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# Sustainable Carbon Dots Production from Oil Palm Empty Fruit Bunch for the Photodegradation of Methylene Blue

**Wan Nuraishah Wan Ishak[1], Huey Ling Tan[1]*, Noor Fitrah Abu Bakar[1], Ying Pei Lim[1]**

[1] *School of Chemical Engineering, College of Engineering*
*Universiti Teknologi MARA, Shah Alam, Selangor, 40450, MALAYSIA*

*Corresponding Author: hueyling@uitm.edu.my
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**Abstract**

This paper presents a sustainable approach to synthesizing carbon dots (CDs) from biomass waste for the effective photocatalytic degradation of Methylene Blue (MB). CDs were successfully synthesized from Empty Fruit Bunch (EFB) via a facile one-step hydrothermal treatment. The CDs exhibited spherical morphology with both amorphous and crystalline structures, characterized by XRD analysis. SEM-EDX analysis indicated the presence of carbon, oxygen, and potassium. FTIR spectroscopy identified the presence of hydroxyl, aromatic, carbonyl, and C-C functional groups. UV-vis spectroscopy revealed characteristic absorption bands in the UV region from 230 to 340 nm, while the solution exhibited a dark brown color under ambient light and green luminescence under UV excitation (365nm). The optimal degradation efficiency of 68.5% was achieved in 160 minutes at pH 10 with 20 mg/L CDs in a 10 mg/L MB solution. Kinetic analysis indicated that the degradation followed pseudo-first-order kinetics with a rate constant of $1.5 \times 10^{-3}$ $min^{-1}$. This research demonstrates a simple and sustainable method for synthesizing spherical, amorphous, and crystalline CDs from biomass waste, showcasing their potential in environmental remediation.

## 1. Introduction

Water is necessary for human survival and is abundant in the natural world. However, environmental pollution caused by industrial activities, due to inappropriate wastewater disposal has become a worldwide issue of increasing concern (Rodríguez-Padrón et al., 2020). A large amount of dye effluent has continuously been discharged into the environment in recent years owing to the fast expansion of dye-related sectors such as textile, cosmetics, food, and dyes (Wang et al., 2022). Methylene Blue (MB) is a common dye for clothing and many other applications. Exposure to MB even in low concentrations of 10 ppm can be detrimental to the well-being of humans and aquatic organisms (Ardila-Leal et al., 2021; Das et al., 2019). Therefore, this research is vital in the development of cost-effective approaches for the degradation and removal of MB and other dyes from water effluents as well as to restore and revitalize the environment (Sawalha et al., 2021).

Biomass waste refers to a diverse, biodegradable, and organic material, that can be derived from multiple resources such as perennial grasses, organic household waste, agriculture residue, and by-products from associated industries such as wood and paper industries (Khairol Anuar et al., 2021; Sharma et al., 2019). Moreover, biomass waste is a renewable and environmental friendly carbon source that is abundantly available for the production of carbon dots (CDs) (Issa et al., 2019; Khairol Anuar et al., 2021). However, a significant amount of biomass waste is currently neglected, either being dumped in landfills or openly burnt which leads to

a loss of resources raises environmental concerns and compromising human health (Wang et al., 2020). Thus, it is of utmost importance to identify and develop technologies that can transform biomass waste into emergent valuable products.

CDs have recently become an interesting topic because they can effectively eliminate dyes as photocatalysts (Khairol Anuar et al., 2021; Khan et al., 2024). CDs are also well-suited for various uses, including cell imaging, cancer treatment, and metal sensing (Khairol Anuar et al., 2021). Carbon-based biomass products that store and convert energy are popular but the utilization of biomass-carbon photocatalysts for pollution photodegradation is still rare due to performance challenges in terms of their photocatalytic efficiency and stability compared to conventional photocatalysts (Akbar et al., 2021; Shi et al., 2025). Furthermore, CDs have attracted a lot of interest due to their non-toxicity, abundant, low cost, small size, and biocompatibility, which enables a wide range of applications in fields like optoelectronics, bioimaging, and sensing (Pudza et al., 2020; Sheikh et al., 2024).

Photocatalysis is a cost-effective, environmentally responsible, and friendly technology (Krishnakumar et al., 2017; Sheikh et al., 2024). It is a chemical process that occurs on the surface of a substance (photocatalyst) and is triggered by light (Abd El Khalk et al., 2021; Yang et al., 2024). The creation of "green" carbon dots is gaining popularity because biomass waste can be utilized as a "carbon-rich precursor" to circumvent the costs associated with waste disposal and raw materials (Liu et al., 2018; M. Yang et al., 2024). In nanoparticles synthesis, the bottom-up method is more financially feasible and consumes less energy. Several research groups have successfully developed hydrophilic CDs formulations made from natural materials such as bananas, candle soot, orange juice, wastepaper, and crushed soybeans (Liu et al., 2020). However, some research show that CDs have some difficulties in physiochemical, morphology, and formulation (Liu et al., 2018; Malitha et al., 2024). Despite these drawbacks, researchers (Mahat & Shamsudin, 2020; Sawalha et al., 2021) prefer the hydrothermal process for producing CDs because it can be modified to be more practical and ecologically friendly. Unfortunately, the study on the synthesis of photocatalysts for the photodegradation of pollutants based on biomass carbon is still in its early stages, with only a few examples reported (Issa et al., 2019; Khairol Anuar et al., 2021). This research aims to comprehensively analyze the CDs synthesized from EFB, a cheap and abundant precursor, as photocatalysts in removing pollutants such as MB from water. Factors affecting the MB photodegradation process such as concentration of MB, pH of MB, the dosage of photocatalyst (CDs), and contact time of the reaction under visible light irradiation using CDs photocatalyst were carefully studied in this research.

## 2. Methodology

### 2.1 Chemicals and Materials

Empty fruit bunch (EFB) was collected locally from an oil palm estate in Pendang, Kedah. All chemicals and reagents, such as Sodium Hydroxide (NaOH), Hydrochloric acid (HCl), and Methylene Blue (MB), were purchased from Sigma-Aldrich and used without further purification. HmbG nylon micro filter 0.22µm was used to filter both the CDs and MB solutions. This filtration process aimed to obtain smaller particles of CDs and to prevent contamination of MB solutions with the photocatalyst. Subsequently, the filtered solutions were analysed using a UV-Vis spectrophotometer.

### 2.2 Synthesis of Carbon Dots

Initially, the EFB was washed four times under running water to remove impurities. Then, it was dried under sunlight for a week to remove all moisture before grinding. A grinding machine with a sieve of 0.25 mm was used to cut the EFB into smaller pieces before mixed with deionized water. CDs was synthesized using a one-step hydrothermal process according to previous studies (Egorova et al., 2018; Mahat & Shamsudin, 2020). Briefly, 6g of EFB powder was mixed with 150 ml of deionized water into a Teflon-lined autoclave reactor. The hydrothermal reactor was heated at 200°C for 3 hours. The reaction vessel was let to cool at room temperature and the brown solution was centrifuged at 10000 rpm for 15 minutes. The resulting supernatant was then filtered through a nylon filter (0.22 µm) to remove precipitates and large particles. The final solution was freeze-dried for about a week and the drying was continued using an oven at 70°C overnight.

### 2.3 Photocatalytic Methods

Fig. 1 shows the photocatalytic experiment setup used in this work. In a photocatalysis experiment, 200 ml of 10 ppm MB and 40 mg/L of CDs was added into a 250 ml beaker, and the reaction was allowed for 160 minutes. Prior to the photodegradation experiment, the MB solution was stirred in dark conditions for 40 minutes to establish adsorption-desorption equilibrium and examine the interaction between CDs and MB solution as an adsorbent. After reaching equilibrium, 4 ml of aliquot was withdrawn to determine the initial concentration ($C_0$) before a 70 W LED bulb ($\gamma$ = 400 nm) was used to illuminate the solution. An aliquot of MB solution was

collected every 20 minutes to quantify the residual MB concentration at 663 nm using a UV-Vis spectrophotometer (Agilent Cary 60 Spectrophotometer). The same irradiation procedure was followed consistently for all experiments to evaluate the effect of different parameters such as photocatalyst dosage (5-50 mg/L), MB concentration (2-10 ppm), and pH (2-10) on photodegradation. A blank MB solution without photocatalyst was used to establish a baseline for comparison. The degradation efficiency was calculated using Eq. (1) (Hak et al., 2020): where $C_0$ is the initial concentration of MB while $C_t$ is the final concentration of MB.

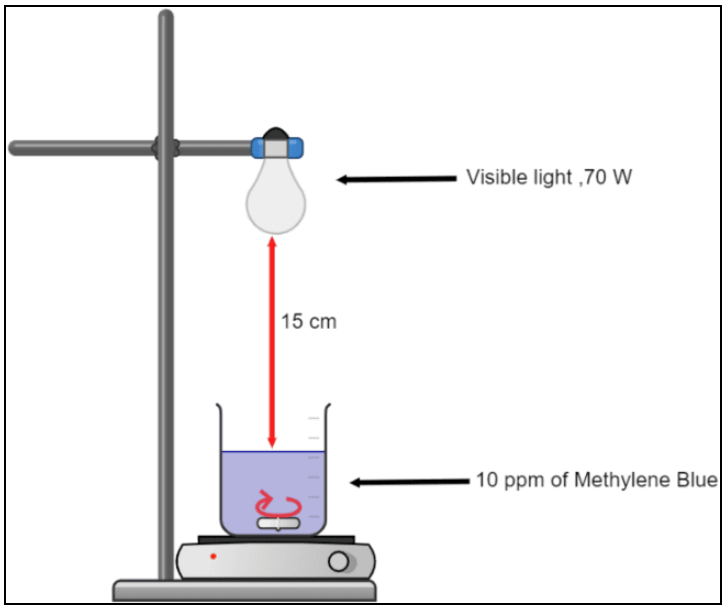



**Fig. 1** *Schematic illustration of the experimental setup for photocatalysis study under visible light using a 70 W LED at the wavelength of 400 nm*

$$\text{Degradation efficiency} = \frac{C_o - C_t}{C_o} \times 100\% \quad (1)$$

## 3. Results and Discussion

### 3.1 Fourier Transform Infrared Spectroscopy (FT-IR)

FT-IR analysis was performed to detect the functional groups presence on the surface of the CDs. The FT-IR spectrum in Fig. 2 shows a prominent absorption peak at 3332 $cm^{-1}$, which is attributed to the O–H stretching vibration of alcohols, phenols, and carboxylic acids as in cellulose, and lignin. This peak confirms the presence of ''free'' hydroxyl on the CDs and was consistent with the study by Jamaludin et al. (2020). Other peaks showed at 2151 $cm^{-1}$, 1638 $cm^{-1}$, and 1538 $cm^{-1}$ were associated with the presence of C=C (aromatic component), C=O (carbonyl), and C-C bonds, respectively. These findings aligned with the previous report that was conducted by Karaca et al. (2023). The results validate that CDs have good water solubility properties without the need for additional chemical treatments.

### 3.2 X-ray Diffraction Analysis (XRD)

XRD was used to assess the crystallinity of the CDs and the results are depicted in Fig. 3. The XRD pattern exhibits strong and sharp diffraction peaks of 2θ value at 26°, which can be attributed to the amorphous CDs that correspond to the (002) hkl plane (JCPDS card no. 26-1076). The peak indicates the presence of an amorphous framework that is randomly built by carbon rings and is useful in the production of a well-defined adsorbent (Liu et al., 2020). In addition, there are three sharp diffraction peaks at 43°, 50.14°, and 58.62°, which indicate the presence of crystalline nature and disordered carbon atoms such as stacked carbon rings (Zulfajri et al., 2021). All the preceding data suggest that hydrothermal transformation of microcrystalline cellulose resulted in the production of CDs. The carbon core structures of these CDs were found to be similar to that of graphite produced by Liu et al., (2020).

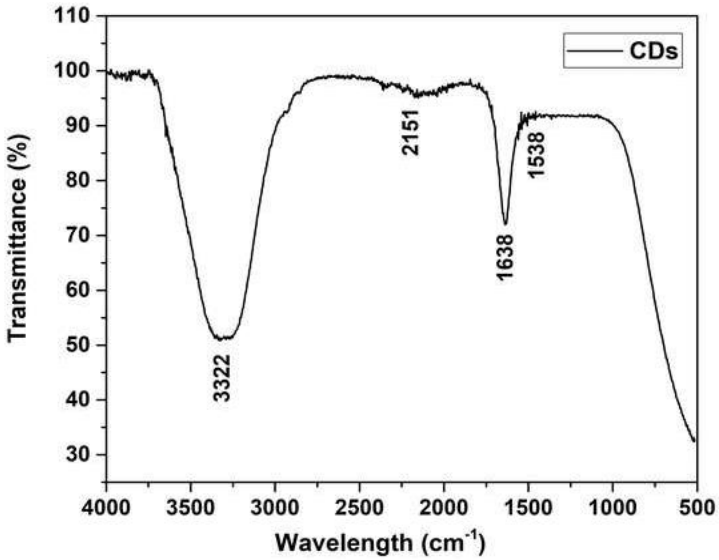



**Fig. 2** *FT-IR spectrum of CDs derived from EFB using hydrothermal method*

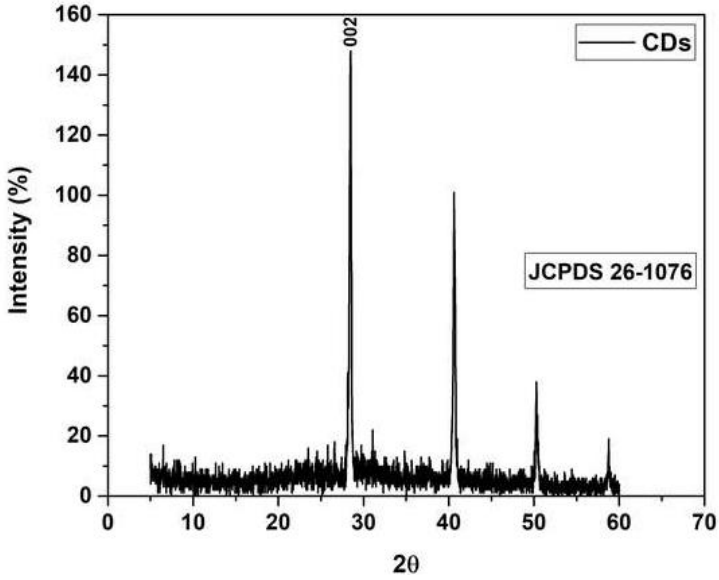



**Fig. 3** *XRD for CDs modified photocatalysts at different peaks to identify the amorphous or crystalline CDs*

## 3.3 Scanning Electron Microscopy (SEM) with Energy Dispersive X-Ray Analysis (SEM-EDX)

SEM-EDX analysis was carried out to confirm the presence of elements, including oxygen (O), carbon (C), and potassium (K), in the CDs. Table 1 shows that the CDs primarily consist of carbon (30.4%) and oxygen (46.1%), which is consistent with previous findings (Baloo et al., 2021). Additionally, trace elements such as chlorine (Cl), silicon (Si), sodium (Na), calcium (Ca), phosphorus (P), sulfur (S), and phosphate (P) were detected, which are typical of elements found in plants (Ozyurt et al., 2023). The low amount of carbon could be attributed to the low yield percentage typically found in carbon dots, which is around 5.6%.

**Table 1** *Elemental composition of CDs using SEM EDX*

| | Element composition of CDs | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | C | O | K | Na | P | S | Ca | Cl | Si |
| Weight, % | 18.5 | 37.4 | 32.4 | 0.7 | 1.2 | 1.2 | 0.8 | 5.1 | 1.8 |
| Atomic, % | 30.4 | 46.1 | 16.3 | 0.6 | 1.2 | 1.2 | 0.4 | 2.8 | 1.2 |

J. of Sustainable Materials Processing and Management Vol. 4 No. 2 (2024) p. 31-41

## 3.4 UV-Vis Spectrometer Analysis

Ultraviolet-visible (UV-vis) spectroscopy was utilized to examine the linearity of the optical absorption behavior of the CDs, and the corresponding spectrum is presented in Fig. 4. The CDs displayed characteristic absorption bands ranging from 230 to 340 nm, with a tail extending into the visible region. As reported by Mahat & Shamsudin (2020), the maximum peak observed at 294 nm is attributed to the π-π transition of aromatic C-C and C=C bonds, while the peak at 300 nm corresponds to the n-π transition of C=O bonds. These findings, illustrated in Fig. 4(a) and (b), further support the identification of specific optical transitions within the CDs. Furthermore, it is worth noting that the CDs solution exhibits a dark brownish color when exposed to natural light, as depicted in Fig. 4(c)(ii), which is consistent with the observations by Sawalha et al. (2021). Meanwhile, under a UV light source with a wavelength of 365 nm, the solution emits a green luminescence, as shown in Fig. 4 (c)(i).

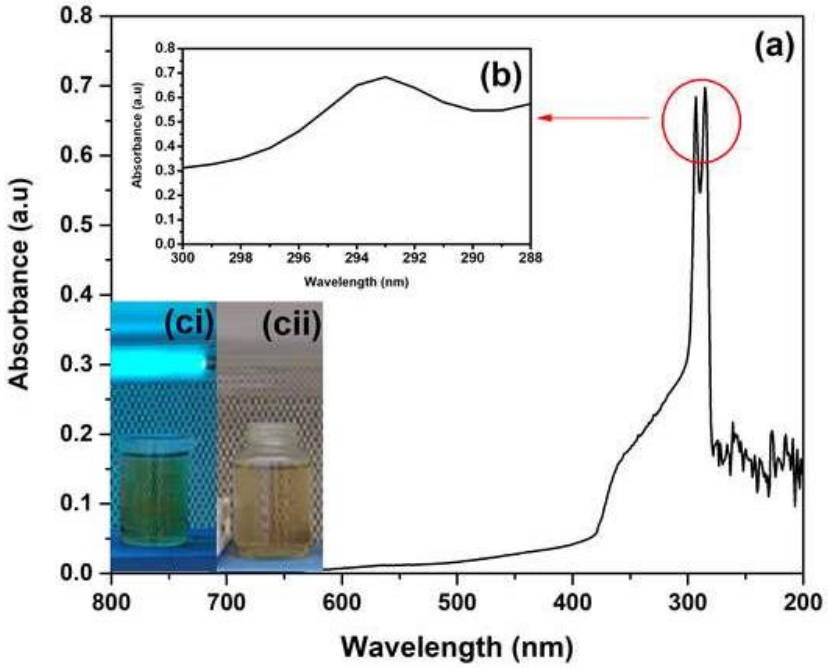
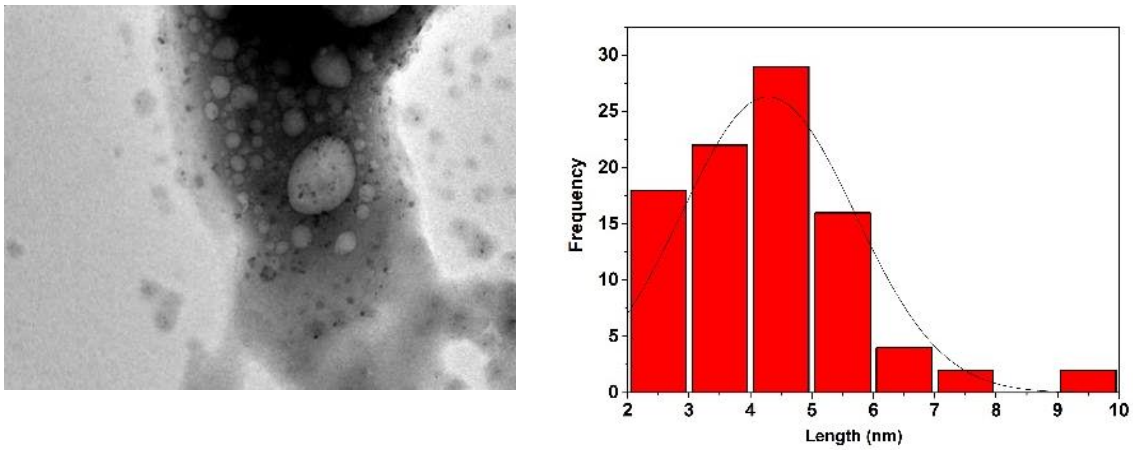



**Fig. 4** *(a), (b), UV-vis absorption spectrum of CDs at wavelength (200 – 400 nm) and (c) CDs solutions under (i) UV lamp with γ = 365 nm and (ii) natural light*

## 3.5 Transmission Electron Microscopy (TEM) Analysis

TEM was employed to investigate the morphological structure of the CDs, as depicted in Fig. 5. The analysis revealed that the pure CDs exhibited a carbonous carbon composition with spherical-shaped particles with size less than 10 nm, which is consistent with the findings reported in the literature by Liu et al. (2021).




**(a)** **(b)**

**Fig. 5** *(a)TEM images and (b) Particle distribution of CDs*

## 3.6 Photodegradation of Methylene Blue

### 3.6.1 Effects of Photocatalyst Dosage

Fig. 6 depicts the effect of CDs concentrations on MB elimination. The solutions were left in the dark for 40 minutes to enhance the adsorption-desorption equilibrium. Negligible reduction in MB concentration indicated that MB did not undergo self-degradation. From Fig.6, it is evident that CDs serve as effective photocatalysts and perform well under visible light. Increasing the photocatalyst dosage from 5 to 50 mg/L enhanced the MB removal, and the optimal CDs dosage of 20 mg/L achieved the highest degradation efficiency of 30.32%. This is due to the morphological characteristics of the CDs observed through TEM analysis. The TEM images reveal that the CDs possess a spherical shape with sizes less than 10 nm, which implies a high surface area-to-volume ratio. This characteristic is advantageous for photocatalytic applications because a higher surface area relative to volume enhances the availability of active sites for the photocatalytic reaction (Heng et al., 2020). The increased surface area allows for better interaction with the methylene blue (MB) molecules and more efficient light absorption, contributing to the observed enhanced degradation efficiency (Atchudan et al., 2020). Thus, the small, spherical morphology of the CDs plays a crucial role in their effectiveness as photocatalysts. As the CDs dosage increased, the number of photodegradation sites (surface area) also increased, leading to an elevated photodegradation rate (Liu et al., 2020). Remarkably, CDs show promise as catalysts capable of breaking down MB even at low concentrations. Furthermore, no degradation of MB occurred in the dark due to the complex carbon bonding in MB. The presence of heterocyclic aromatic chemical compounds in MB, which includes three interconnected aromatic rings with functional groups such as (C-S), (CN), and (CN = --), contributes to the difficulty in the adsorption process between CDs and MB (Atchudan et al., 2020; Heng et al., 2020).

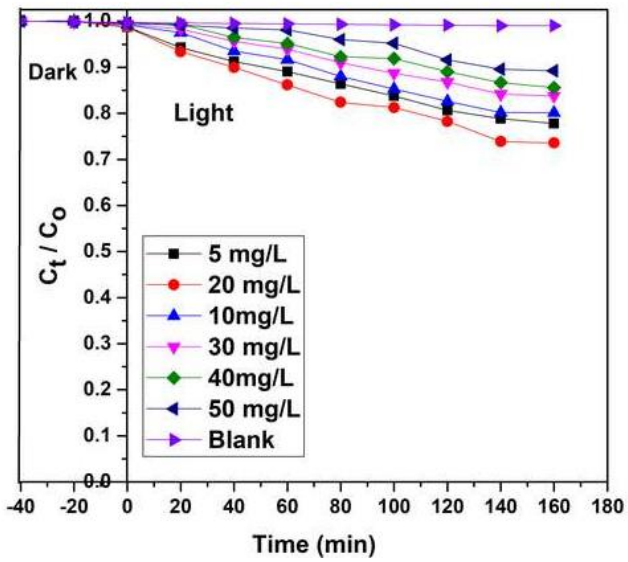



**Fig. 6** *Effects of dosage on photodegradation by CDs at pH=6 and 10ppm of MB under visible light*

### 3.6.2 Effects of MB Concentration

Fig. 7 depicts the CDs removal rate as a function of MB concentrations using 40 mg/L of CDs at pH 6. The result shows that increased MB concentration led to a reduction in photocatalytic activity. At a low concentration of 2 ppm, dye molecules inhibit the available sites without any competition, allowing the majority of the compound to be adsorbed on the photocatalyst surface. As the concentration of MB increases, a decrease in solute adsorption due to the inaccessibility of active sites necessary for the high initial concentration of the MB solution (Edakkaparamban et al., 2023). In addition, the amount of light (quantum photons) penetrating the MB solution and reaching the surface of CDs decreases due to light hindrance (Edakkaparamban et al., 2023; Widiyandari et al., 2023). Consequently, this significantly impacts the performance and efficiency of the photocatalytic process, leading to lower photocatalytic reaction rates (Ali et al., 2024; Yadav et al., 2023). Therefore, it is crucial to maintain an optimal photocatalyst and dye concentration to achieve maximum degradation efficiency. In addition, the slight difference between each concentration can be attributed to the low yield of 5.6 % of found in the CDs solution.

J. of Sustainable Materials Processing and Management Vol. 4 No. 2 (2024) p. 31-41

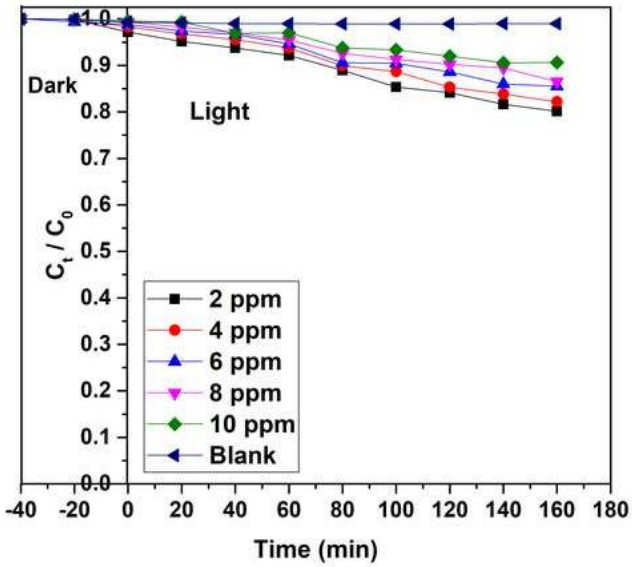



**Fig. 7** *Effects of concentration on photodegradation by 40 mg/L of CDs at pH=6 under visible light irradiation*

### 3.6.3 Effects of pH

pH plays a significant role in the photocatalyst reaction as it affects various factors such as the surface charges of photocatalysts, electrostatic interactions between dye and photocatalysts, pollutant generation, and the ionization states of MB (Chen et al., 2022). As shown in Fig. 8, the MB degradation efficiency significantly increased from 33.84% to 68.50% as the pH of the solution elevated from 2 to 12. This finding is in line with the observations of Wang et al. (2022) and Pavel et al. (2023), who noted that efficient and rapid reactions occur when dye molecules are successfully adsorbed on the surface of the photocatalyst. The increase in negative charge is due to the rise in hydroxyl surface groups, which act as surface traps, preventing greater recombination of electrons and holes. Since the surface of the CDs is negatively charged (-47.2 mV at pH 7), it can readily interact with positively charged dye molecules in the solution (Liu et al., 2021; Mukherjee et al., 2021). However, in severely acidic environments, the generation of reactive intermediates is less favorable and hence less spontaneous. According to Eskelinen et al., (2010), an increase in pH can reduce electron recombination, resulting in enhanced photocatalytic activity. Additionally, the increase in surface area of the photocatalyst leads to a higher concentration of negative charge carriers. This facilitates the formation of Coulombic interactions between these negative and positive charges, further enhancing the photocatalytic process (Yan et al., 2023). Lowering the pH also results in the production of positive charges on the surface of the photocatalyst, causing severe repulsion between the MB cation species and the photocatalyst's surface (Bhatti et al., 2024; Goodarzi et al., 2023).

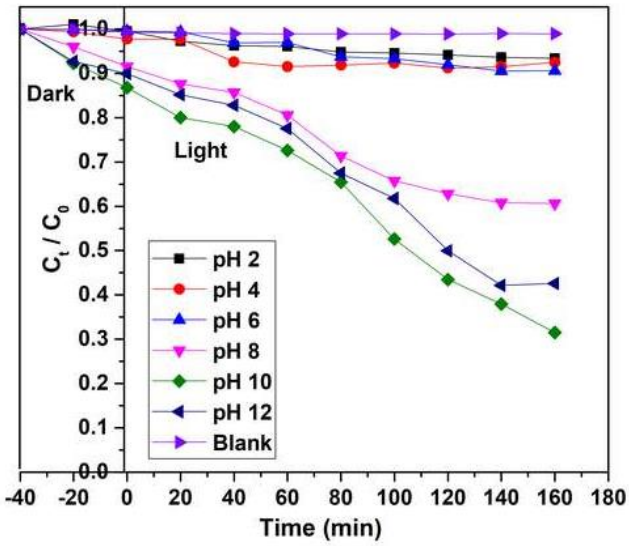



**Fig. 8** *Effects of pH on photodegradation by 40 mg/L of CDs at 10 ppm MB under visible light irradiation*

## 3.7 Kinetic Study of Photodegradation of MB

The rate constant of the photodegradation of MB are obtained by fitting into the pseudo-first-order kinetic data law as shown in Eq. (2), [33]:

$$-(\ln C - \ln C_o) = kt \quad (2)$$

Where: k = The pseudo-first order reaction rate coefficient, C = The concentration of MB

The graph was plotted using natural logarithm of MB initial concentration and the concentration preceding photocatalytic degradation (ln $C_o/C$) against irradiation time and the rate constant, k is obtained from the slope of the graphs as shown in Fig. 9.

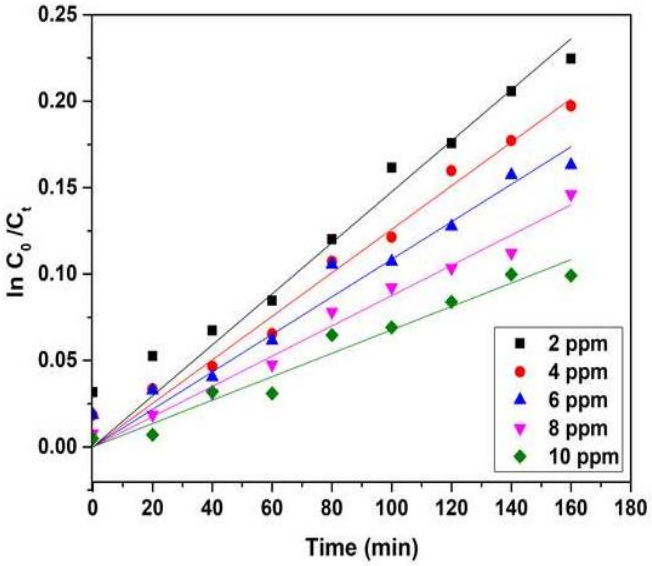



**Fig. 9** *Kinetics photodegradation of MB at optimum concentration*

The half-life of each concentration is described as the time taken for photocatalyst to decrease by half and can be obtained from the reaction rate coefficient using the Eq. (3) below:

$$t_{\frac{1}{2}} = \frac{\ln 2}{k} \quad (3)$$

Table 2 summarizes the half-life and rate constant, k each MB concentration. As shown in Table 2, the highest rate constant 1.5 x $10^{-3}$ $min^{-1}$ corresponded to a half-life of 462.10 min for the reaction with 2 ppm of MB at 160 minutes. This finding aligned with previous literature where a shorter half-life was associated with an increased rate constant (Syahin et al., 2019). The rate constant measured in this study was lower than those reported in some other studies, for instance, Edakkaparamban et al. (2023), that synthesized carbon quantum dots derived from coconut husk for the photodegradation of methylene blue found a rate constant range of 0.013 – 0.021 $min^{-1}$. This difference could be attributed to the low yield (5.6%) of CDs and degradation in visible light.

**Table 2** *Half-life and rate coefficient at each concentration*

| Concentration(ppm) | k (min-1) | R2 | Half-life(min) |
|---|---|---|---|
| 2 | 0.0015 | 0.9833 | 462.10 |
| 4 | 0.0013 | 0.9895 | 533.19 |
| 6 | 0.0011 | 0.9817 | 630.13 |
| 8 | 0.0009 | 0.9894 | 770.16 |
| 10 | 0.0007 | 0.9840 | 990.21 |

## 4. Conclusion

Waste products derived from biomass that are prevalent across natural and living environments are found in large quantities. This study presented a simple one-step hydrothermal technique to synthesize fluorescent nanomaterials from biomass waste. The photocatalysis activity of the CDs was assessed by measuring the breakdown of MB over 160 minutes' period under visible light exposure. The CDs was found to perform best at a dosage of 20 mg/L with a MB degradation 30.23 %. The photodegradation process was greatly accelerated by increasing the pH of the solution to 10, with a 68.50 % of efficiency after 160 minutes. The kinetic study showed that the removal of MB was found to be highest at the concentration (2 ppm), reaching 30.23 %. The corresponding rate constant was determined to be 1.5 x $10^{-3}$ $min^{-1}$ and a half-life of 462.10 min. These results indicate that CDs hold significant potential in the field of photocatalysis for the degradation of MB under visible light conditions.

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## Conflict of Interest

Authors declare that there is no conflict of interests regarding the publication of the paper.

## Author Contribution

*The authors confirm contribution to the paper as follows:* ***study conception, design & data collection:*** *Huey Ling Tan, Ying Pei Lim;* ***analysis and interpretation of results:*** *Wan Nuraishah Wan Ishak;* ***draft manuscript preparation:*** *Wan Nuraishah Wan Ishak Author, Huey Ling Tan, Noor Fitrah Abu Bakar, Ying Pei Lim. All authors reviewed the results and approved the final version of the manuscript.*

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