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Enhanced photocatalytic degradation of methylene blue dye using CuO nanoparticles from the fruit extracts of *Diplocyclos palmatus* (L) C. Jeffrey for wastewater remediation

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ABSTRACT

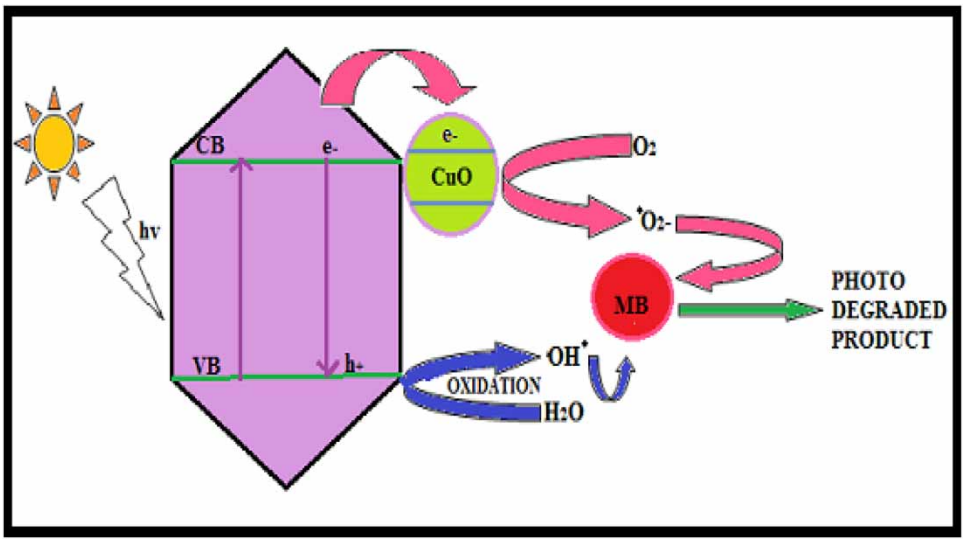
The degradation of synthetic organic dyes through photocatalysis requires structured nanoparticles (NPs). Copper oxide (CuO) NPs were used as the newest developed catalyst for the organic dye degradation under the irradiation of sunlight: The metal oxide NPs were synthesized using the fruit extract of *Diplocyclos palmatus* (L) C. Jeffrey. UV–visible light spectra showed an absorption band for CuO NPs at 277 nm. X-ray diffraction confirmed the monoclinic phase of CuO NPs. Scanning electron microscopy confirmed rod-shaped stacked particles. Energy-dispersive X-ray spectra indicating the presence of Cu in oxide form. For the degradation of methylene blue dye (MB), CuO NPs were used as photocatalyst for dye degradation under sunlight irradiation. This study confirmed the effects, such as irradiation time, concentration, pH, and catalyst dosage. The CuO NPs showed high photocatalytic degradation, degrading 90% of the dye in 120 min. The increased degradation of dye was observed on exposure to longer irradiation time. The increased concentration of dye resulted in a decreased rate of photodegradation. 95% degradation achieved at pH 9 showing that the pH medium also enhances the degradation. A higher dye degradation percentage was achieved by increasing the higher catalyst dosage of 0.013 g degraded 93% of dye. Thus in the treatment of wastewater containing pollutants, the synthesized CuO NPs acted as an efficient photocatalyst.

Key words: CuO nanoparticles, *D. palmatus* fruit extracts, methylene blue, photocatalytic dye degradation, wastewater treatment

HIGHLIGHTS

- The latest development for the removal of organic pollutants is of significant concern.
- The dye degradation is affected by the catalyst amount, surface area, and size.
- The CuO nanoparticles from the fruit extract of *palmatus* exhibited an advanced oxidation process that degrades pollutants with higher concentrations.
- Photocatalytic methods are discussed.
- This can be used for more efficient wastewater remediation in the future.

GRAPHICAL ABSTRACT



INTRODUCTION

In the wake of worldwide populations and industrial sectors, water resources have increased to epidemic levels due to various pollutants. This contagion pollutant causes a serious threat to water sources, resulting in the scarcity of water and affecting the quality of water. Therefore, it is necessary to prevail over the problems that protect the water supply, human health, and environment (Amdeha 2021; Mostafa & Amdeha 2022). Water pollution is an important problem arising. The main cause of pollution is the release of hazardous, toxic, and non-degradable (Qiu *et al.* 2008) dyes from industrial sectors. Organic dyes are an important cause of water pollution. These organic dyes not only affect the process of photosynthesis but also the health of living beings through skin irritation, allergies, and even cancer (Royer *et al.* 2010).

Moreover, 10–15% of dyes released into water resources are in the form of effluents from the textiles during the process of dyeing. Methods like biological and physicochemical processes have been used to treat the dyed water (Kumar *et al.* 2011; Mahmoodi *et al.* 2011; Singh & Dhaliwal 2020). These methods are sometimes found to be unsustainable for the environment. The development of nanotechnology created a possibility for the treatment of wastewater. Copper oxide nanoparticles (CuO NPs) are found to be effective. The intrinsic properties found in the CuO NPs are greatly suitable for the photocatalytic degradation process (Nagar & Devra 2019).

Organic molecules contained in the contagion wastewater are broken down by the technique of photocatalytic dye degradation, which releases hydroxyl ($^{\bullet}OH$), radicals using the necessary catalyst and radiation. The toxic compounds present in the water pollutants are converted into environmentally safe end products such as H_2O and CO_2 through these radicals (Kamat 2012; Hammad *et al.* 2018). Compared with other conventional processes, photocatalytic degradation is extensively used. Photocatalytic degradation can be applied to main stream activity due to its outstanding sustainability, low price, greater effectiveness, and lower pollution (Kumar *et al.* 2014).

Under temperate and suitable environmental conditions, the photocatalytic process undergoes excellent dye degradation and converts to hazardous minerals (Zhao *et al.* 2023). Thus, the degradation of synthetic organic dyes can be achieved (Velusamy & Lakshmi 2017). The most commonly used photocatalysts are metal and metal oxide NPs because of their distinct properties with a size of less than 100 nm. Methylene blue (MB) is used in paper, wool, silk, and cotton as a synthetic dye (Khodaie *et al.* 2013). In cosmetics, pharmaceuticals, and food colors, those dyes are also used. Unlike polluted water, MB is used in therapeutic fields (Dardouri & Sghaier 2017).

The present work involves the synthesis of CuO NPs from the fruit extracts of *Diplocyclos palmatus* (L) C. Jeffrey (*D. palmatus*) (Gautam *et al.* 2013) as a photocatalyst through the co-precipitation method, to improve the efficacy of the degradation of synthetic organic dyes. Plants are used because of their availability, less toxic nature, and capping behavior. Among metals, CuO NPs are used because of their unique properties. UV–visible

light spectra, scanning electron microscopy (SEM), and energy-dispersive X-ray spectroscopy (EDX) had been used to study the catalyst's characteristics. The aim of the work is to synthesize a cost-effective and efficient photocatalyst for MB dye degradation. The parameters that affect the dye degradation include pH medium, time, dosage of the photocatalyst, and dye concentration. On studying this aspect, it is much needed to optimize the degradation of synthetic organic dyes.

MATERIAL AND METHODS

Materials and chemicals

The chemicals copper sulfate pentahydrate ($CuSO_4{\cdot}5H_2O$) and sodium hydroxide (NaOH) were purchased from Merck. From Sathyamangalam in the Tamil Nadu district of Erode, fruits of *D. palmatus* were gathered.

Preparation of fruit extracts

The fruits were washed three or four times with distilled water and dried for 40 days. The dried fruits were crushed and processed into powder. The powder weighed 25 g, and 250 mL of distilled water was added. The mixture was stirred using a magnetic stirrer for 2 h. The solution was then filtered using Whatman filter paper No. 1 and stored for further nanoparticle synthesis.

Synthesis of CuO NPs

Plant-mediated green synthesis has been used to produce CuO NPs, in which different functional groups found in fruits act as reducing agents, culminating in the formation of NPs. In a beaker, 25 ml of fruit extract was added, followed by 30 ml of 1 M aqueous $CuSO_4{\cdot}5H_2O$. The mixture was then placed on a magnetic stirrer for 1 h, after which NaOH was added drop by drop, and a color change was observed. The resultant extract solution was centrifuged at 7,000 rpm for 20 min, and its supernatant was discarded. The resulting pellet containing CuO NP precipitate was collected and rinsed with distilled water to remove contaminants before being transferred to a China dish. The precipitate was dried in an oven at 70 °C. The dried fine powder was used for characterization.

Photodegradation of MB dye using CuO NPs

The breakdown of MB dye under UV irradiation was investigated using CuO NPs (Jassim *et al.* 2016). In a standard flask, approximately 0.0025 g of MB dye was mixed with 250 ml of distilled water. Various ppm dye solutions (10, 20, 30, and 40 ppm) have been produced. Each beaker holding 100 ml of (10 ppm) MB dye solution had 0.005 g of CuO NPs added separately (Dulta *et al.* 2022; Aroob *et al.* 2023). They were placed in the dark for approximately 30 min to achieve adsorption–desorption equilibrium. The mixtures were then exposed to sunlight for an assortment of time periods. The degradation of MB dye has been examined using a UV–visible spectrophotometer.

The study examines the effects of catalyst dosage, length of time, dye concentration, and pH medium on the photocatalytic degradation of MB. Different quantities of catalyst were employed to investigate the catalyst dose. The pH medium was tested using varied pH solution concentrations of 5, 7, and 9 to determine the impact of pH on MB dye degradation. Before synthesizing the dye solution, 1M HCl and 1M NaOH solutions were added to distilled water to create acidic and basic solutions. Different time intervals (5, 15, 30, 45, 60, 75, 90, 105, and 120 min) were investigated. Similarly, different dye concentrations of 10, 20, 30, and 40 ppm are investigated for the photocatalytic degradation of MB dye.

Dye degradation UV–visible analysis

A UV–visible spectrophotometer was used to study the photocatalytic breakdown of MB dye. The dye degradation percentage was calculated using the formula shown below.

$$\text{Degradation rate}(\%) = \left(\frac{C_0 - C}{C_0}\right) \times 100 \tag{1}$$

$$\text{Degradation rate}(\%) = \left(\frac{A_0 - A}{A_0}\right) \times 100 \tag{2}$$

where C stands for dye concentration, C_0 stands for initial dye concentration, A is the absorbance of dye after irradiation with sunlight, and A_0 shows the initial absorbance.

Characterization

The synthesized CuO NPs from *D. palmatus* fruit extracts were characterized using a UV–visible spectrophotometer, X-ray diffraction (XRD), SEM, and EDX analysis. The absorption spectra of CuO NPs have been studied using UV–visible spectroscopy. A scanning electron microscope (Carl Zeiss USA-Model-Sigma with Gemini Column) was used to examine the particle size and surface morphology of the produced CuO NPs. The elemental composition of NPs was studied using EDX investigations.

RESULTS AND DISCUSSION

UV–visible analysis

In UV–visible spectrum, CuO NPs typically exhibit a prominent absorption band of about 200–300 nm. The synthesized CuO NPs from *D. palmatus* fruit extracts showed an absorption band at 277 nm (Abboud *et al.* 2014), resulting in the production of CuO NPs as shown in Figure 1.

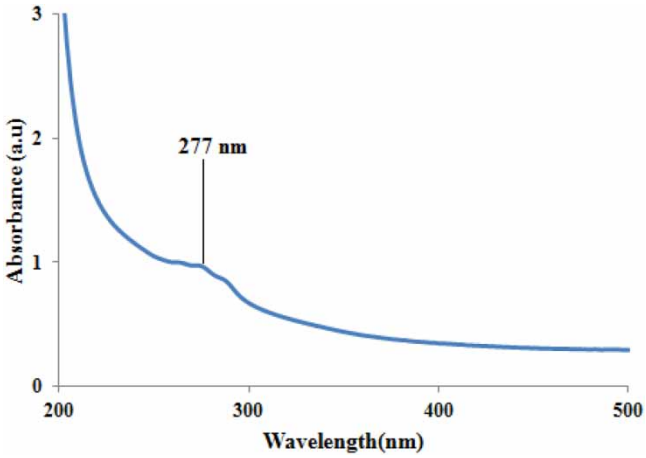


Figure 1 | UV–visible graph of prepared CuO NPs.

XRD analysis

The XRD analysis was studied to understand the crystalline structure. Major peaks were observed during the XRD analysis at Braggs angles of 23.8°, 35.4°, 48.8°, and 66.1°, which corresponded to the lattice planes of (110), (111), (202), and (311) as shown in Figure 2, which are in good agreement with the monoclinic phase

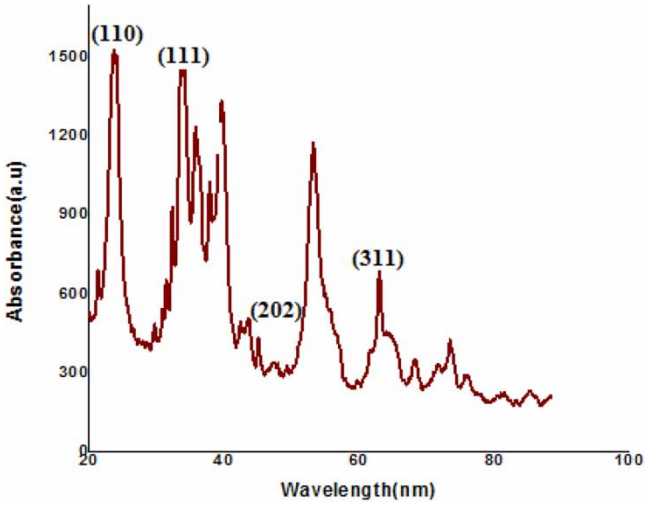


Figure 2 | XRD graph of prepared CuO NPs.

of CuO NPs (JCPDS no. 48-1548). The NP size was determined using the Debye-Scherer formula.

$$D = \frac{K\lambda}{\beta \cos \theta} \tag{3}$$

In this equation, D represents particle size, K is a shape factor (approximately equal to 0.9), λ is its X-ray wavelength (1.5418 A° with Cu K radiation), β is the full line width at half-maximum (FWHM) of the primary intensity peak, and θ is the Bragg's angle. The average particle size for the synthesized CuO NPs is 22 nm.

SEM and EDX analysis

The surface morphology of CuO NPs was analyzed using a scanning electron microscope. The CuO NPs exhibited a rod-shaped stacked structure (Xu *et al.* 2018). The elemental composition of CuO NPs was determined by EDX analysis. The weight percentages of copper and oxygen were found to be 30 and 11%, respectively, as illustrated in Figure 3.

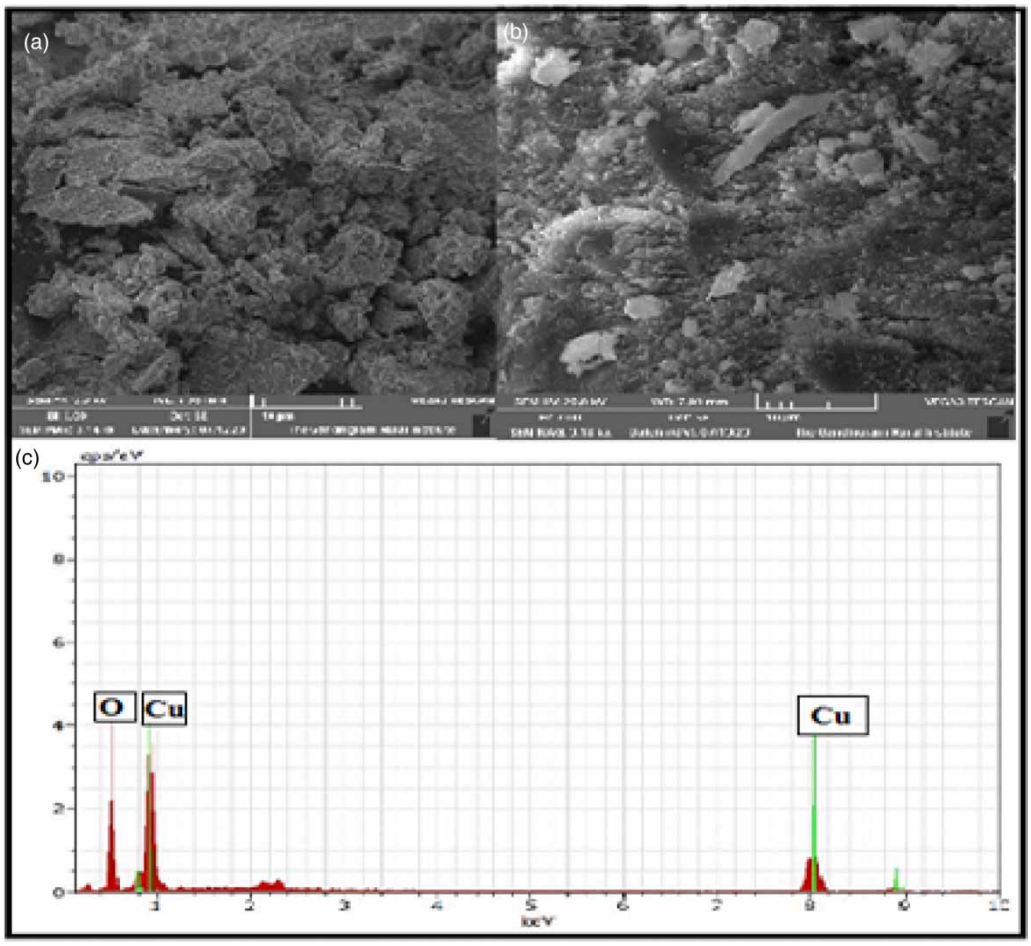


Figure 3 | Scanning electron microscopy image of CuO (a, b) and EDX spectrum (b).

Mechanism for photodegradation of MB dye using CuO NPs from *D. palmatus* fruit extract

The photocatalytic dye degradation process involves multiple phases, including adsorption–desorption, electron–hole pair generation, and chemical reaction (Din *et al.* 2017). (1) Systematic adsorption by MB dye molecules; (2) adsorption of incident light photons; and (3) formation of oxidizing and reducing molecules, all of which result in charge-transfer reactions (Elkady & Hassan 2021). When exposed to sunlight, electrons (e^-) are elevated from the surface of the photocatalyst (CuO NPs) to the conduction band, where they get captured by positive holes created in the valence band (VB) of the synthesized CuO NPs. The reactive intermediates are promptly oxidized

through the positive holes by dye molecules with high oxidative properties, or hydroxyl radicals (•OH) are generated when they react with water (H_2O). The superoxide anions are developed as a result of the reaction between O_2 molecules and electrons in the CuO NPs conduction band.

$$CuO - NPs + h\nu \rightarrow CuO - NPs\ (h^+ + e^-) \tag{4}$$

$$h^+ + \text{MB dye} \rightarrow \text{Oxidation of dye molecules } h^+ + \frac{OH^-}{H_2O} \rightarrow {}^\bullet OH \tag{5}$$

$$e^- + O_2 \rightarrow {}^\bullet O_2^- \tag{6}$$

$${}^\bullet OH + {}^\bullet O_2^- + \text{MB dye} \rightarrow \frac{\text{Degradable}}{\text{Toxic less}} \tag{7}$$

The difference in energy between the oxidation and reduction processes is determined by the material band gaps. The photocatalytic degradation of metals is determined by their band gaps. A narrower band gap indicates greater photocatalytic dye degradation activity.

Effect of dye concentration of CuO NPs on the photocatalytic degradation

The photocatalytic dye degradation process of MB dye was initially studied at concentrations ranging from 10 to 40 ppm. Figure 4 depicts the dilution of the MB dye solution at various doses (10, 20, 30, and 40 ppm). The

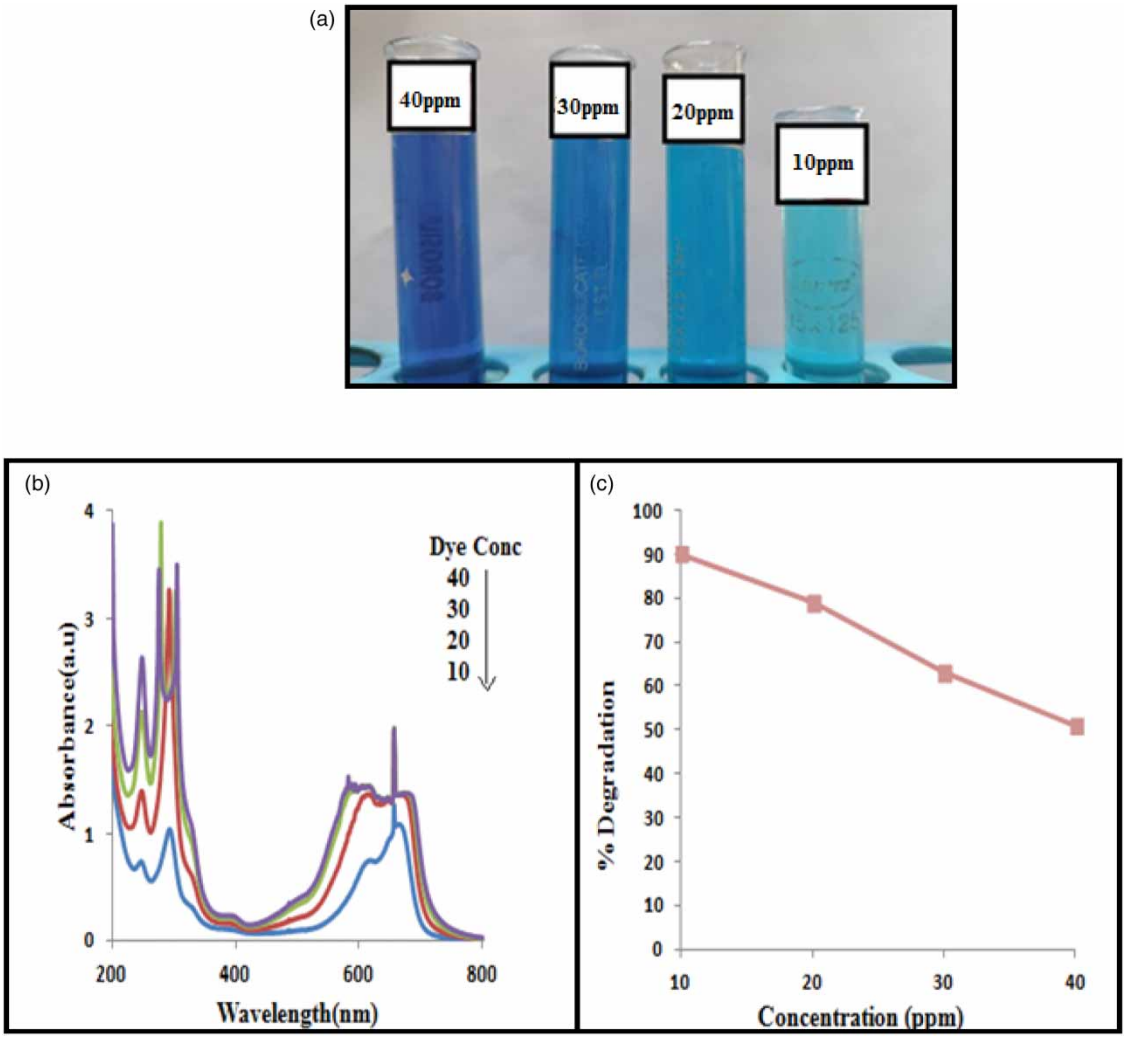


Figure 4 | Photographic images of dilution at different dye concentrations (a), UV–visible graph for 10, 20, 30, and 40 ppm dye solutions of MB dye degraded for 120 min (b), and percentage degradation of MB dye at 120 min (c).

degrading conduction of dye was examined during a 120-min period. The dye intensity and color remained unchanged at 10 ppm; as the concentration increased to 20, 30, and 40 ppm, the color became more intense. The rate of disintegration slowed as the dye concentration increased. The UV–visible graph indicates that degradation decreases with increasing dye concentrations. Beer–Lambert's law states that as dye concentration increases, the number of photons entering the path length decreases, resulting in poor photon absorption and photodegradation (Sundar & Kanmani 2020). The percentage degradation for 10, 20, 30, and 40 ppm over 120 min is 90, 79, 63, and 51%, respectively.

Effect of time of CuO NPs on the photocatalytic degradation

The solution containing 10 ppm of MB dye solution was subjected to dye degradation by CuO NPs over a period of time. The MB dye absorption peak at 665 nm decreases with sunshine exposure over time intervals of 0, 5, 15, 30, 45, 60, 75, 90, 105, and 120 min. Figure 5(a) shows the intense color as the dye gradually saturated to lighter

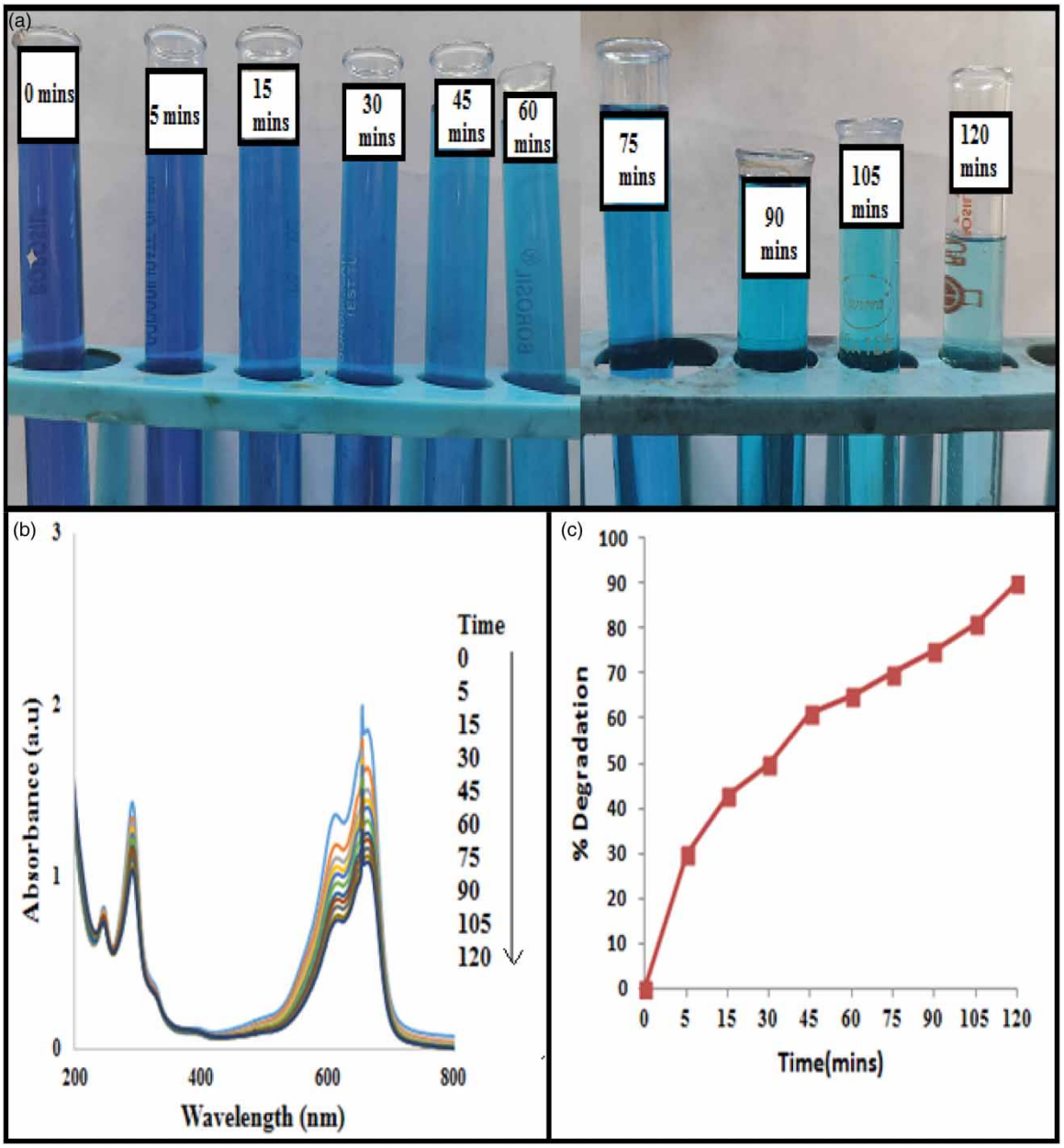


Figure 5 | Photographic images of dilution and catalytic activity of MB dye after degradation at different time intervals (a), UV–visible graph of MB dye degradation at different time intervals (b), and percentage degradation of MB dye at different time intervals (c).

hues, showing that photocatalytic degradation is ongoing through the breakdown of dye molecules. The dye degraded at a rate of 30, 43, 50, 61, 65, 70, 75, 81, and 90% for 5, 15, 30, 45, 60, 75, 95, 105, and 120 min. The gradual increase in MB dye molecule degradation on photocatalytic activity confirms the continuous breakdown of dye molecules.

Effect of pH concentration of CuO NPs on photocatalytic degradation

The pH of the medium influences the surface properties of the chemical reaction. The colors emitted by diverse industries, such as textiles and paints, have varying pH levels. The effect of pH on photocatalytic dye degradation was investigated for pH 5, 7, and 9. pH influences the formation of hydroxyl ions in the breakdown process of textile dyes. Figure 6(a) depicts the hue transition that occurs at different pH levels during photocatalytic

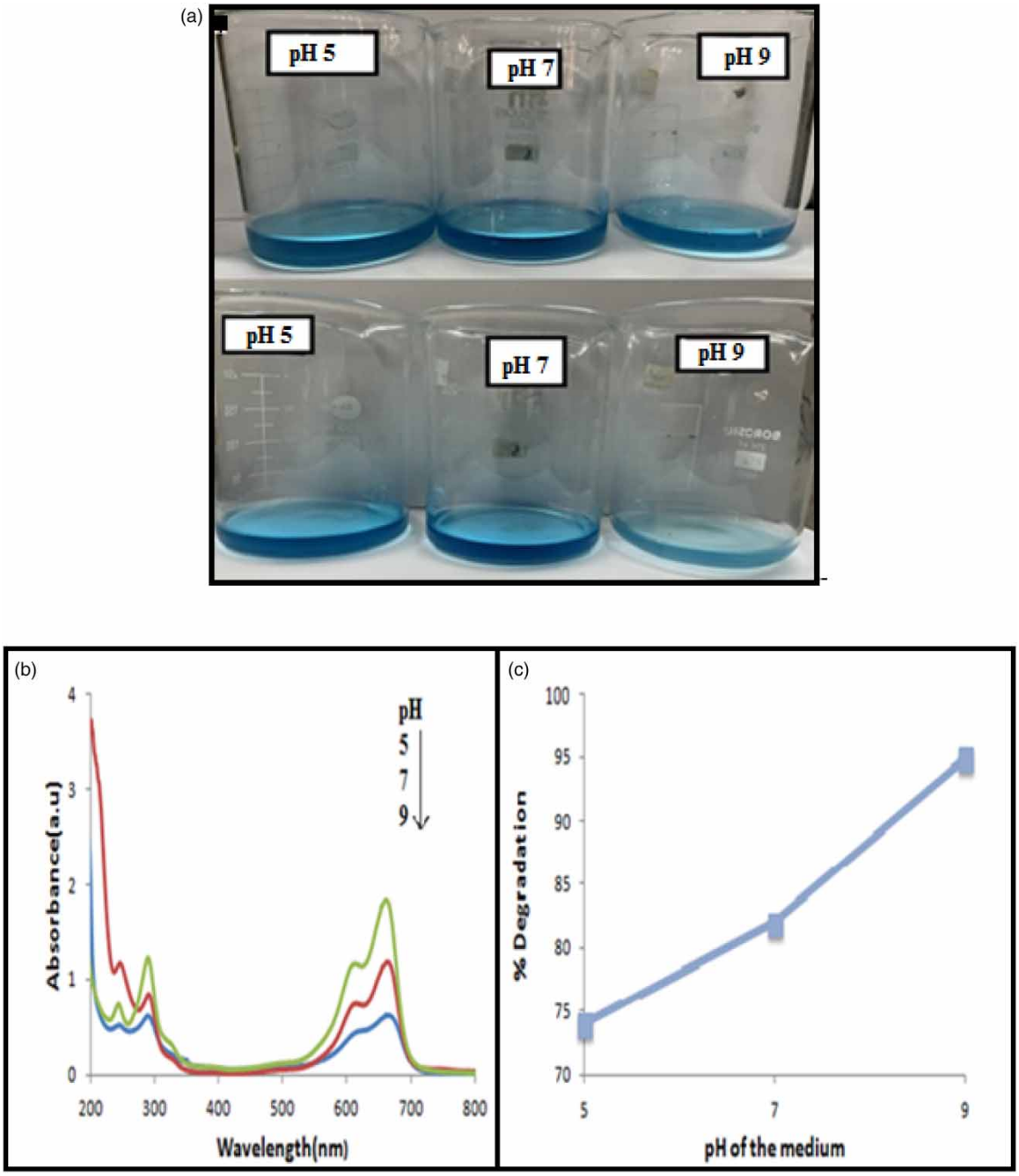


Figure 6 | Original solutions before and after degradation at pH 5, 7, and 9. The top image pairs indicate original solution before degradation while the bottom image represents original solution after degradation (a), UV–visible graph of MB dye degraded at 120 min at different pH mediums (b), and percentage degradation of MB dye at different pH levels (c).

degradation. The color of the dye solution becomes less bright as the pH increases, resulting in the breakdown of MB dye molecules due to the production of active OH radicals at higher pH levels (Hassaan *et al.* 2016). The degradation percentages at pH 5, 7, and 9 were 74, 82, and 95%, respectively.

Effect of catalyst dosage on photocatalytic degradation

Figure 7(a) depicts the degradation process with catalyst dosages of 0.005, 0.007, 0.009, 0.011, and 0.013 g. The effect of the catalysts on the degradation of MB dye was examined by increasing the catalyst concentration from 0.005 to 0.013 g per 20 ml of MB dye solution for 120 min, and their ratio is presented in Table 1. As the catalyst dosage increases, so does the degradation of MB dye molecules due to catalyst particle agglomeration or aggregation, which limits the availability of active surface sites. An increased surface area at a higher catalyst dosage leads to better catalyst interaction, resulting in a larger percentage of dye removal. After 120 min, the degradation rates were 55, 61, 72, 84, and 93% for 0.005, 0.007, 0.009, 0.011, and 0.013 g, respectively.

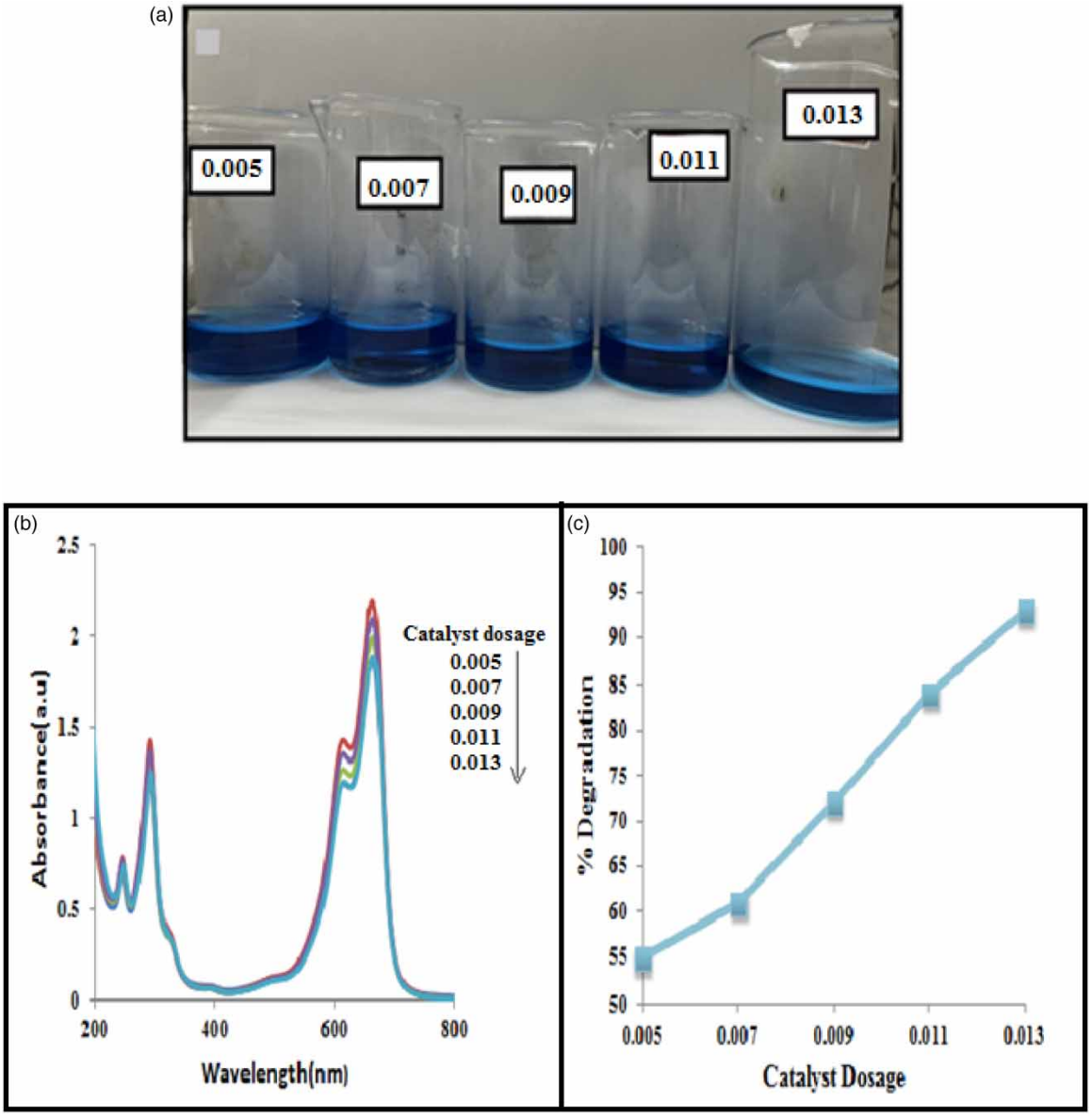


Figure 7 | Photographic images of different catalyst dosages dilution (a), UV–visible graph of MB dye degradation with different amounts of photocatalyst for 120 min (b), and percentage degradation of MB dye at 120 min (c).

Table 1 | Ratio of dye to CuO NPs

Photocatalyst dosages	Ratio of catalyst to dye	Ratio	% degradation
0.005	10:5	2.1	55
0.007	14:7		61
0.009	18:9		72
0.011	22:11		84
0.013	26:13		93

CONCLUSION

CuO NPs synthesized from D. palmatus fruit extracts demonstrated high photocatalytic activity in the degradation of MB organic dye. After 120 min, 90% of the dye had been destroyed. Longer irradiation times lead to increased dye degradation. Furthermore, the degradation of MB dye was improved by employing a larger amount of catalyst; with 93% degradation achieved using 0.013 g of photocatalyst over 120 min. Conversely, larger dye concentrations resulted in lower photocatalytic degradation activity. 95% of the dye was destroyed in just 120 min at pH 9, demonstrating that increasing the pH of the medium enhances organic dye degradation.

DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

CONFLICT OF INTEREST

The authors declare there is no conflict.

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