cu_2O-based materials exhibit strong performance in the photocatalytic degradation of organic dyes and other pollutants under visible light irradiation [9]. However, conventional synthesis methods for Cu_2O frequently require toxic reducing agents, high energy input, and complex procedures, which are inconsistent with green chemistry principles. Recently, plant-extract-mediated green synthesis has emerged as an environmentally sustainable alternative,

employing natural biomolecules such as polyphenols, flavonoids, and organic acids as reducing and stabilizing agents [10-12]. This method not only minimizes the environmental impact of material synthesis but also enables improved control over particle size, morphology, and surface functionality, all of which are critical factors influencing photocatalytic performance.

Using leaf extracts as bio-reductants for Cu_2O synthesis is a sustainable and cost-effective approach, since leaves are a plentiful, renewable resource containing phytochemicals that facilitate metal ion reduction and nanoparticle stabilisation. Terminalia catappa leaves, in particular, are rich in polyphenols, flavonoids, tannins and organic acids, enabling them to act as natural reducing and stabilising agents. Terminalia catappa is widely available in tropical regions, is low-cost, and is often regarded as agricultural waste, which supports its potential for scalable green synthesis. The phytochemical composition of Terminalia catappa may also promote the controlled nucleation and growth of Cu_2O, resulting in well-defined morphology and good crystallinity, all without the need for external surfactants or toxic reagents [9].

However, the use of Terminalia catappa leaf extract for Cu_2O photocatalyst synthesis and its role in dye photodegradation remain insufficiently explored. In this study, Cu_2O was synthesized via a green leaf-extract route and evaluated for RhB photodegradation under light irradiation, with emphasis on synthesis, photocatalytic performance, and degradation behavior for sustainable wastewater treatment.

2. EXPERIMENTAL

2.1. Materials and Chemicals

Copper(II) salt ($CuSO_4 \cdot 5H_2O$), NaOH, ethanol, and Rhodamine B (RhB) were of analytical grade and used without further purification. Fresh *Terminalia catappa* leaves were washed thoroughly with deionized water and air-dried.

2.2. Preparation of *Terminalia catappa* leaf extract

Dried leaves were cut into small pieces and boiled in deionized water (5 g in 100 mL) at 95 °C for 60 min under stirring. The mixture was cooled to room temperature and filtered (Whatman No. 1). The filtrate was stored for synthesis.

2.3. Green synthesis of Cu_2O

A 0.05 M $CuSO_4$ solution was stirred at room temperature while the leaf extract was added dropwise under continuous agitation. The pH was adjusted to approximately 11-12 using NaOH to facilitate Cu_2O formation. The resulting suspension was stirred for 1-2 hours until a brick-red precipitate formed, followed by a 30-minute aging period. The precipitate was isolated by centrifugation, washed multiple times with deionized water and ethanol, and subsequently dried under a stream of dry air.

2.4. Characterization of Cu_2O

The crystalline phase was identified by X-ray diffraction (XRD, Cu Kα). SEM was used to examine morphology and particle size. Surface functional groups were analyzed by FTIR, and optical properties were evaluated by UV-Vis diffuse reflectance spectroscopy (DRS) to estimate the band gap.

2.5. Photocatalytic degradation of RhB

Photocatalytic activity was assessed using a RhB solution (5 $mg.L^{-1}$, 100 mL) with a controlled Cu_2O dosage (0.5-1.0 $g.L^{-1}$). Before irradiation, the suspension was magnetically stirred in the dark for 60 minutes to achieve adsorption-desorption equilibrium. Visible-light irradiation was applied using a 350 W xenon lamp. At predetermined intervals, aliquots were collected, centrifuged to separate the catalyst, and analyzed by UV-Vis spectroscopy at 554 nm. Degradation efficiency and reaction kinetics were determined using the concentration ratio (C_t/C_0) and the

pseudo-first-order kinetic model, $\ln(C_0/C_t) = kt$.

2.6. Cu_2O catalyst reusability

To evaluate recyclability, the catalyst was recovered after each cycle by centrifugation, then washed and dried. This process was repeated for five cycles. X-ray diffraction (XRD) analysis confirmed the catalyst's structural stability after repeated use.

3. RESULTS AND DISCUSSION

3.1. Characterization of Cu_2O

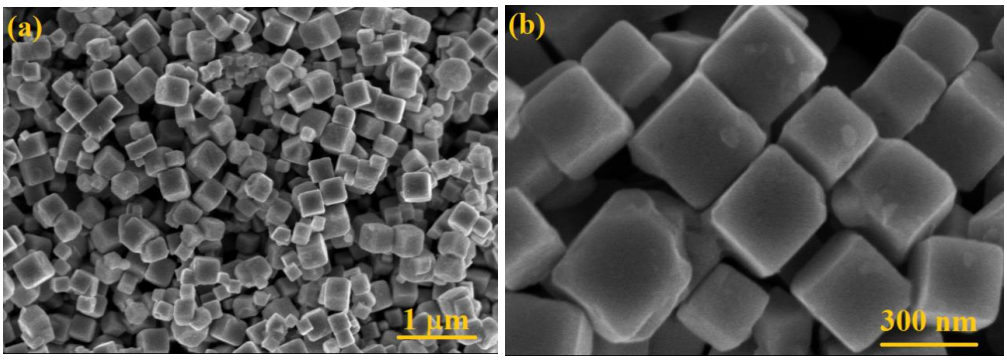

Figure 1. *SEM images of Cu_2O produced by green synthesis.*

Figure 1 displays scanning electron microscopy (SEM) images of Cu_2O synthesized using the *Terminalia catappa* leaf-extract method, demonstrating a highly uniform, well-defined morphology. At low magnification (Figure 1a), the sample predominantly comprises densely packed, cubic-like particles exhibiting high dispersion and minimal agglomeration, suggesting effective stabilization by phytochemicals present in the leaf extract. Higher-magnification imaging (Figure 1b) reveals sharp-edged nanocubes with smooth surfaces and an average particle size of approximately 200-400 nm. This regular morphology indicates controlled nucleation and growth during green synthesis under alkaline conditions. The well-faceted Cu_2O nanocubes offer numerous exposed crystal planes and active sites, which are advantageous for light absorption, charge transfer, and improved photocatalytic degradation of RhB.

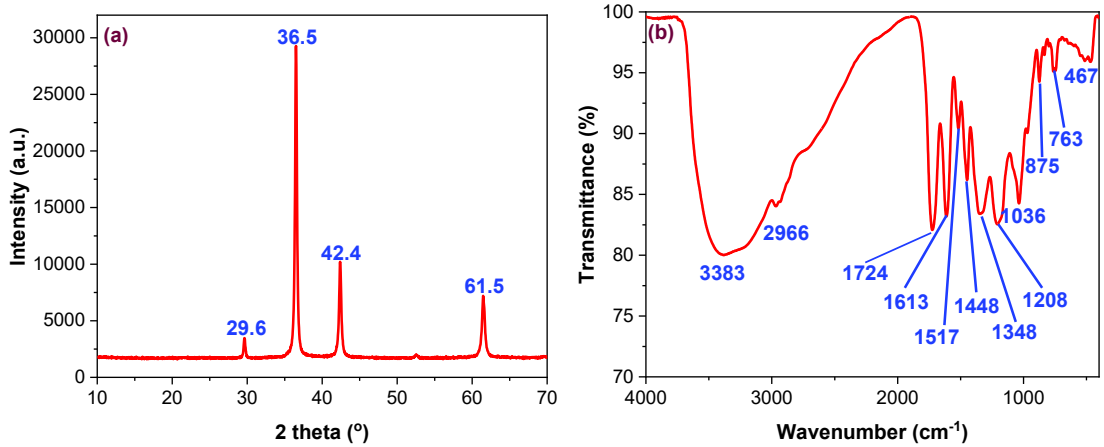

Figure 2. *XRD pattern (a) and FTIR spectroscopy (b) of Cu_2O via green synthesis.*

Figure 2a displays the XRD pattern of Cu_2O synthesized through the green method, with distinct diffraction peaks at $2\theta = 29.6°$, 36.5°, 42.4°, and 61.5°, corresponding to the (110), (111), (200), and (220) crystal planes of cubic Cu_2O. The absence of peaks associated with CuO or metallic Cu indicates high phase purity of the synthesized Cu_2O. The sharp, intense diffraction

peaks indicate satisfactory crystallinity, suggesting that the phytochemicals present in the leaf extract facilitated controlled crystal growth. Figure 2b illustrates the FTIR spectrum of Cu_2O, showing broad absorption at 3383 cm^{-1} due to O–H stretching of surface hydroxyl groups and adsorbed water, and a band at 2966 cm^{-1} attributed to C–H stretching vibrations. Peaks at 1724, 1613, and 1517 cm^{-1} are assigned to C=O and aromatic C=C vibrations from residual organic compounds. Bands below 500 cm^{-1}, particularly at 467 cm^{-1}, are indicative of Cu–O stretching, confirming the formation of Cu_2O.

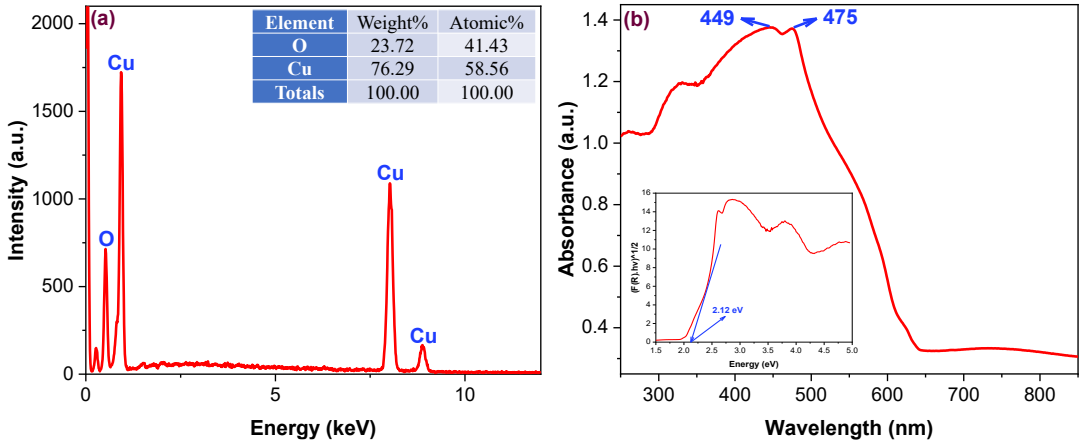

Figure 3. *EDX pattern (a) and UV-Vis DRS spectroscopy with band gap energy diagram (b) of Cu_2O prepared by green synthesis.*

Figure 3a displays the EDX spectrum of green-synthesized Cu_2O, confirming the presence of only copper and oxygen, with no detectable impurities. Quantitative analysis indicates weight percentages of 76.29% Cu and 23.72% O, closely matching the theoretical stoichiometry of Cu_2O and demonstrating successful formation of phase-pure cuprous oxide via the leaf-extract method. The pronounced signals for Cu and O suggest that phytochemicals effectively reduce Cu^{2+} ions without introducing contamination. Figure 3b presents the UV-Vis DRS spectrum, where Cu_2O shows strong visible-light absorption with characteristic maxima at 449 and 475 nm, consistent with its narrow band-gap semiconductor properties. The Tauc plot yields a band gap of 2.12 eV, confirming efficient utilization of visible light. This optical characteristic enhances photocatalytic activity under visible-light irradiation.

3.2. Photocatalytic degradation of RhB

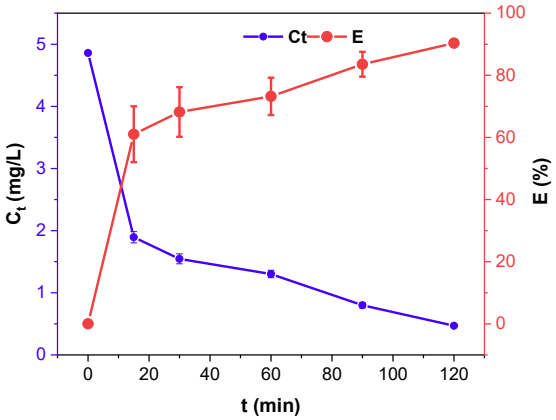

Figure 4. *Temporal variation of Rhodamine B concentration (Ct) and photocatalytic degradation efficiency (E) over Cu_2O under visible-light irradiation (initial RhB concentration = 4.86 $mg.L^{-1}$).*

Figure 4 presents the temporal evolution of RhB concentration (Ct) and photocatalytic degradation efficiency (E) over Cu_2O under visible-light irradiation. The RhB concentration decreases rapidly from an initial value of 4.86 $mg.L^{-1}$ to 1.89 $mg.L^{-1}$ within the first 15 minutes, resulting in a sharp increase in degradation efficiency to approximately 61%. This observation indicates rapid photocatalytic activity during the initial stage, which can be attributed to the abundance of available active sites and efficient generation of reactive species. With continued irradiation, Ct gradually declines to 0.469 $mg.L^{-1}$ after 120 minutes, while the degradation efficiency increases steadily to 90.35%. The slower degradation rate observed at later stages is likely due to the reduced RhB concentration and partial occupation of active sites. These results indicate that green-synthesized Cu_2O maintains high and sustained photocatalytic efficiency for RhB degradation.

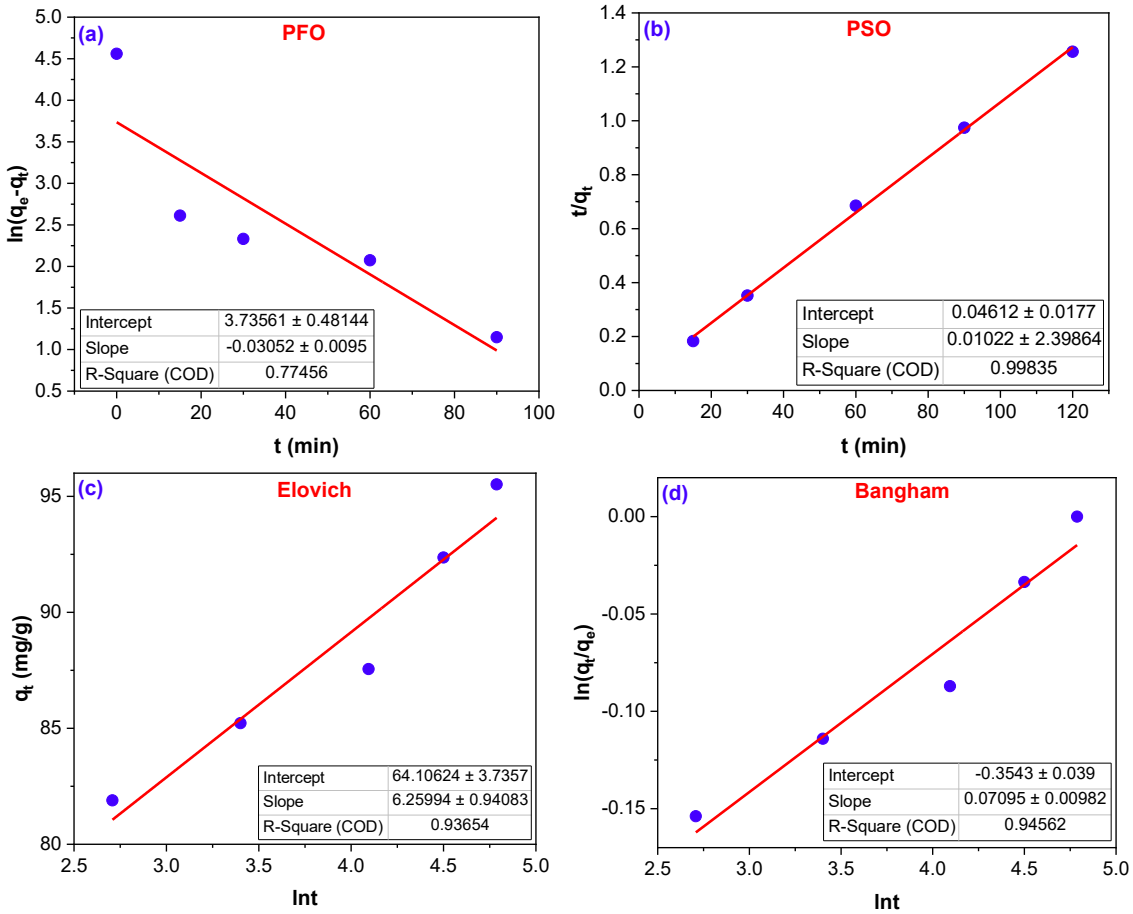

Figure 5. *Kinetic models for RhB photodegradation over Cu_2O: (a) PFO, (b) PSO, (c) Elovich, and (d) Bangham.*

Figure 5 and table 1 show the results of kinetic modeling for the photodegradation of RhB over Cu_2O using different models. The pseudo-first-order (PFO) model yields a relatively low correlation coefficient (R^2 = 0.7746), demonstrating its inadequacy in describing the photodegradation process. The pseudo-second-order (PSO) model, on the other hand, fits very well, with an R^2 value of 0.9984 and a calculated q_e of 10.23 $mg.g^{-1}$. This means that the rate of photodegradation is mainly controlled by surface reactions involving photogenerated charge carriers. The Elovich model also shows a high degree of linearity (R^2 = 0.9365), indicating that the catalytic surface is nonuniform and that the reaction rate is high at the start. Additionally, the Bangham model exhibits a satisfactory correlation (R^2 = 0.9456), signifying that diffusion within

the catalyst pores contributes to the overall degradation process. Collectively, these results indicate that PSO kinetics most accurately represent RhB photodegradation by Cu_2O.

For clarity, the PFO, PSO, Elovich, and Bangham models were employed as apparent kinetic tools to analyze the overall RhB removal behavior under irradiation, rather than to describe elementary photocatalytic reaction steps. It should be noted that the pseudo-first-order and pseudo-second-order models are empirical kinetic approaches. In the present photocatalytic system, the excellent fit of the PSO model is therefore interpreted as an apparent kinetic behavior, suggesting that surface chemisorption and surface reactions involving photogenerated charge carriers play a dominant role in the rate-controlling step. The good agreement with the Elovich model further indicates a heterogeneous catalyst surface and a high initial reaction rate, while the Bangham model implies that intraparticle diffusion also contributes to the overall RhB degradation process. These results confirm that RhB photodegradation over Cu_2O is governed by a combined adsorption–photocatalysis mechanism rather than a simple homogeneous reaction.

Table 1. *Kinetic parameters for RhB photodegradation over Cu_2O under visible ligh.*

Models	**Parameters**	**Value**	**R^2**
Pseudo-first order	k_1 (min^{-1})	0.0305	0.7746
	q_e ($mg.g^{-1}$)	4.3842	
Pseudo-second order	k_2 ($g.mg^{-1}.min^{-1}$)	0.0217	0.9984
	q_e ($mg.g^{-1}$)	10.2302	
Elovich	a ($mg.g^{-1}$)	18347.75	0.9365
	b ($g.mg^{-1}$)	1.5272	
Bangham	α	0.0710	0.9456
	k_B	0.7017	

3.3. Cu_2O catalyst reusability

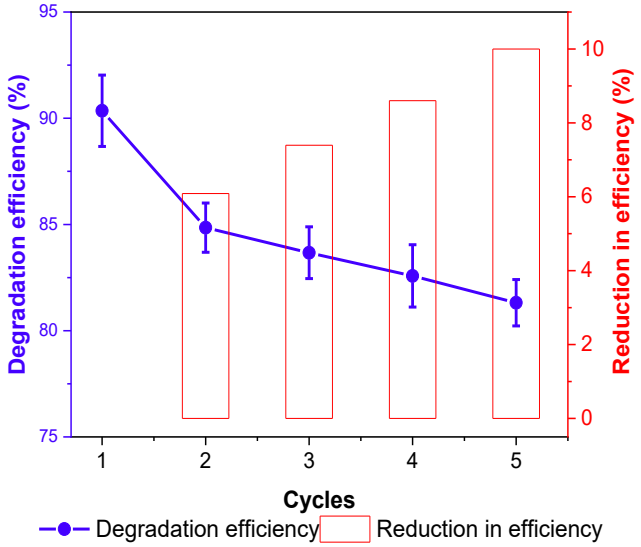

Figure 6. *Recyclability and stability of Cu_2O photocatalyst for RhB photodegradation under visible light.*

Figure 6 presents the recyclability of Cu_2O in RhB photodegradation across five consecutive cycles. The degradation efficiency decreases from 90.35% in the first cycle to 81.32% after the

fifth cycle, which suggests sustained catalytic stability during repeated use. The reduction in degradation efficiency increases from 0% to approximately 9.99% over five cycles. This moderate decline is likely due to partial surface fouling, loss of active sites, or minor photocorrosion during repeated irradiation and recovery. Despite this, Cu_2O maintains over 80% degradation efficiency after five cycles, indicating satisfactory reusability and structural integrity, which supports its potential for practical photocatalytic wastewater treatment applications.

4. CONCLUSIONS

A Cu_2O photocatalyst was synthesized via a green method using *Terminalia catappa* leaf extract as a natural reducing and stabilizing agent. Structural and spectroscopic analyses confirmed the formation of phase-pure, well-crystallized Cu_2O nanocubes with a narrow band gap of 2.12 eV and strong visible-light absorption. The green-synthesised Cu_2O demonstrated high photocatalytic activity for RhB degradation, achieving over 90% removal within 120 minutes under similar sunlight irradiation. Kinetic analysis indicated that the photodegradation process followed a pseudo-second-order model, suggesting that surface reactions involving photogenerated charge carriers were predominant. Reusability tests revealed that Cu_2O maintained more than 80% degradation efficiency after five cycles, confirming its stability and recyclability. These findings underscore the effectiveness and sustainability of leaf-extract-mediated Cu_2O synthesis and its significant potential for eco-friendly wastewater treatment applications.

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TÓM TẮT

Phân hủy quang xúc tác Rhodamine B sử dụng Cu_2O được tổng hợp theo phương pháp xanh từ dịch chiết lá bàng (*Terminalia catappa*)

Một phương pháp xanh và bền vững để tổng hợp xúc tác quang cuprous oxide (Cu_2O) đã được xây dựng, trong đó dịch chiết từ lá bàng (Terminalia catappa) được sử dụng như tác nhân khử và ổn định tự nhiên. Vật liệu Cu_2O thu được thể hiện hình thái lập phương rõ ràng, độ kết tinh cao và độ tinh khiết pha tốt, được xác nhận thông qua các phân tích SEM, XRD, FTIR và EDX. Phổ phản xạ khuếch tán UV-Vis cho thấy Cu_2O hấp thụ mạnh trong vùng ánh sáng khả kiến, với các dải hấp thụ đặc trưng tại 449 và 475 nm và năng lượng vùng cấm hẹp 2,12 eV, chứng tỏ khả năng ứng dụng hiệu quả trong xúc tác quang dưới ánh sáng khả kiến. Hoạt tính quang xúc tác của Cu_2O được đánh giá thông qua quá trình phân hủy Rhodamine B (RhB) dưới chiếu xạ ánh sáng khả kiến, đạt hiệu suất phân hủy trên 90% sau 120 phút. Phân tích động học cho thấy quá trình phân hủy quang RhB phù hợp nhất với mô hình giả bậc hai, cho thấy tốc độ phản ứng chủ yếu bị chi phối bởi các quá trình trên bề mặt xúc tác, bao gồm hấp phụ và các phản ứng liên quan đến các hạt tải điện quang sinh. Thử nghiệm tái sử dụng chứng minh vật liệu có độ ổn định tốt, khi vẫn duy trì hiệu suất phân hủy trên 80% sau năm chu kỳ. Những kết quả này khẳng định tiềm năng của Cu_2O tổng hợp xanh từ dịch chiết lá bàng như một xúc tác quang thân thiện môi trường, chi phí thấp và hiệu quả cao cho xử lý nước thải.

Từ khoá: Tổng hợp xanh; Xúc tác quang Cu_2O; Dịch chiết lá bàng (Terminalia catappa); Rhodamine B; Phân hủy quang.