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ORIGINAL ARTICLE

Photocatalytic and Anti Oxidant Properties of Carbon Quantum Dots Synthesized Using Microwave Assisted Method from Leaves of *Melochia corchorifolia*

K Pallavi[1&2] , B Mallikarjuna[2] , Abhinash Marukurti[4] ,R Lohitha Kavya[5] , R Ramesh Raju[3] , N Madhavi[1]*

1*Department of Chemistry, JKC College, Guntur
2Department of Chemistry, Government College (A), Rajahmundry
3Department of Chemistry, Acharya Nagarjuna University, Guntur
4School of Life and Health Sciences, Adikavi Nannaya University, Rajahmundry
5Central Instrumentation Laboratory (CIL), DST-FIST, Government College (A), Rajahmundry
*Corresponding Author's email: madhavijkcchempg@gmail.com

ABSTRACT

This study explores the synthesis and photocatalytic and antioxidant properties of carbon quantum dots (CQDs) derived from Melochia corchorifolia (MC) for the degradation of methylene blue (MB) and eosin yellow (EY) dyes. MC-CQDs were synthesized using a microwave-assisted method, followed by extensive characterization using UV-Vis spectroscopy, FTIR, fluorescence spectrophotometry, XRD, and HRSTEM. The synthesized MC-CQDs demonstrated significant antioxidant activity with a moderate DPPH free radical scavenging capacity. Photocatalytic degradation experiments were conducted using different concentrations of MC-CQDs (25-100 mg L-1) and dye solutions (10 µM and 20 µM) under 100 W light irradiation. The results indicated effective degradation of both dyes, with higher CQD concentrations enhancing the degradation rate. The degradation efficiency was time-dependent, with maximum degradation observed at 70-80 minutes for MB and 60-80 minutes for EY. The study highlights the potential of MC-CQDs as eco-friendly and efficient photocatalysts for dye degradation in wastewater treatment applications.

Keywords: Carbon Quantum Dots, Melochiacorchorifolia, Methylene Blue, Eosin Yellow, Photocatalytic degradation.

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INTRODUCTION

Carbon dots (CQDs) are quasi spherical sp^2 hybridized zero dimensional nanomaterials which have key properties such as photostability, multi-color fluorescence emission, hydrophilicity, chemical inertness and biocompatibility have attracted researchers to utilize them in various scientific fields such as drug delivery, photocatalysis, phototherapy, bioimaging, energy storage and biomedical applications [1]. The CDs can be synthesized using two strategies i.e; top-down and bottom-up strategy. The top-down is involved in breakdown of bulk precursors into small molecules using chemical, physical and electrochemical methods. The, Bottom-up approach (Hydrothermal, pyrolysis, solvo thermal and microwave assisted methods) is most commonly used method for the synthesis of CDs which involves chemical reactions that's drives small biomolecules into water soluble CDs [2].

Adverse number of chemical and natural precursor molecules have been used for the synthesis of CQDs using bottom-up approach. Many of the chemical precursors are toxic chemicals and requires surface passivation. Several studies have reported the synthesis of CQDs using various natural precursors which are renewable resources, high availability, low cost and eco-friendly such as plant parts, bio-waste, prawn shell and crab shell etc.[3]. A detailed mechanism of carbon dots formation is yet to be studied. However, previous literature reported hypothetical mechanism of formation of CDs, which involves three main steps

as follows: (i) Hydrolysis and Condensation, (ii) Self-polymerization/Aggregation and (iii) Transformation to core and surface passivation at higher temperature over a period of time [4].

Carbon quantum dots (CQDs) derived from plants have emerged as highly effective photocatalysts for the degradation of dyes such as methylene blue and eosin yellow. These CQDs are eco-friendly, cost-effective, and exhibit excellent optical properties and high surface area, enhancing their photocatalytic efficiency. The natural origin of plant-based CQDs ensures biocompatibility and minimal environmental impact, making them a sustainable alternative in wastewater treatment. Utilizing plant-derived CQDs leverages renewable resources and aligns with green chemistry principles. Degrading methylene blue and eosin yellow is essential as these dyes are toxic, carcinogenic, and persistent in aquatic environments, posing significant ecological and health risks [5-7].

Melochia corchorifolia, also referred to as chocolate weed, is a medicinal plant that possesses a diverse array of therapeutic characteristics. Historically employed in several cultural contexts, it demonstrates anti-inflammatory, antibacterial, and antioxidant properties. The plant's extracts have been utilized in the management of conditions such as pyrexia, gastric ulcers, and dermatological disorders [8]. *Melochia corchorifolia* is highly esteemed for its bioactive constituents, such as flavonoids and phenolics that enhance its medicinal effectiveness. This plant exhibits a wide distribution in tropical and subtropical environments, flourishing in many countries throughout Asia, Africa, and the Americas. It is frequently encountered in damp, low-lying regions and is frequently observed thriving as an unwanted plant in agricultural fields, roadsides, and areas of garbage. The resource's widespread availability and plentifulness make it easily available for local communities that depend on traditional medicine [9-10].

Despite the extensive literature survey to date, no study has been conducted on the synthesis of CQDs using *Melochia corchorifolia*. Thus, the aim of the present study is to synthesis the CQDs using *Melochia corchorifolia* and evaluate their photocatalytic properties on methylene blue and eosin yellow dyes.

MATERIAL AND METHODS

One-Pot Synthesis of Carbon quantum dots:

2 grams of pulverized powder of *Melochia corchorifolia* (MC) leaves was suspended in distilled water and subjected to stirring at 100^{o}C for 1hr to achieve homogeneous distribution. Then, the suspended solution was exposed to microwave irradiation for 10 mins at 640W power. The reaction mixture was cooled down to room temperature and filtered using Muslin cloth and Whatman No.1 filter paper followed by centrifugation to remove larger particles. The obtained supernatant was purified with 0.22 µ PVDF membrane syringe filters [11].

Characterization:

The optical properties such as absorbance, functional groups and photoluminescence properties of MC-CQDs were evaluated using UV-Visible spectroscopy (Shimadzu 2600), Fourier Transform Infrared Spectroscopy (FTIR) (BRUKER ALPHA II) and Fluorescence Spectrophotometer (PERKIN ELMER). The phase identification and morphological parameters of MC-CQDs were analyzed using X-ray Diffractometer (XRD) (BRUKER) and High-Resolution Scanning Transmission Electron Microscope (HRSTEM) (Thermo Scientific).

2,2-Diphenyl-1-picryhydrazyl (DPPH) free radical scavenging activity:

The DPPH free radical scavenging activity of MC-CQDs was conducted to determine their antioxidant property. Briefly, 3mM of DPPH was dissolved in methanol and various concentrations of MC-CQDs (10 to 50 µg mL^{-1}) were added to the DPPH reagent. The reaction mixtures were incubated at room temperature under dark conditions for 1 hr and centrifuged at 5000 rpm for 5 min. The absorbance of supernatant was recorded at 517 nm. DPPH with methanol was used as control and Ascorbic acid was used as standard. The percentage (%) of free radical scavenging activity and inhibitory concentration 50 (IC50) were calculated using equation 1 & 2 respectively.

$$\% \text{ of Activity} = \frac{Absorbance\ of\ the\ control - Absorbance\ of\ the\ sample}{Absorbance\ of\ the\ control} \times 100 \quad \text{Eqs..1.}$$

$$IC_{50} = Y = \frac{Max - Min}{1 + \left(\frac{X}{IC_{50}}\right)^{Hill\ coefficient}} \quad \text{Eqs..2.}$$

PHOTOCATALYTIC DEGRADATION:

In this study, photocatalytic degradation of methylene blue and eosin yellow dyes using *Melochia corchorifolia*-derived carbon quantum dots (MC-CQDs) was conducted. Two different concentrations of each dye, 10 µM and 20 µM, were prepared. The MC-CQDs were used in varying concentrations, ranging from 25 mg L^{-1} to 100 mg L^{-1}. Initially, the MC-CQDs were added to the respective dye solutions and

incubated in the dark for 30 minutes to achieve adsorption-desorption equilibrium. After incubation, the solutions were transferred to a photocatalysis chamber equipped with a 100 W light source. Samples of 2 mL were collected every 10 minutes during a total run time of 90 minutes. These samples were then centrifuged at 5000 rpm for 5 minutes to remove any particulate matter. The absorbance of the supernatant was measured using a UV-Vis spectrophotometer at wavelengths of 664 nm for methylene blue and 517 nm for eosin yellow to monitor the degradation process.

RESULT AND DISCUSSION

The microwave assisted synthesis of CQDs from plant biomass is a conventional bottom-up approach that relays on transfer of heat by conduction or convection [11] and mechanism involved in the formation of CQDs is still yet to revealed. Jiang et al proposed formation of fluorophores as intermediates between the precursor molecules and purified CQDs [12].

In the present study the CQDs were synthesized from leaves of *Melochia corchorifolia* were subjected to various characterization techniques to evaluate their morphological properties.

CHARACTERIZATION

The absorbance spectrum of MC-CQDs was recorded using UV-Vis spectroscopy from 200-800 nm wavelength. As depicted in Fig 1(a) the CQDs showed to consecutive absorbance peaks at 280 nm and 340 nm. The former represents the π-π* and ascribed to C=C bond transition and later represents the n-π* which is ascribed to C-C and C=C non-bonding electron transitions [13]. Various functional groups associated with MC-CQDs were identified using FTIR from 400-4500 cm^{-1} and found three vibrational signals at 3335, 2093 and 1637 cm^{-1} (Fig 1(b)). These vibrational signals are corresponding to strong O-H stretching/N-H stretching, weak stretching of C≡C and a strong C=O stretching respectively. The results are in strong correlation with hydrophilic nature of CQDs with surface functionalization of hydroxyl and carbonyl groups. Surface functional groups offers sustainable, ecofriendly CQDs with valuable photoluminescence properties and act as probes for biomedical research [14].

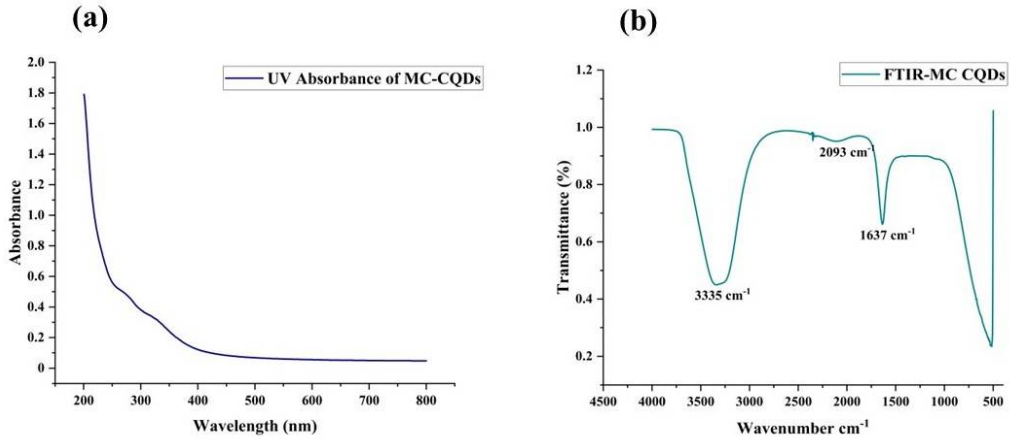

Figure 1: Optical Properties of MC-CQDs (a) UV-Vis absorbance spectrum and (b) Surface functional groups by FTIR.

The photoluminescence (PL) properties of MC-CQDs were identified using fluorescence spectrophotometer and the strongest emission was recorded at λ_{max} = 430 nm with an excitation wavelength of λ_{max} = 270 nm and represented in Fig 2. The photoluminescence properties of CQDs are depend upon the pyrophores of carbogenic center and size distribution [15].

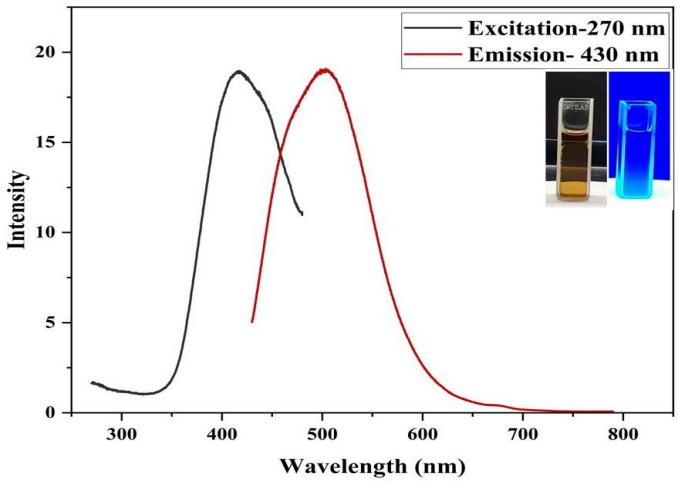

Figure 2: Photoluminescence spectrum of MC-CQDs. The inset represents the liquid CQDs at day light (left) and UV light (365 nm) (right).

As depicted in Fig 3(a), the X-ray diffractogram of MC-CQDs showed a characteristic intense peak at 2θ = 28.51° corresponding to lattice parameter (1 1 0). The amorphous nature of CQDs were confirmed and closely matches with the standard ICDD graphite carbon with card No. 01-089-8491. Other low intense peaks ascribed to surface functional groups of CQDs. The HRSTEM micrograph (Fig 3(b)) reveals the CQDs with quasi-spherical and irregular shapes and EDAX spectrum confirmed the presence of elements C, N, and O (Fig 3(c)). The appearance of Cu in EDAX spectrum is due to copper grid used to observed the HRSTEM micrographs. The Fig 3(d) clearly indicating the particle size diameter distribution between 1.5 nm to 5.5 nm and an average particle size diameter of 3.27 nm which confirms the formation of CQDs.

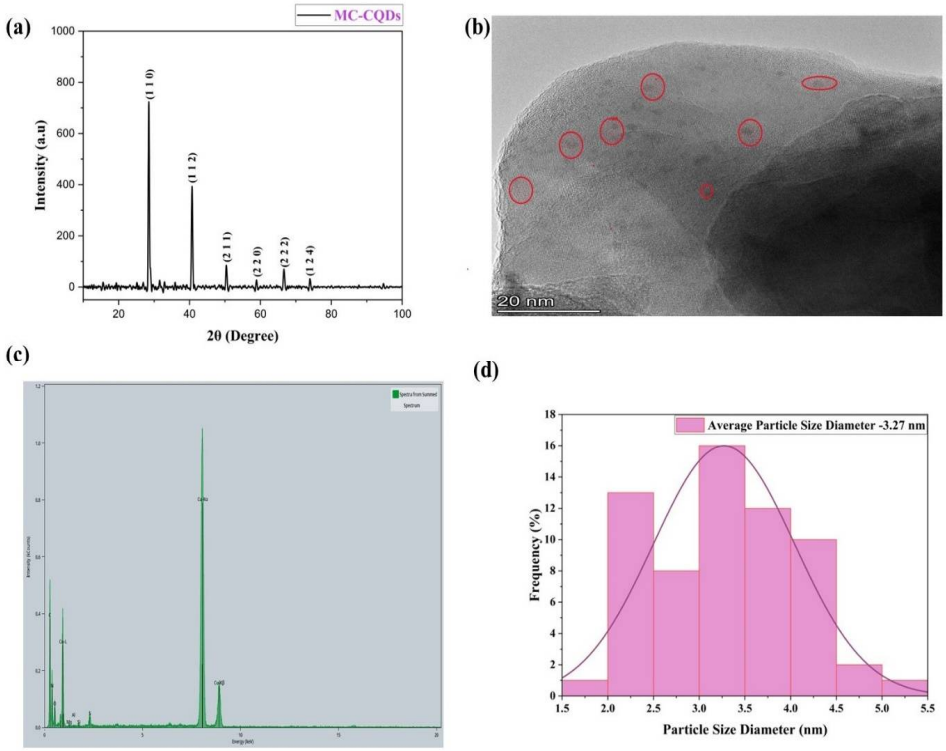

Figure 3: XRD and HRSTEM analysis of CQDs (a) X-ray diffraction pattern (b) HRSTEM micrograph (c) EDAX pattern and (d) Histogram of Particle size diameter distribution.

DPPH free radical scavenging activity:

The DPPH free radical scavenging activity of MC-CQDs revealed moderate activity when compared to the standard ascorbic acid. The % of activity was 7.38 to 29.84 % at 10 to 50 µg mL^{-1} (Fig 4) concentrations

and IC_{50} value was 186.65 µg mL^{-1} which indicates the amount of MC-CQDs required to eradicate the 50% of DPPH free radicals in 1mL of reaction mixture. The IC_{50} value of CDs derived from *Azadirachta indica* reported by *Gedda et al*13.87 µg mL^{-1}[16], which is lower concentration than the present study.

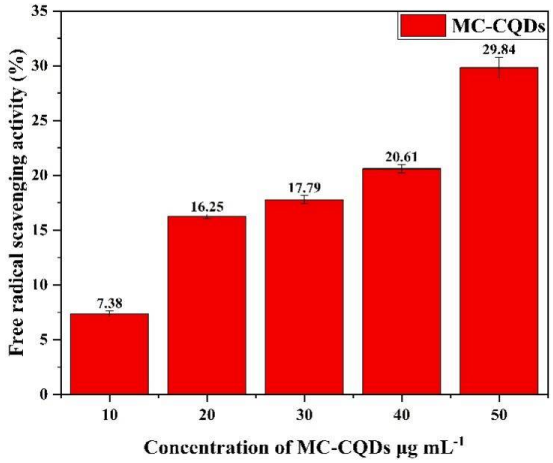

Figure 4: DPPH free radical scavenging activity of MC-CQDs

PHOTOCATALYTIC DEGRADATION OF MB AND EY

In the present study, two different concentrations of (10 µM and 20 µM) of MB and EY dyes were exposed to four different concentrations of MC-CQDs (25-100 mg L^{-1}) with light irradiation using 100 W tungsten bulb. The degradation efficiency was measured by observing the changes in absorption spectra of two dyes with regular time intervals (0 -80 min). The characteristic absorption band for MB and EY dyes can be observed at 664 nm and 517 nm respectively. The exposure of photocatalyst (MC-CQDs) to the light irradiation result in occurrence of various photochemical reactions on its surface ultimately leads to efficient dye degradation process. The Figures 5-8 represents the dye degradation efficiencies (%) of MC-CQDs at various concentrations. Table 1 represents the rate constants of MC-CQDs at each concentration for each dye concentration.

EFFECT OF TIME

The effect of time on the photocatalytic degradation of methylene blue (MB) and eosin yellow (EY) using MC-CQDs was significant. For methylene blue at a concentration of 10 µM, maximum degradation was achieved at 70 minutes, while at 20 µM, the maximum degradation was reached at 80 minutes. In the case of eosin yellow, at 10 µM concentration, maximum degradation occurred at 60 minutes, whereas for 20 µM, it took 80 minutes to achieve the highest degradation. This suggests that the degradation process is time-dependent, with longer durations leading to higher degradation rates for both dyes.

EFFECT OF DYE CONCENTRATION:

The degradation efficiency varied with different dye concentrations. For methylene blue at a 10 µM concentration, the percentage of degradation increased with higher concentrations of MC-CQDs, achieving 57.58%, 61.06%, 65.79%, and 71.09% at 25, 50, 75, and 100 mg L^{-1} respectively. For the 20 µM concentration of methylene blue, the degradation percentages were slightly lower, at 52.85%, 54.33%, 56.38%, and 57.29% for the same MC-CQD concentrations. Eosin yellow also showed similar trends: at 10 µM concentration, the degradation percentages were 50.19%, 51.01%, 55.01%, and 59.39%, while at 20 µM, the percentages were 47.66%, 48.94%, 50.19%, and 50.76% for increasing MC-CQD concentrations. This indicates that higher dye concentrations generally result in lower degradation efficiency, highlighting the importance of optimizing dye and catalyst concentrations for effective degradation.

EFFECT OF CATALYST CONCENTRATION

The effect of catalyst concentration on the rate constants of dye degradation was also evident. For methylene blue at a 10 µM concentration, the rate constants were 0.00996, 0.01059, 0.0115, and 0.01351 min^{-1} for MC-CQD concentrations of 25, 50, 75, and 100 mg L^{-1}, respectively. For the 20 µM concentration of methylene blue, the rate constants were slightly lower, at 0.00828, 0.00889, 0.00935, and 0.00969 min^{-1}. Eosin yellow showed different trends: at 10 µM concentration, the rate constants were 0.00719, 0.00592, 0.00621, and 0.00654 min^{-1}, whereas at 20 µM concentration, the values were 0.00825, 0.00621, 0.00635, and 0.00597 min^{-1}. These results suggest that increasing the catalyst concentration

generally enhances the degradation rate, although the effect can vary depending on the dye and its concentration.

CQDs derived from plant sources have demonstrated significant efficiency in the degradation of dyes, indicating their potential as effective photocatalysts. Recent studies have shown that plant-derived CQDs possess high quantum yield and stability, making them suitable for environmental applications. For instance, [17,18] reported the high efficiency of plant-based CQDs in degrading organic dyes due to their excellent light absorption properties and high surface area. The mechanism of photocatalytic degradation using CQDs involves the absorption of light, leading to the generation of electron-hole pairs. These pairs interact with water and oxygen molecules to produce reactive oxygen species (ROS) such as hydroxyl radicals (•OH) and superoxide anions (O2•−), which then degrade the dye molecules into less harmful substances. This mechanism has been well-documented in recent literature, including studies by [19,20] highlighting the effectiveness of CQDs in photocatalytic processes. Overall, plant-derived CQDs show promise as sustainable and efficient photocatalysts for environmental remediation, especially in the degradation of harmful dyes from wastewater. Their high degradation efficiency, influenced by factors such as dye concentration, catalyst concentration, and exposure time, underscores their potential for practical applications in pollutant degradation.

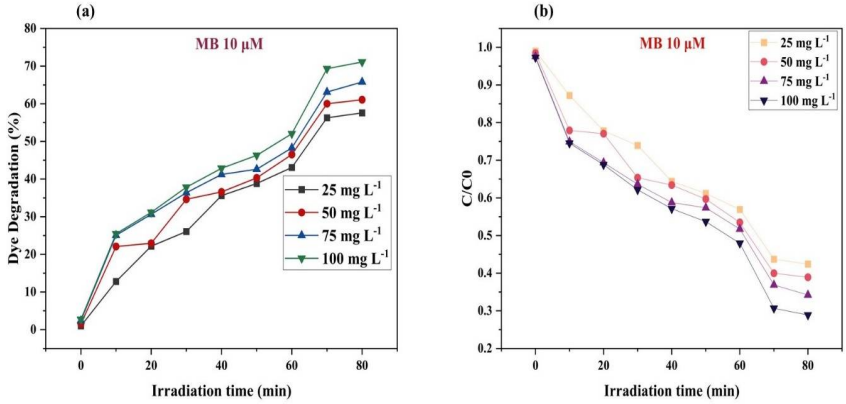

Figure 5: Photocatalytic degradation of Methylene blue (10 µM) (a) Dye degradation (%) at various concentrations of MC-CQDs and (b) C/C0 plot.

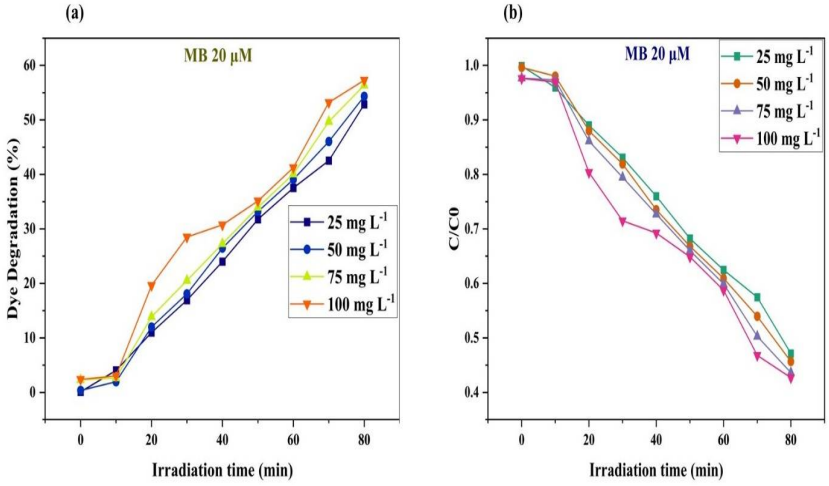

Figure 6: Photocatalytic degradation of Methylene blue (20 µM) (a) Dye degradation (%) at various concentrations of MC-CQDs and (b) C/C0 plot.

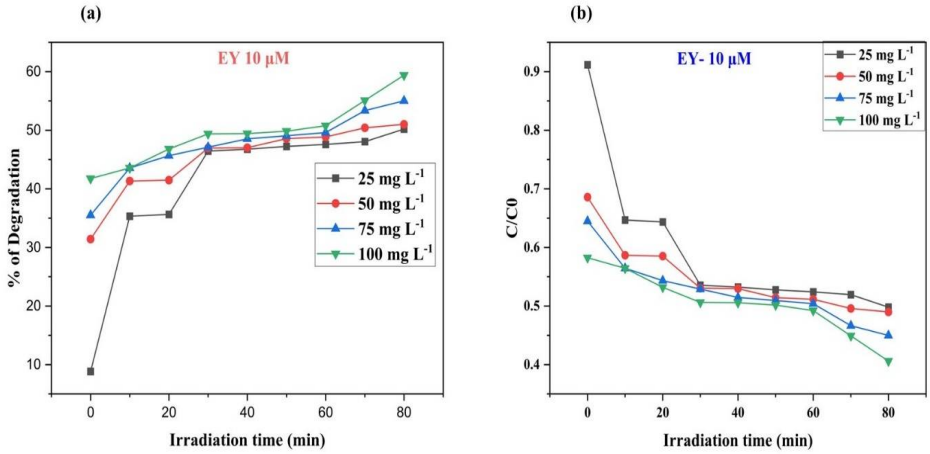

Figure 7: Photocatalytic degradation of Eosin Yellow (10 µM) (a) Dye degradation (%) at various concentrations of MC-CQDs and (b) C/C0 plot.

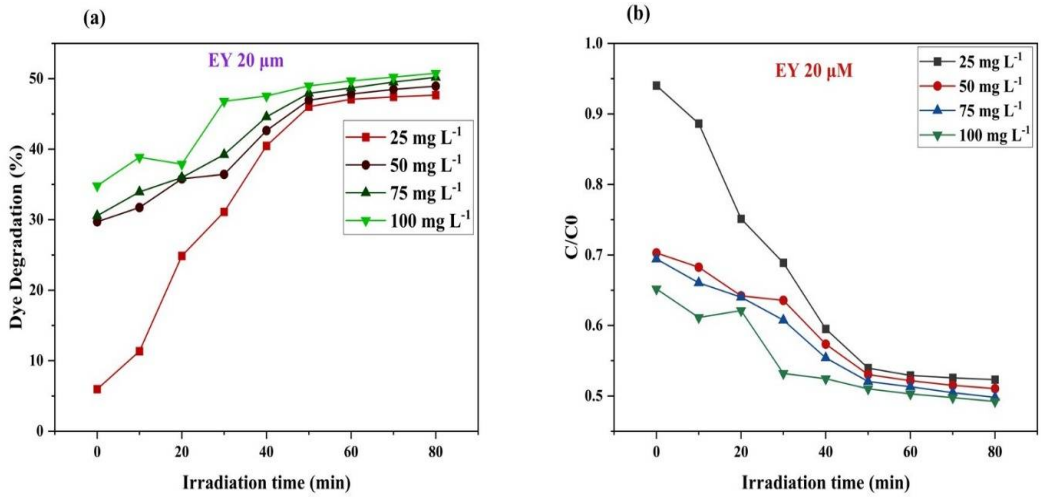

Figure 8: Photocatalytic degradation of Eosin Yellow (20 µM) (a) Dye degradation (%) at various concentrations of MC-CQDs and (b) C/C0 plot.

Table 1: Rate constant of MC-CQDs at various concentrations with different concentrations of dyes.

Concentration of MC-CQDs	Rate Constant min^{-1}			
	MB 10 µM	MB 20 µM	EY 10 µM	EY 20 µM
25 mg L^{-1}	0.00996	0.00828	0.00719	0.00825
50 mg L^{-1}	0.01059	0.00889	0.00592	0.00621
75 mg L^{-1}	0.0115	0.00935	0.00621	0.00635
100 mg L^{-1}	0.01351	0.00969	0.00654	0.00597

CONCLUSION

This research demonstrates the successful synthesis of *Melochia corchorifolia*-derived carbon quantum dots (MC-CQDs) and their effective application in the photocatalytic degradation of methylene blue and eosin yellow dyes. The MC-CQDs showcased excellent optical properties, structural integrity, and significant antioxidant activity, contributing to their high photocatalytic efficiency. The degradation efficiency was influenced by the concentration of CQDs, dye concentration, and exposure time, with optimal results achieved at higher CQD concentrations and longer irradiation periods. This study underscores the potential of using plant-derived CQDs as sustainable and cost-effective photocatalysts for environmental remediation. Future research should focus on elucidating the detailed mechanisms underlying the formation and photocatalytic activity of MC-CQDs. Additionally, exploring the application of these CQDs in the degradation of other organic pollutants and their potential for large-scale wastewater treatment is crucial. Investigating the reusability and long-term stability of MC-CQDs will also

be essential to establish their practicality in real-world applications. Integrating MC-CQDs with other advanced materials to enhance their photocatalytic performance and developing novel synthesis methods to further optimize their properties are promising directions for future studies. This research paves the way for innovative and eco-friendly solutions to address environmental pollution using plant-derived nanomaterials.

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