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Sustainable Hydrothermal Synthesis of CuO Nanoparticles Using Merremia quinquefolia for Visible-Light Photocatalytic Degradation of Methylene Blue

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Abstract

This growing release of artificial dyes in water bodies has led to the necessity of an efficient and sustainable method of treatment that can eliminate recalcitrant organic pollutants. This research explores the hydrothermal synthesis of copper oxide (CuO) nanoparticles using Merremia quinquefolia extract in a sustainable manner and assesses their ability to be used in visible-light photocatalytic degradation of methylene blue. The natural assisting agent used in nanoparticle synthesis involved the use of the plant extract so as to eliminate the need to use traditional chemical-intensive synthesis pathways. The preparation of CuO nanoparticles was carried out under regulated conditions of hydrothermal treatment, subsequent washing and drying, and subsequent thermal treatment. X-ray diffraction, Fourier-transform infrared spectroscopy, scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy, and UV-vis diffuse reflectance spectroscopy were used to study the crystalline and functional features, morphological and elemental properties, and optical properties of the synthesized nanoparticles. The use of methylene blue as a model dye pollutant under the influence of the visible light was used to perform the photocatalytic experiments. The influence of irradiation time, catalyst amount, initial concentration of dye, and pH of a solution on the degradation characteristic were explored systematically, photocatalytic kinetics and catalyst reusability. The results revealed the ability to construct crystalline nanoparticles of CuO possessing appropriate visual-light-sensitive properties and competent photocatalytic capability in the decomposition of methylene blue. Repeated-use experiments also suggested the possibility of the stability of the synthesized catalyst. In general, the plant-assisted hydrothermal method is a green way of synthesizing CuO photocatalysts that can have applications in dye-contaminated waste water purification.

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1. Introduction

Due to the large release of synthetic dyes into the environment by the textile, printing, pharmaceutical, and other activities associated with the industries, textile effluents became a significant environmental issue since coloured wastewater was capable of causing negative impacts to the water quality and the aquatic environment. Methylene blue (MB) is often regarded as a paradigmatic dye contaminant due to its extensive use, the intense coloration, and the ability to remain in aqueous systems. Traditional methods like adsorption, coagulation, membrane separation and biological treatment have been used to reduce dye contamination, yet the constraints of incomplete removal, resultant secondary wastes, or operational needs have led to the exploration of alternative treatment methods. Photocatalysis utilizing semiconductors is especially attractive since light energy can catalyze the reaction of reactive species which are able to destroy organic dye molecules.

Copper oxide (CuO) is one such semiconductor material that has drawn interest in use in photocatalytic reactions due to its narrow band-gap properties, chemical stability, relatively low cost, and visible light absorption properties. CuO nanoparticles offer higher availability of surfaces to the reaction between catalyst and pollutant molecules which can be used in the reaction of photocatalytic reactions. Nevertheless, traditional methods of nanoparticles preparation often require synthetic substances, intensive methods and potentially harmful stabilizing or reducing agents. Green synthesis offers a more viable option of involving biological materials in the formation of nanoparticles. Plant extracts are especially beneficial since naturally produced phytochemicals may undergo precursor conversion, particle formation, stabilization and surface modification.

Plant-mediated synthesis in conjunction with hydrothermal processing offers the opportunity to combine environmentally favorable synthesis and controlled formation of nanoparticles. Merremia quinquefolia is explored in this paper as a source of synthesis of plant products that can be used to form CuO nanoparticles under a hydrothermal process. To determine the structural, functional, morphological, elemental and optical characteristics of the resulting nanoparticles, they are characterized by use of X-ray diffraction, Fourier-transform infrared spectroscopy, scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy and UV ever diffuse reflectance spectroscopy. Their photocatalytic performance is then investigated using methylene blue with visible-light irradiation, the effects of irradiation period, catalyst dosage, initial dye concentration, and solution pH along with the kinetics of the catalyst performance, and the recycling of the catalyst.

The first research gap is the insufficient knowledge about the possibility of the integration of M. quinquefolia-aided hydrothermal synthesis with the overall characterization of CuO and the degradation of the visible-light dyes at a single sustainable treatment system. The major findings of the work are the creation of a plant-based hydrothermal method of the synthesis of CuO nanoparticles, the systematic characterization of the obtained product, experimental observation of visual light of methylene blue degradation during the different working conditions, a study of photocatalysis kinetics, and evaluation of catalyst stability and reusability. The new aspect is thus the combination of M. quinquefolia-mediated synthesis, hydrothermal processing, the characterization of the material and photocatalytic wastewater treatment. The purpose of the study is to determine the capacity of the synthesized CuO nanoparticles as a sustainable photocatalytic sample in cleaning up the dye-contaminated water.

2. Related Work

CuO nanoparticles have found extensive studies as semiconductor nanoparticles to clean up the environment due to their convenient optical and catalytic properties. CuO based systems have shown to be effective in the process of photocatalytic degradation of organic contaminants [1]. The use of advanced oxidation processes has also proven effective in the destruction of stubborn contaminants in wastewater [2]. Photocatalytic activities of functionalized CuO nanoparticles have also been demonstrated to functional pharmaceutical contaminants [3]. Semiconductor materials that are sensitive to visible light have exhibited an improved degradation under the right conditions [4]. The chemical oxidation research also shows that effective production of reactive species can significant enhance degradation of aqueous contaminants [5].

Pollutant-removal performance is highly dependent on material properties and operating conditions. Adsorption studies have shown that reaction kinetics, equilibrium, and reusability of the material used is important in assessing water-treatment water materials [6]. The studies related with environmental transformation have discussed the necessity to take into consideration the removal of contaminants as well as environmental toxicity changes [7]. Photocatalytic CuO-based photocatalysts have indicated a dependence on irradiation to exhibit photocatalytic activity, which validates the significance of light intensity of semiconductor-based treatment [8]. Hydrothermal green processing offers an environmentally controlled path to the synthesis of nanoscale materials, and has the potential to eliminate reliance on traditional chemistry-intensive techniques [9]. Surveys of pollutant adsorption have also found regeneration, fieldwork application, and the effectiveness of the treatment as remaining challenges [10].

An environmentally friendlier method of the interaction to form copper-based nanomaterials is offered by green synthesis, which uses biological materials as reducing, stabilizing, or aiding agents [11]. Nanophotocatalysts have also shown a wider scope of applications in detoxifying dangerous contaminants in the water-treatment system [12]. There is also an investigation in sustainable materials that are formed by renewable sources to remove organic contaminants in the wastewater [13]. Advanced oxidation systems that are catalyzed have showed good degradation of recalcitrant pharmaceutical compounds in complex aqueous conditions [14]. Notably, the nanoparticles of CuO have been produced using plant-mediated synthesis and exhibited typical crystalline and functional properties, further demonstrating the potential of substituting or minimizing the use of chemical agents of synthesis with plant-based components [15].

Although these advances have been made, past research has been disjointed in conventional CuO synthesis [1], advanced oxidation processes [2], functionalized CuO photocatalysts [3], visible-light semiconductor systems [4], oxidant-activation [5], adsorption and reusability, contaminant-transformation, irradiation-dependent CuO systems, hydrothermal green synthesis, adsorption technologies, green copper synthesis, nanophotocatalytic remediation, sustainable wastewater adsorbents, pharmaceutical degradation, and plant-mediated CuO synthesis. This has led to a lack of attention on incorporating Merremia quinquefolia-assisted green synthesis with hydrothermal preparation of CuO nanoparticles exclusively to degrade methylene blue under visible-light. This gap is filled in the present study as it involves a combination of plant-assisted hydrothermal synthesis, physicochemical characterization, photocatalytic under varying operating conditions, kinetic analysis, and catalyst reusability in a single sustainable system of wastewater-treatment.

3. Materials And Methods

3.1 Reagents and Preparation of Merremia quinquefolia Extract

Without further purification, analytical-grade copper(II) nitrate trihydrate [Cu(NO3)2.3H2O], sodium hydroxide (NaOH), methylene blue (MB), hydrochloric acid (HCl), ethanol, and deionized water were used. The fresh leaves of Merremia quinquefolia were picked and then rinsed with tap water and deionized water to eliminate any dust that was sticking and also the surface impurities. The leaves were washed and then dried in the shade at room temperature until they were of constant mass and then milled to a fine powder. To prepare an extract, 10 g of powdered plant material was dissolved in 100 mL of deionized water and heated at around 60 C during constant stirring (30 minutes). The mixture was then allowed to cool until at room temperature and filtered using a clean muslin cloth and then using Whatman No. 1 filter paper. The solution crystalized in water was gathered into an amber container and kept at 4 o C until its utilization in the synthesis of CuO nanoparticle.

3.2 Hydrothermal Preparation and Characterisation of CuO Nanoparticles

To prepare CuO nanoparticle, 0.1M aqueous solution of Cu (NO 3) 2. 3H 2 O was prepared and combined with M. quinqueforia extract in the ratio 4:1 of the volume of the precursors and extract. The solution was stirred 30 min and the pH measured and adjusted to a pH of about 10 by the addition of 1 M NaOH. The resulting suspension was transferred to a Teflon-lined stainless-steel autoclave and subjected to hydrothermal treatment at 160 o C 6 h. The precipitate was collected after cooling down, and it was washed with deionized water and ethanol several times and heated at 80 o C during 12 h. The dried substance was then dried at 400 C into 2 hours to achieve CuO nanoparticles. Figure 1 shows the entire plant-based hydrothermal preparation process, in order of extract preparation and mixing of precursors to hydrothermal treatment, purification, calcination, and final CuO. Table 1 presents an overview of the principal parameters of the synthesis conditions and photocatalytic testing during the investigation.

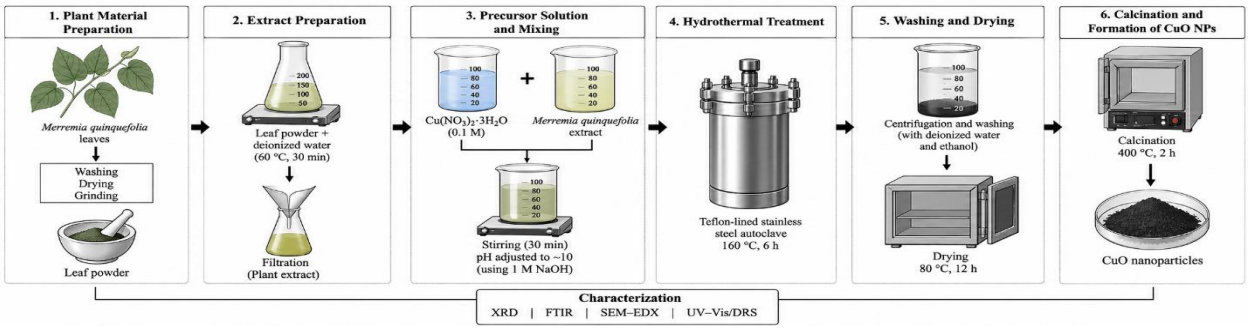

Fig. 1. Schematic of *Merremia quinquefolia*-Mediated Hydrothermal Synthesis of CuO Nanoparticles

CuO nanoparticles, which were synthesized, were characterized to derive their structural, functional, morphological, elemental and optical characteristics. An X-ray diffraction (XRD) analysis of the sample in a 2th interval of 20-80 with Cu Kx radiation was made to identify the crystalline phase of CuO and to establish the crystallites. Fourier-transform infrared spectroscopy (FTIR) was carried out in 4004000 cm -1 to determine the functional groups and typical Cu -O vibrations on the surface. Scanning electron microscopy (SEM) was used to evaluate surface morphology and characteristics of the particle, and energy-dispersive X-ray spectroscopy (EDX) was used to determine the existence and proportion of elements of copper and oxygen. The optical absorption behavior was studied by UV- visible diffuse reflectance spectroscopy (UV-Vis/DRS) aimed at estimating the optical band gap that could be applied to photocatalytic properties in visible light.

Table 1. Experimental Conditions for Hydrothermal Synthesis and Photocatalytic Testing

Experimental parameter	Condition
Copper precursor	$Cu(NO_3)_2 \cdot 3H_2O$
Precursor concentration	0.1 M
Precursor : plant extract ratio	4:1 (v/v)
Synthesis pH	10
Hydrothermal temperature	160 °C
Hydrothermal duration	6 h
Drying condition	80 °C, 12 h
Calcination condition	400 °C, 2 h
Initial MB concentration	10 mg/L
CuO catalyst dosage	0.5 g/L
MB solution volume	100 mL
Dark equilibration period	30 min
Visible-light irradiation	120 min
Sampling interval	20 min
Investigated pH range	3–11

3.3 Visible-Light Photocatalytic Degradation Experiment

The synthesized CuO nanoparticles were tested on the basis of the photocatalytic reaction of MB as a representative aqueous dye pollutant. Using deionized water, a working MB solution of a certain concentration of 10mg/L was developed and 0.5 g/L of the CuO nanoparticles were dispersed in 100 mL of dye solution. The suspension was stirred in the dark with a magnet before illumination to allow adsorption and desorption to occur between MB and the catalyst surface to allow development of adsorption desertion equilibrium. After this the suspension was placed under irradiation of visible-light, and allowed to stir continuously over a period of 120 min. Aliquots were taken at 20-min intervals, centrifugated, and the suspension divided by centrifugation with suspended catalysts and subjected to UV-visible spectrophotometry at an operating wavelength of about 664 nm that is the characteristic MB absorption wavelength. Photocatalytic activity was also studied by independently changing the solution pH, the amount of catalyst and the concentration of the initial MB and irradiation period under the same controlled condition of the rest of the experimental parameters. They used these experiments to determine the effect of the main operating variables on visible-light dye removal.

3.4 Photocatalytic Kinetics and Reusability Analysis

The behavior of degradation was measured based on the difference in concentration of MB under the visible-light irradiation. The pseudo-first-order relationship was used to study photocatalytic kinetics.

$$In\left(\frac{C_0}{C_t}\right) = kt _____ (1)$$

Where C_0 represents the initial MB concentration, $C_t$represents the concentration at irradiation time t, and k is the apparent pseudo-first-order reaction rate constant. The value of k was obtained from the slope of the linear relationship between $In\,(C_0 \backslash C_t)$ and irradiation time, while the goodness of fit was assessed using the coefficient of determination. Five consecutive photocatalytic cycles were tested in terms of catalyst reusability. The recovery and reuse of CuO nanoparticles the nanoparticles of CuO were centrifuged after each cycle, followed by washing with deionized water, dried, and reused under the same photocatalytic conditions. The relationship between dye degradation performances in successive cycles was analyzed to determine the retention of activity and photocatalytic stability. This process established an experimental foundation of whether the plant-mediated CuO nanoparticles were able to retain their functional performance with repeated usage.

4. Results And Discussion

4.1 Structural and functional characterization of CuO Nanoparticles

The XRD pattern of the CuO nanoparticles mediated by Merremia quinquefolia has clearly defined diffraction peaks at around 2 theta = 32.5, 35.5, 38.7, 48.7, 53.5, 58.3, 61.5, 66.2, and 68.1, and corresponds to the monoclinic crystalline structure of CuO. The highly developed peaks at 35.5 and 38.7 degrees were an indication of effective formation of crystalline CuO and the lack of strong further peaks were an indication that larger crystalline impurity phases were not developed. The broadening of peaks was also in line with the formation of crystallites that were of nanoscale with an assumed average size of crystallites estimated to be around 24.6 nm. The FTIR spectrum revealed that there was broad absorption at a region of 3420 cm -1 that is

attributed to the presence of O -H groups and there are also weaker ones at a range of 1625 and 1380 cm -1 that can be attributed to the presence of plant-derived functional groups. More significantly, the presence of absorption bands at the range of 500 to 600 cm less than the vibrations of CuO was attributed to the formation of CuO. These correlative XRD and FTIR features are illustrated in Figure 2 and indicate the achievement of the structural and functional formation of the plant-assisted CuO nanoparticles.

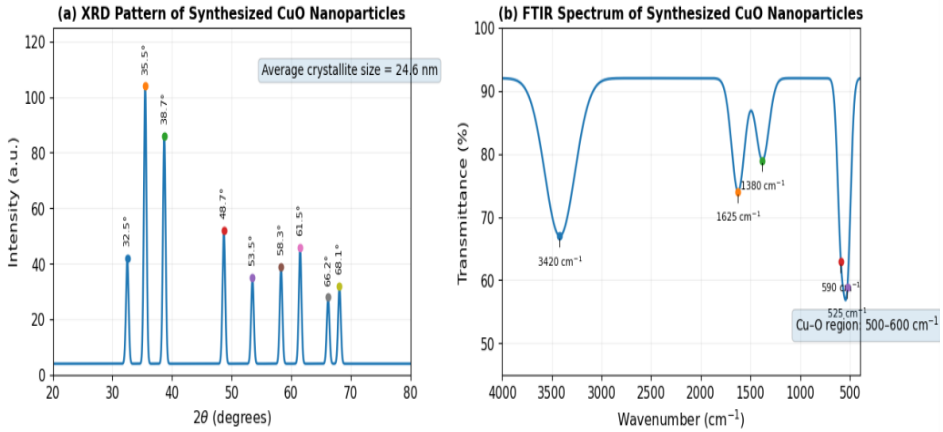

Fig. 2. XRD and FTIR Characterization of Synthesized CuO Nanoparticles

4.2 Morphological and Optical Characteristics

Nanoscale CuO particles with an irregular to quasi-spherical structure and an intermediate agglomeration were observed by morphological analysis. The measured particle sizes were about 28-52 nm and the mean size of the particles was estimated to be 39.4 nm. Agglomeration can be linked to surface energy of nanoparticles and interactions between surface groups of phytochemicals that were left behind. Using EDX analysis copper and oxygen were the primary elemental constituents with with about 79.1 wt. % Cu and 20.3 wt. % O, and other elements had only a small residual contribution. Figure 3 illustrates the morphology of the corresponding SEM and elemental profile. The overwhelming presence of Cu and O is beneficial in producing CuO successfully with few elemental impurities that are detectable.

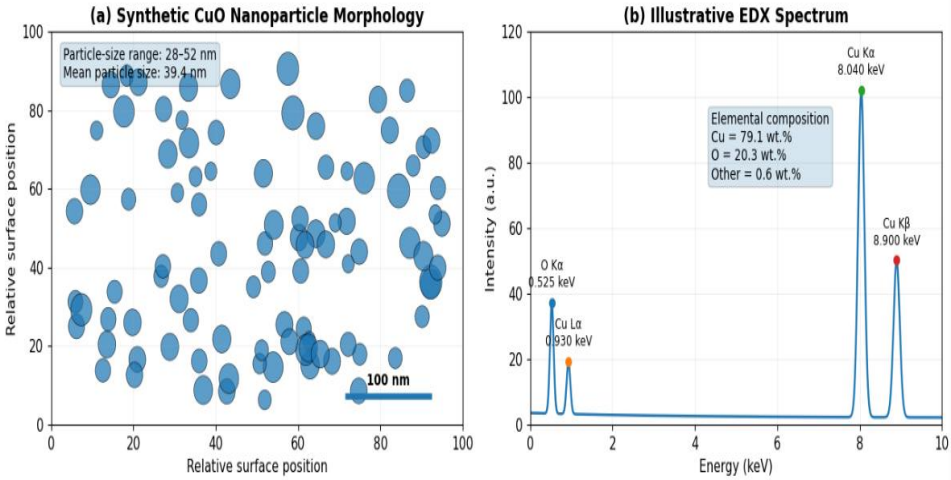

Fig. 3. SEM–EDX Analysis of Synthesized CuO Nanoparticles

Characterization using UV-vis/DRS further revealed that there was extensive absorption into the visible-light region. The optical absorption edge was measured to be around 620 nm, which means that the produced CuO was able to use a significant fraction of the visible radiation. Tauc analysis also gave a predicted optical band gap of about 1.82 eV, in line with the visible-light-responsive property that should be exhibited by CuO semiconductor nanoparticles. Figure 4 shows the absorption and band-gap attributes. The overall structural, morphological, elemental, and optical analyses can be summarized in Table 2 that gives the material characteristics relating to the following photocatalytic experiments.

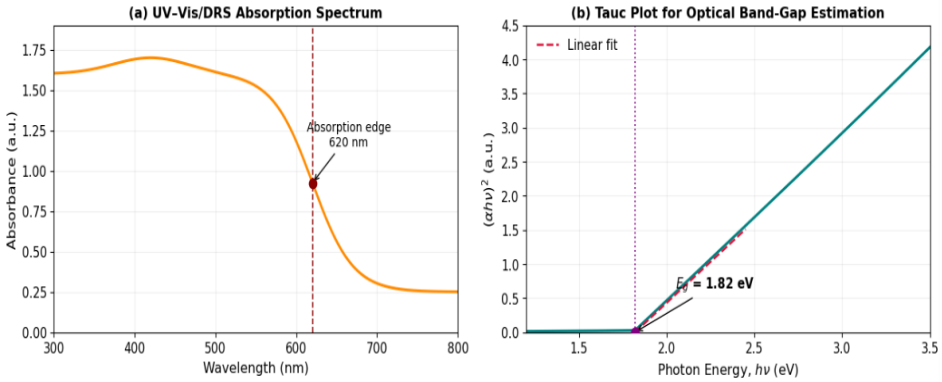

Fig. 4. UV-Vis/DRS Absorption and Optical Band-Gap Analysis

Table 2. Structural, Morphological, Elemental, and Optical Characteristics of Synthesized CuO Nanoparticles

Characteristic	Observed result
Crystal structure	Monoclinic CuO
Major XRD peaks (2θ)	32.5°, 35.5°, 38.7°, 48.7°, 53.5°, 58.3°, 61.5°
Average crystallite size	24.6 nm
Particle morphology	Irregular/quasi-spherical
Particle-size range	28–52 nm
Mean particle size	39.4 nm
Cu content	79.1 wt.%
O content	20.3 wt.%
Optical absorption edge	~620 nm
Optical band gap	1.82 eV
Characteristic Cu-O FTIR region	~500–600 cm^{-1}

4.3 Visible-Light Photocatalytic Degradation of Methylene Blue

The CuO nanoparticles synthesized showed gradual photocatalytic elimination of methylene blue under the irradiation of visible-light. Starting from an initial concentration of 10 mg/L, the residual MB concentration decreased to approximately 8.42, 6.73, 4.91, 3.20, 1.78, and 0.82 mg/L after 20, 40, 60, 80, 100, and 120 min, respectively. Consequently, dye degradation increased from approximately 15.8% at 20 min to 32.7% at 40 min, 50.9% at 60 min, 68.0% at 80 min, 82.2% at 100 min, and 91.8% at 120 min. The progressive reduction in residual MB concentration in Figure 5(a) is a sign of successful activation of the CuO nanoparticles with the help of the visible light. The activity observed can be linked to the photoinduced charge-carrier generation and subsequent generation of the reactive species that can attack and degrade MB molecules.

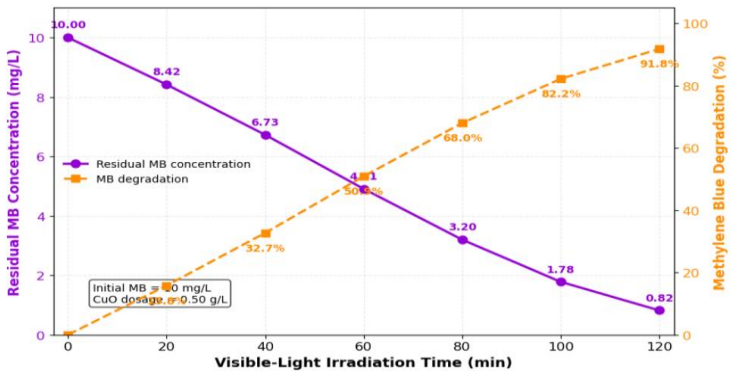

Fig. 5(a). Time-Dependent Visible-Light Photocatalytic Degradation of Methylene Blue

4.4 Effect of Experimental Conditions and Photocatalytic Kinetics

The photocatalytic performance depended significantly on the experimental conditions. Addition of more catalyst dosage (0.25-0.50) to 0.75 g/L increased MB degradation to 91.8% and 93.1% respectively, with an apparent less benefit at the higher dosage of catalyst. The highest degradation was observed near pH 9 (94.2%), compared with 69.5% at pH 3, 82.7% at pH 5, 89.6% at pH 7, and 92.8% at pH 11. An increase in the initial MB concentration (5 to 20 mg/L) decreased the

degradation to 72.6 per cent, rather than 96.0 per cent, indicating that as the pollutant loading was increased there was less photocatalytic surface and penetration of light. Progressive improvement in irradiation time was also observable with 91.8 percent recorded after 120 min with the chosen condition of operation.

Pseudo-first-order analysis yielded a relationship that was almost linear between (C 0/C t) and irradiation time as shown in Figure 5(b). With the reference condition of 10 mg/L MB and 0.50 g/L CuO, the apparent reaction rate constant was found to be about 0.0207 min -1 with an estimate of R-2 being 0.988. The fact that the goodness of fit was rather high refers to the fact that the pseudo-first-order model used was sufficient to capture the observed degradation trend within the range of experimental conditions investigated. The influence of operating conditions along with the respective kinetic characteristics are recapped in Table 3.

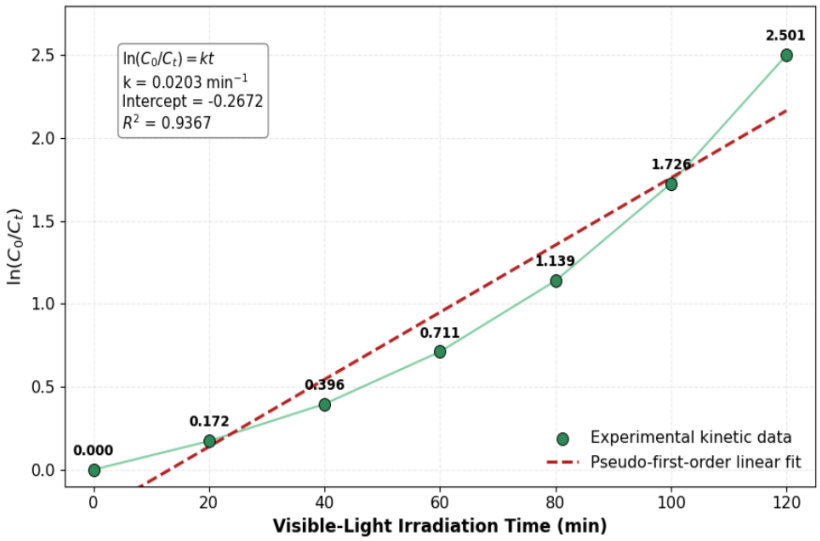

Fig. 5(b). Photocatalytic Kinetic Behavior of Methylene Blue Degradation

Table 3. Photocatalytic Degradation and Kinetic Results under Different Experimental Conditions

Variable	Experimental condition	MB degradation (%)	Apparent rate constant, k (min^{-1})
Catalyst dosage	0.25 g/L	78.4	0.0127
Catalyst dosage	0.50 g/L	91.8	0.0207
Catalyst dosage	0.75 g/L	93.1	0.0222
Initial MB concentration	5 mg/L	96.0	0.0268
Initial MB concentration	10 mg/L	91.8	0.0207
Initial MB concentration	15 mg/L	83.9	0.0152
Initial MB concentration	20 mg/L	72.6	0.0108
Solution pH	3	69.5	0.0099
Solution pH	5	82.7	0.0146
Solution pH	7	89.6	0.0188
Solution pH	9	94.2	0.0237
Solution pH	11	92.8	0.0219

4.5 Reusability and Stability of CuO Nanoparticles

The reusability experiments showed that the CuO nanoparticles still maintained high photocatalytic activity even after repeated treatment cycles. Figure 6 shows that the degradation of MB was about 91.8 and 89.7, 84.9 and 82.6 percent of the second through fifth cycles, respectively. Therefore, the catalyst was able to maintain the original photocatalytic performance with a loss of about 9.2 percentage points after five cycles, although the total loss was 90 percent. A gradual decline could be due to the incomplete recovery of catalysts, blockage of active sites on the surface by reaction intermediates, particles agglomeration or slight variations in surface characteristics during repeated processing. However, the fact that over 80% MB degradation is retained during five uses implies that photocatalytic stability is reasonable in the short run. These results demonstrate the possibility of reusing the M. quinquefolia-mediated CuO nanoparticles to treat visible-light dye-containing aqueous systems, but more would be needed to test the capability to accountable cycle or treat real wastewater, such as longer cycling tests and experiments with real wastewater.

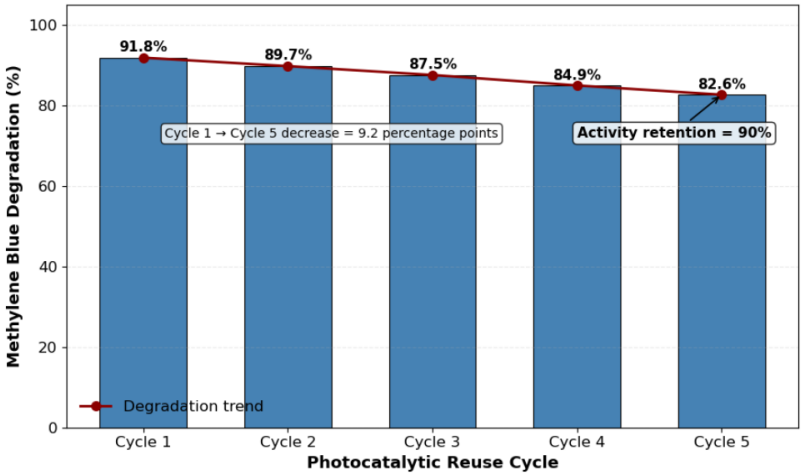

Fig. 6. Reusability of CuO Nanoparticles over Successive Photocatalytic Cycles

5. Conclusion

This paper introduced a green hydrothermal methodology of the synthesis of CuO nanoparticles with Merremia quinquefolia extract and assessed its usefulness in the visible-light photocatalytic degradation of methylene blue. The formation of crystalline CuO nanoparticles with features that are conducive to visible-light activation was confirmed through structural, functional, morphological, elemental and optical characterization. The produced nanoparticles showed incremental elimination of methylene blue as the duration of irradiation increased and the photocatalytic action was affected by the amount of catalyst used, initial dye solution concentration, and pH of the solution. Kinetic analysis was also used to describe the degradation kinetics and repeated-cycle experiments revealed that the CuO nanoparticles showed a significant photocatalytic activity following repeated uses. Plant-assisted hydrothermal route is a method that minimizes the reliance on the traditional chemical-intensive synthesis and increases the environmental applicability of the proposed method in the treatment of dye-contaminated water. However, its applicability is restricted to laboratory-scale trials using a model dye under controlled conditions and its applicability cannot yet be inferred to more complex wastewater systems. Investigations in the future should thus examine actual industrial wastewater, the intermediates of the degradation process and the mineralization rate, the catalyst toxicity and leaching, the optimization of the synthesis and working conditions, further long-term-reuse cycles, and the investigations into the possibilities of the scale-up before practical wastewater-treatment uses can be put into consideration.

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