

Research Article

Novel and Facile Synthesis of Carbon Quantum Dots from Chicken Feathers and Their Application as a Photocatalyst to Degrade Methylene Blue Dye

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Methylene blue (MB) is a most commonly used synthetic dye in the textile industry. It is an extremely carcinogenic phenothiazine derivative and therefore needs to be removed from the water bodies. In the present study, a single-step hydrothermal novel synthesis of carbon quantum dots (CQDs) extracted from biomass of chicken feathers has been performed, and the synthesized CQDs were applied to remove MB present in the aqueous samples. A number of techniques such as ultraviolet-visible (UV-Vis) spectroscopy, Fourier-transform infrared (FTIR) spectroscopy, scanning electron microscopy (SEM), energy-dispersive spectroscopy (EDS), and X-ray diffraction (XRD) were used to characterize the samples for the conformity purposes. SEM and XRD analysis showed that CQDs are highly crystalline and have spherical structures with an average particle diameter of 35 nm. In the presence of 0.2 g of synthesized CQDs, MB dye degraded drastically under the sunlight. The rate of degradation was studied by determining the absorbance of the degraded sample with time relevant to untreated sample. The % degradation achieved during first 60 min of time was approximately 92% which increased minimally to a value of only 95% after 100 min of time. The ease of synthesis of carbon dots at low cost contributes hugely to their utilizations as an efficient photocatalyst for the degradation of aqueous pollutants. The opted approach to synthesize CQDs is cost-effective and eco-friendly and demonstrates excellent potential to remove MB from the aqueous samples.

1. Introduction

Industrialization and urbanization have resulted in a massive influx of organic dyes into the environment, particularly in the aquatic system [1, 2]. Synthetic dyes find extensive applications in a large number of industrial sectors including leather industries, food industries, cosmetic industries, textile industries, and paint industries. These dyes are poisonous and non-biodegradable, not only hazardous for marine life but are also toxic for land animals and humans [3–5]. Humans are indirectly affected due to bioaccumulation and bioamplification

of the dyes, and thus, the human health can face some really serious threats upon the ingestion of this dye including cancer [6, 7]. Methylene blue (MB) dye is a most commonly used dye in the textile industry for the finishing purposes. This dye is an extremely carcinogenic phenothiazine derivative that causes hypertrophication and thus deteriorates the living conditions for aquatic flora and fauna. MB can cause skin allergies, vomiting, nervous system disorder, cardiovascular damage, breathing difficulties, nausea, and gastric infection [8–10]. Therefore, MB must be removed from the aqueous samples to avoid the harmful effects of this dye on the public health [11].

A number of different methodologies were reported for the elimination of MB from water bodies. The reported techniques include photo-oxidation, chemical reduction [12–15], adsorption [16], biological treatment [17], membrane filtration [18], flocculation [19], and photocatalytic degradation [20, 21]. Though all the methods mentioned above are more or less effective in the removal of dyes, but the photocatalysis of MB has a big advantage over other processes. Photocatalysis of MB is a cost-effective approach and has simplicity of operation, and the involvement of sunlight in photocatalysis degrades MB into simpler less hazardous organic products. In material science, nanotechnology is becoming increasingly important because of its potential to develop materials of unique characteristics by manipulating matter at the nanoscale. Nanostructures have gained considerable importance in recent years due to their distinguished physicochemical properties over bulk materials [22–24]. Some of the most distinct features of nanostructures are their higher surface area to volume ratio, surface energy, and chemical reactivity. However, the synthesis of nanostructures through physical or chemical methods involves the addition of hazardous toxic chemicals and higher pressure/temperature which makes these methods much expensive and unfriendly for the environment [25–31]. Therefore, it is imperative to develop cost-effective and eco-friendly methods for the preparation of nanostructures. A number of studies have reported the applications of nanostructures to eradicate a variety of pollutants from various water samples. Recently, some comprehensive studies have also been conducted to remove various persistent organic pollutants through photocatalytic degradation [32–36].

In this context, the use of chicken feathers to develop nanostructures such as carbon quantum dots (CQDs) can have a dual benefit in terms of environment friendliness and sustainability. Also, the use of chicken feathers in the preparation of CQDs can considerably reduce tons of waste being produced every day. Chicken feathers make approximately 5–7% of a chicken's body weight. Millions of tons of these important poultry byproducts are produced every year throughout the world [37, 38]. By weight, chicken feathers are roughly 50% barbs (feather fiber) and 50% rachis (quill). Both barbs and rachis consist of keratin, which is an insoluble and long-lasting protein found in hooves, hair, and horns of animals [39]. Fibers from chicken feathers have a number of prominent characteristics including their flexibility, surface toughness, hydrophobicity, an organized morphology, and a higher length-to-diameter ratio [40]. In addition, these protein fibers are biodegradable, self-sustainable, and renewable because of their natural biopolymer origin [41]. Feathers are almost completely disposed of by incineration despite their aforementioned unique features, which have led to a number of severe environment related problems [42]. Many research studies are concentrating on recycling this renewable source of biopolymer because of its biocompatibility, biodegradability, and high protein contents [43]. In recent years, a large number of data have been published which suggest the applications of feathers such as ingredients of animal feed,

reinforcement in different composites, sorbents of toxic and hazardous compounds, filtration and insulation material, and films [44]. Among carbon-based materials, CQDs have gained a huge importance due to their key role in the progress of material science [45]. CQDs are quasi-spherical nanomaterials and possess excellent properties such as cost-effectiveness, high solubility in water or organic solvents, high dispersibility, less toxicity, good biocompatibility, and great fluorescent characteristics [46, 47]. Various strategies have been adopted for the synthesis of CQDs including TiO_2 -wsCQDs, CDs/N- TiO_2 , NCQDs/ TiO_2 , WO_3 /GO/NCQDs, ZnO/C-dots, ZnO-CDs, NCQD/g- C_3N_4 , ZnO/CQDs/AuNPs, CQDs/ $\text{BiO}(\text{COOH})/\text{uCN}$, and CQDs/Au/BMO, for the photodegradation of numerous organic pollutants especially the dyes. The abovementioned CQDs require expensive chemicals to synthesize them; however, in the present study, chicken feathers which are being wasted were utilized to prepare CQDs. So, this approach of converting the waste material into useful products demonstrates the novelty of our current studies.

In this study, a single-step hydrothermal process was employed to synthesize CQDs from chicken feathers. The approach is believed to be an extremely promising one because of milder processing conditions while leaving behind no harmful byproducts. In literature, electrochemical, microwave, ultrasonic, hydrothermal, and solvothermal approaches have been employed to manufacture CQDs [48–50]. In this study, we have used hydrothermal approach to synthesize CQDs. The hydrothermal method is facile, cost-effective, and eco-friendly as compared to other methodologies reported in the literature [51]. The successful synthesis of CQDs was confirmed by using UV-visible spectrophotometer, FTIR, SEM, EDS, and XRD techniques. Photocatalysis of MB was carefully evaluated to investigate the photocatalytic performance of synthesized catalyst.

2. Experimental

2.1. Materials. Chicken feathers (Bioprecursor) were obtained from a local market of Lahore, Pakistan. Methylene blue was procured from Hajvery Scientific Store, Lahore, Pakistan (originate to Merck, Germany). Ultrapure water (18 M Ω -cm resistivity) from the GenPure water system (Thermo Scientific, USA) was used to prepare different dilutions of the solutions.

2.2. Preparation of Extract and Synthesis of CQDs. The CQDs were synthesized in the presence of chicken feathers biomass using a single-step hydrothermal approach. The hydrothermal treatment commonly involves heating at around 150°C–200°C. This study used the intermediate condition of temperature (180°C) as the reaction condition and duration of hydrothermal treatment varied from 6 to 24 h [6]. Chicken feathers biomass was used as a precursor for carbon source. The obtained chicken feathers were washed several times with tap water followed by a couple of thorough washings with distilled water. After repeated washings, the

feathers were dried in an oven (Vision Scientific, Korea) for 48 h at 80°C. By using a grinder machine (GRINDOMIX GM 300, Thomas Scientific, USA), the dried feathers were shredded to transform them into the powder form. The obtained powder was then passed through a sieve of mesh size 80 to remove the coarser particles present in it. Afterwards, 3 g of dried powder was dissolved in 30 mL of doubly distilled water and the solution was then stirred at 900 rpm for 2 h.

Finally, the solution was transferred into a stainless-steel hydrothermal autoclave reactor and heated at 180°C for four different time durations (6, 12, 18, and 24 h). After the reaction, the reactor was cooled down to room temperature naturally. The products were collected by removing the large particles through centrifugation at 12000 rpm for 20 min and then filtrated with a 0.22 μm filter membrane. Column chromatography was used to separate raw products, where aluminum oxide having particle size ranging between 63 and 200 μm (Merck, Millipore Germany) was used as a stationary phase. For fractionation, acetonitrile: Millipore-water with a volume ratio of 1:1 eluent was used isocratically. The synthesized CQDs were stored at 4°C for future use and characterization. The aqueous phase was dried at 80°C, and then, the obtained solid was dried at 105°C for 2 h under vacuum. The schematic diagram for CQDs preparation is presented in Figure 1.

2.3. Photocatalytic Degradation of Methylene Blue and Its Regeneration. Stock solution (1000 ppm) of MB dye was prepared. From this standard solution, a 15 ppm solution was made and mixed with different concentration ranges of 0.05–0.2 g of CQDs. To ensure attainment of adsorption-desorption equilibrium in a photocatalytic reactor, the attained mixture was stirred for 30 min in the dark. An LED lamp (4410R–18 W) was used to irradiate the equilibrated solution, while aliquots (5 mL) were collected periodically from 10 min to 100 min. To remove the photocatalyst composite, the withdrawn samples were centrifuged for 3 min at the rate of 12,000 rpm. The maximum absorption band (665 nm) was recorded as a function of time by analyzing the supernatant through a UV-Vis spectrophotometer (PG Instruments, UK). The kinetic rate constant of the MB degradation was calculated using the Langmuir–Hinshelwood model as shown in the following equation:

$$\ln \frac{C_o}{C} = k \times t. \quad (1)$$

Moreover, the photodegradation efficiency was calculated as well by using the following equation:

$$\text{Degradation efficiency} = \frac{C - C_o}{C_o} \times 100 \%, \quad (2)$$

where C_o = initial MB concentration, C = MB concentration at irradiation time t , and k is kinetic rate constant of photocatalyst (CQDs). The results have been reported in terms of percentage degradation with respect to time (10–100 min). The CQDs were recollected from degraded MB dye solution by performing column chromatography of

the mixture and the stability of CQDs was evaluated. Next, the collected CQDs were analyzed using XRD to check their stability and were reused for photodegradation activity for 5 cycles.

2.4. Characterization of CQDs. For the spectrophotometric analysis, UV-Visible spectra of all the samples were obtained by using a Spectroquant® Prove 300 UV-Vis spectrophotometer having a wavelength range from 200 to 800 nm. It is considered a good tool to study the optical structural property as well as to confirm the synthesis of the sample. Agilent technologies Cary 630 FT-IR spectrometer was used to obtain the FT-IR spectra of the sample in the range of 400–4000 cm^{-1} . Scanning electron microscopy (SEM, Hitachi S-4700) operating at 25 kV was used to investigate the morphological characteristics of the CQDs. The X-ray diffraction (XRD) analysis was performed by using XRD diffractometer D8 Advance, Bruker Corporation, USA, operating at the working conditions of 30 kV, 20 mA, and Cu-K α radiations with the wavelength of 1.54 Å. Elemental composition of the synthesized CQDs was examined through energy dispersive spectroscopy (EDS) using an Oxford Inca Penta FETx3 EDS instrument attached to Carl Zeiss EVO MA 15 scanning electron microscopy.

3. Results and Discussion

3.1. Characterization. The chicken feathers contain peptide bonds [52] which act as a precursor for the formation of CQDs. Thermal decomposition begins with random chain breakage after uncoiling of tertiary structure of feathers. A prolong heat treatment provides enough crosslinks in the protein matrix which helps to keep the structure of peptide intact. A series of transformation occurs in the protein matrix. These transformations include aromatization and cyclization reactions at a temperature higher than the melting point. Such transformations cause the degradation of the matrix and thus the release of cyclic amines and aromatic carbons. These pyrolysis processes can help as a guide to develop catalysts and other materials with desired properties [53, 54].

The UV-Vis spectrum of CQDs synthesized from chicken feathers is shown in Figure 2. The spectrum showed two separate absorbance peaks at 215 and 275 nm, respectively [55]. The first broad peak at 215 nm can presumably be attributed to the $\pi - \pi^*$ transitions within C=C bonds. The second peak demonstrates the C=O functional group. Figure 1 displays UV-Vis spectra of CQDs prepared at different time intervals ranging from 6 to 24 h at 180°C, while the peak at 670 nm is due to the color of CQDs. The CQDs synthesized show almost same absorption spectra at different time intervals. The spectrum obtained at 18 h of time depicts optimum conditions for the preparation of CQDs from chicken feathers. It can be concluded from the spectroscopic analysis that the strong $\pi - \pi^*$ peak at higher absorption intensity is because of organic domains within the carbon framework. The presence of C=C and C=O functional groups presents a rich source of its surface functionality.

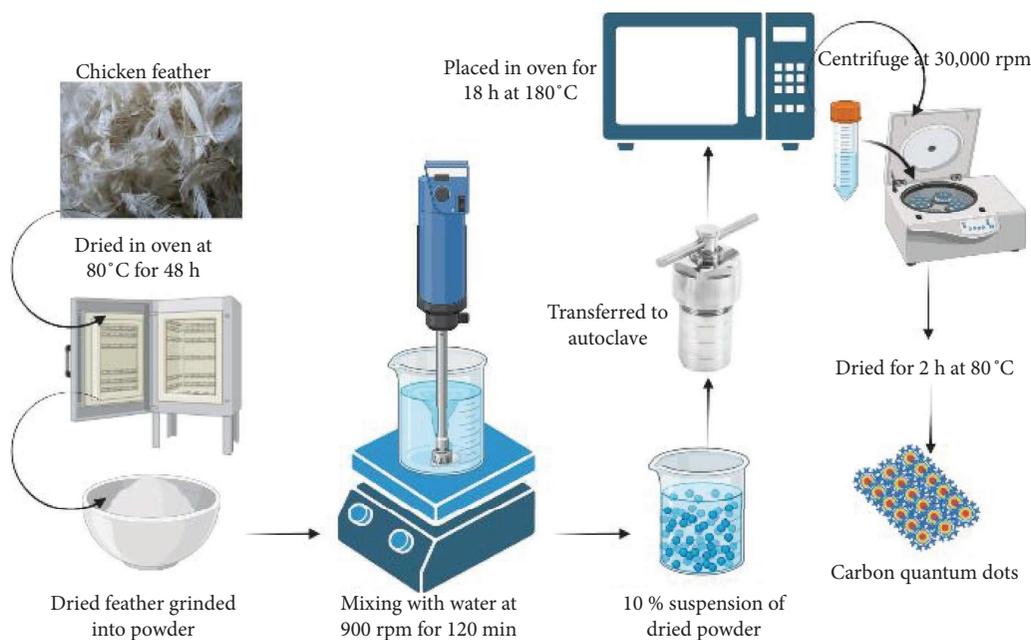


FIGURE 1: Schematic diagram for the preparation of CQDs.

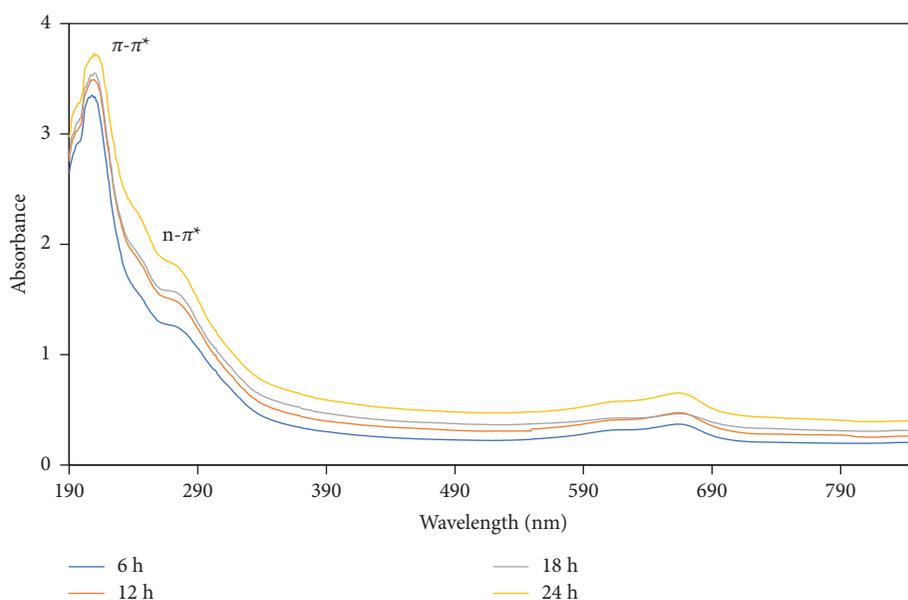


FIGURE 2: UV-Vis spectra of CQDs prepared at 180 °C after different time intervals.

FTIR spectroscopy was used to explore the presence of different functional and nonfunctional groups in the CQDs. In Figure 3, the absorbance peak at 1360–1367 cm^{-1} shows C-H bending vibrations of alkanes. The region 1715–1793 cm^{-1} was assumed for the C=O stretching vibrations which correspond to the esters and acid halides. The C-H stretching vibrations of alkanes and alkenes are shown by the absorbance peak at 2901–3011 cm^{-1} and 3100–3199 cm^{-1} , respectively. The peak at 3476–3481 cm^{-1} indicates N-H stretching vibrations whereas 3616–3619 cm^{-1} specifies the presence of O-H (Figure 3). Thus, during the hydrothermal process, polyaromatic

nanostructures are most likely developed [56–58]. The functional group such as O-H, N-H, C=O, C=C, and C-H indicating the rich presence of functionalities such as hydroxyls, amines, and carbonyls. These functionalities were further confirmed by EDS studies.

The average particle size and morphologies of biogenically synthesized CQDs were analyzed through SEM analysis. Figure 4(a) shows the SEM analysis of CQDs and size morphology was scrutinized by using image J software that confirms spherical morphology of CQDs with particle size diameter 35 nm which is in agreement with reported size of CQDs by SEM [59–61]. Furthermore, Figure 4(b) displays

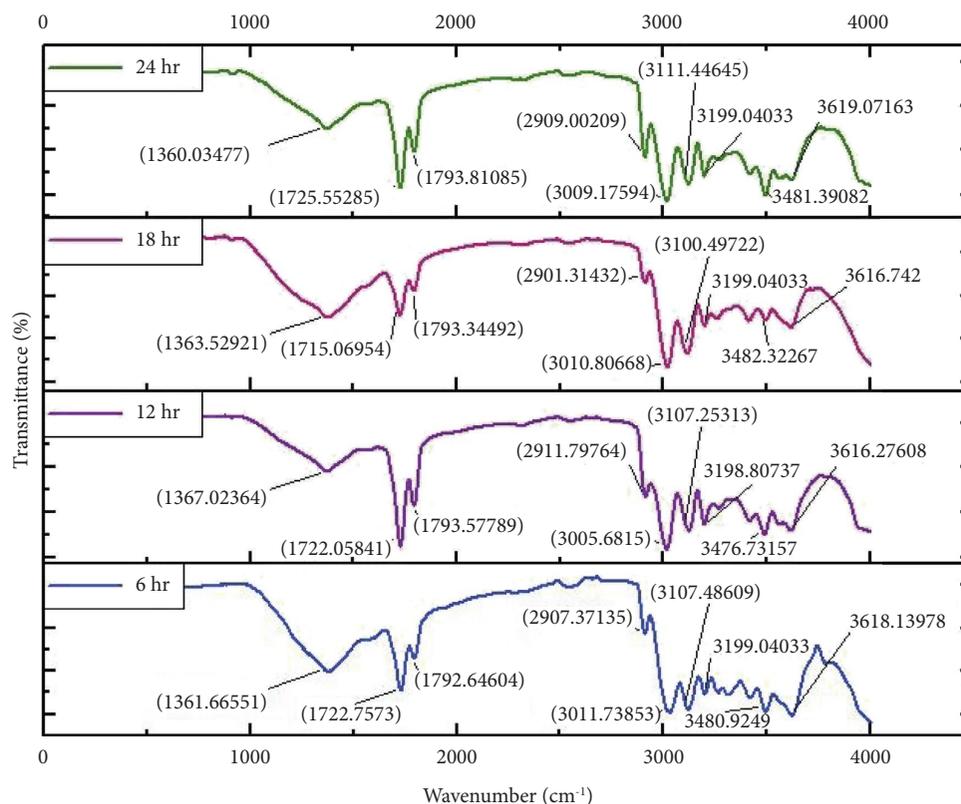


FIGURE 3: FTIR spectra of CQDs.

EDS analysis of biogenically manufactured CQDs. The EDS image showed that CQDs sample contains 62.9% carbon, 1.3% nitrogen, and 35.8% oxygen with the corresponding peaks at 0.284, 0.399, and 0.531 keV, respectively. Since the nitrogen content is very low and its signal in EDS is obscured. To the best of our knowledge, no study synthesized CQDs from chicken feathers having 62.9% of high purity of carbon.

The XRD studies were performed to determine the phase purity and crystal structure of synthesized CQDs. The results of XRD analysis of CQDs are shown in Figure 5. Hence, in XRD spectra, several strong and well-resolved peaks appeared in the 2θ region of $20-80^\circ$. The values of 2θ for characteristic peaks are 32.5° , 37.4° , 43.6° , 63.96° , and 76.98° , respectively.

Several broad diffraction and sharp peaks were obtained in the diffractogram of CQDs. The presence of amorphous carbon and organic materials is evident from the diffraction peaks appearing between $2\theta = 20-30^\circ$. However, the presence of sharp diffraction peaks confirms a better crystallinity of CQDs rather than the presence of ordinary carbon nanoparticles.

3.2. Photodegradation of Ethylene Blue Dye. In the current study, UV-Vis spectroscopy was used to investigate the percentage degradation of methylene blue dye in the presence of CQDs acting as a catalyst. Methylene blue degraded drastically in the presence of 0.2 g CQDs (photocatalyst) under sunlight. Almost 50% of the degradation of dye was achieved during first 10 min. However, the percentage

degradation of MB achieved during the next 10 min of time was much lower as compared to the percentage degradation obtained during the first 10 min. After 20 min, the achieved % degradation reached a value of 65%. Likewise, the percentage degradation achieved after every next 10 min of time interval continued declining and no significant differences in the percentage degradation values were found after 60 min of time. The absorbance peak due to MB at 650 nm in the UV-Vis spectra did not decrease further significantly after 60 min. However, the spectra were taken continuously after every 10 minutes of time interval until 100 min of time. The percentage degradation achieved during first 60 min of time was approximately 92% which increased minimally to a value of only 95% after 100 min of time (Figure 6(a)). These results indicate that the performance of CQDs was much efficient at the start, but their efficiency declined with time. A gradual decrease in the performance of CQDs is likely due to surface coverage of the CQDs by the dye molecules in the first condition (equilibrium adsorption followed by photocatalysis). Such an adsorption of dye molecules on the surface of CQDs can reduce the efficiency by suppressing the further adsorption and hence the photodegradation [61]. However, a significant achievement of percentage degradation of MB indicates that CQDs still show a great potential to be reused for the removal of MB from aqueous samples. As shown in the Figure 6(b), the CQDs synthesized via hydrothermal process produced a higher rate constant having a value of $3.15 \times 10^{-2} \cdot \text{min}^{-1}$, which indicates that the CQDs synthesized from the chicken feathers can potentially

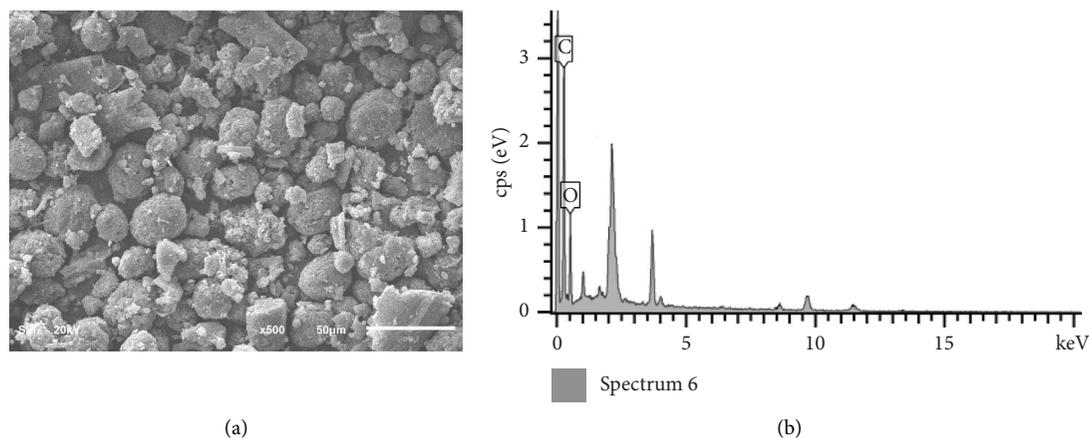


FIGURE 4: SEM (a) and EDS (b) of CQDs.

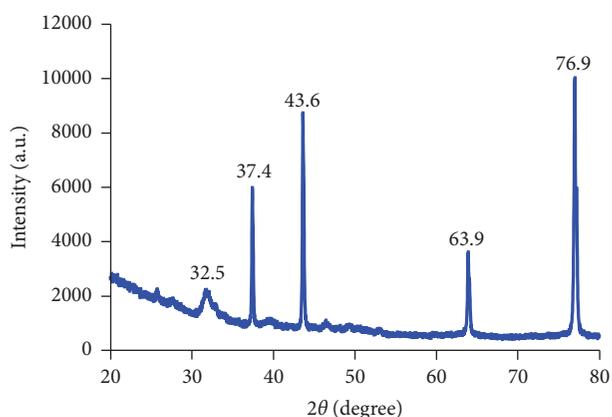


FIGURE 5: XRD pattern of CQDs.

degrade the MB dye present in the water samples at a faster rate. The degradation of MB obeys pseudo first-order kinetics, where MB degrades almost linearly. The rate constant and regression coefficients (R^2) value for MB dye degradation are $3.15 \times 10^{-2} \cdot \text{min}^{-1}$ and 0.9728, respectively.

UV-Vis studies were performed at a constant MB concentration (15 ppm) to examine the degradation performance of CQDs. As shown in Figure 6(c), the degradation rate of MB increases with an increase in the concentration of CQDs. In the presence of sunlight, the degradation of MB increased remarkably while using 0.2 g of CQDs. The characteristic peak of MB at 665 nm in UV-Vis spectrum almost disappeared after degradation.

3.3. Mechanistic Study for Photocatalysis of MB and Its Regeneration. The photodegradation performance of synthesized CQDs was observed by mineralization of MB dye under sunlight. The sunlight irradiation generates electron hole pairs on the surface of CQDs. The photogenerated electrons add to oxygen (O_2) to produce superoxide anionic radicals (O_2^-) while photogenerated holes (h^+) adsorb water molecules to generate hydroxyl radicals (OH). The mechanism for photocatalysis of MB dye in the presence of CQDs has been illustrated in Figure 7.

As suggested by the reported studies [62–64], the $\bullet\text{OH}$ attacks the cationic sulfur group and heteroaromatic ring of the MB that causes the opening of the aromatic ring. As a result, hydroxylated intermediate and sulfoxide are produced. The dissociation of the two rings can result because of the further oxidation of the sulfoxide groups to sulfone. Eventually, the volatile lower molecular weight compounds (CO_2 , H_2O , NH_2 , NO_2 , and SO_3) are formed as a result of decomposition of these aromatic compounds.

Consecutive photodegradation experiments were performed to check the reusability of CQDs. A slight reduction in the dye degradation percentage was observed in consecutive five cycles (Figure 8). A decrease in the percentage of MB dye degradation is possibly due to some losses of CQD particles during the activity of separation performed in column chromatography.

To ensure the stability of synthesized CQDs, the XRD spectra of CQDs were taken before and after the degradation studies (Figure 9). For at least 5 cycles of reusability, the CQDs showed an appreciable stability for repeated photocatalysis.

3.4. Comparison of Photodegradation Results with Previous Studies. Mostly the bottom-up and top-down strategies are used for the preparation of CQDs. The CQDs can be separated from each other by the direction in which the expansion of size of the implemented materials occurs. The bottom-up technique employs thermal decomposition, microwave synthesis, hydrothermal treatment, plasma treatment, and template-based routes to synthesize CQDs from molecular precursors e.g., glucose, sucrose, and citric acid. The synthetic raw materials and photodegradation studies of MB which were investigated by CQDs in present study were compared with already reported data for photodegradation of MB. The comparison is provided in Table 1.

CQDs are the carbon-based nanomaterials that demonstrate enhanced features and exceptional properties. They have a huge potential to serve as most suitable alternatives for a large number of applications. Some of the most attractive characteristics of CQDs are their ultra-small size,

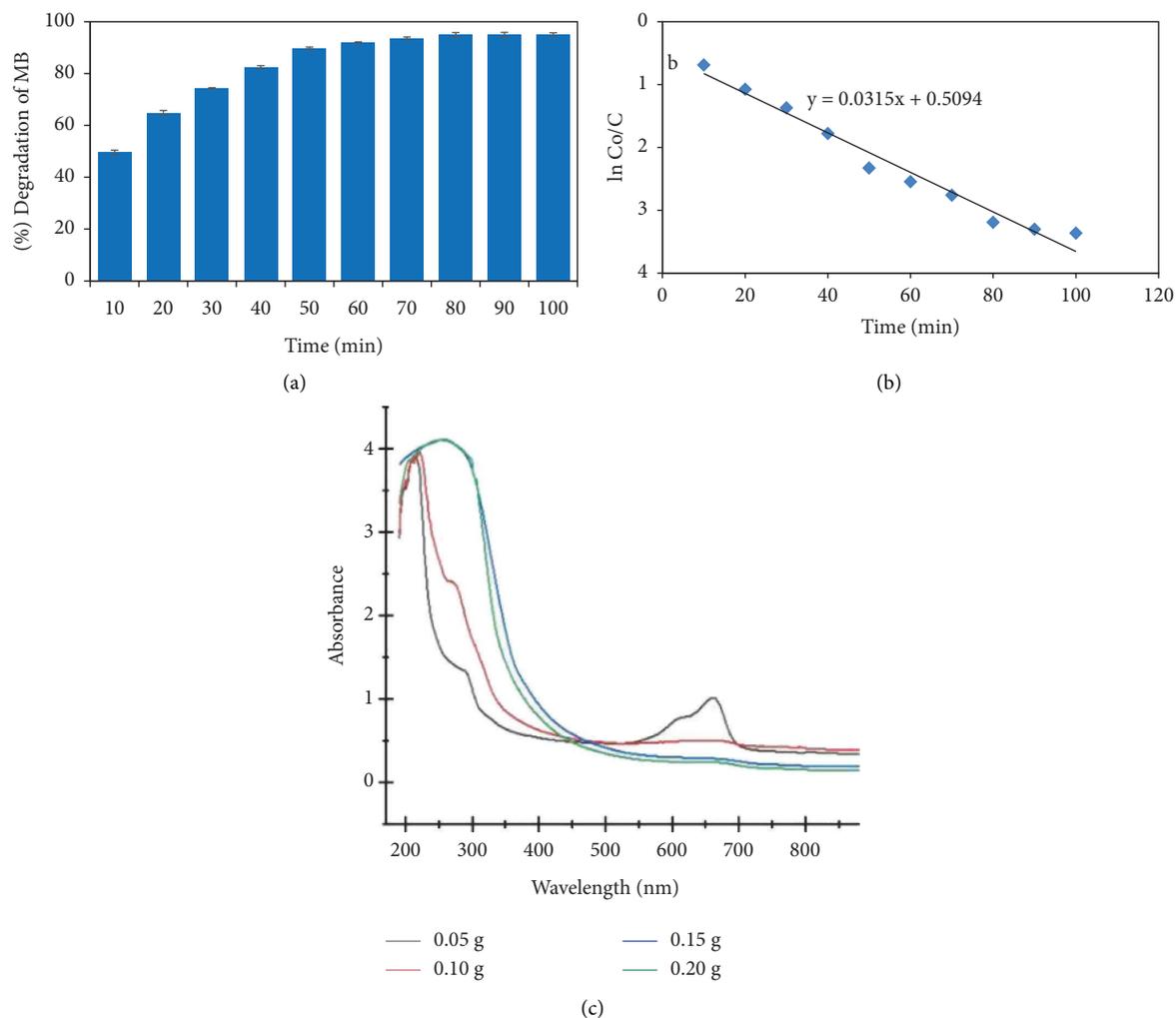


FIGURE 6: Photodegradation of methylene blue with the passage of time (a), kinetic rate constant (b), and UV-Vis spectrum of degraded MB dye (c).

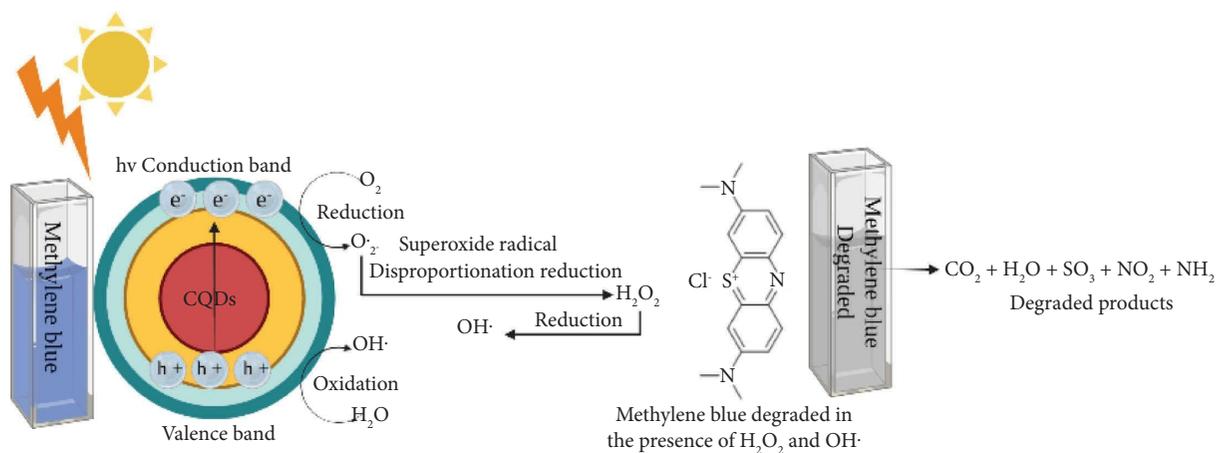


FIGURE 7: Schematic representation for photocatalysis of MB dye in the presence of CQDs.

cytotoxicity, biocompatibility, water solubility, ease of functionalization, and abundance nature of their precursors. Some disadvantages of CQDs include their variation in

photoluminescent behavior, impurities in the product, and nonuniformity. However, these disadvantages do not undermine the importance of CQDs.

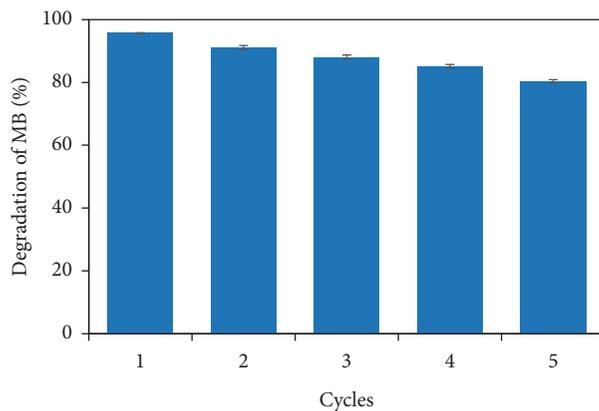


FIGURE 8: Reusability of CQDs in photocatalytic degradation of MB dye.

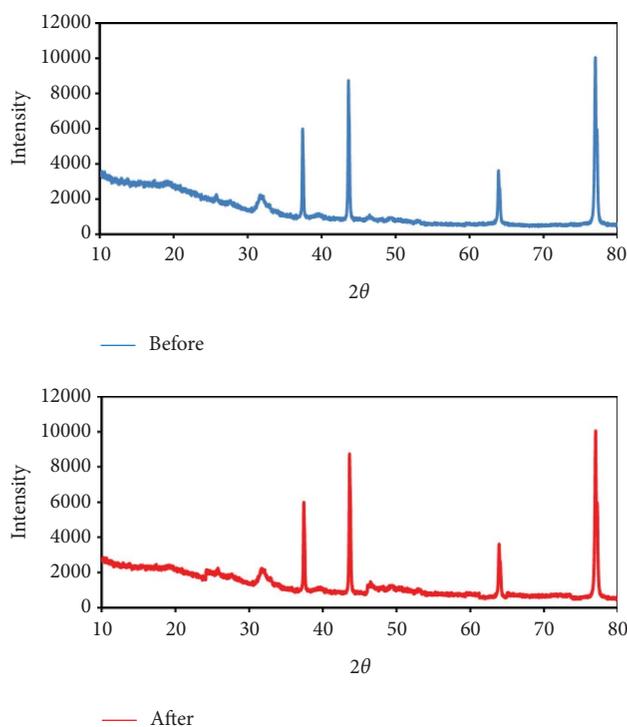


FIGURE 9: XRD pattern before and after the photocatalytic performance of CQDs.

TABLE 1: CQDs-based materials' synthesis and photocatalytic degradation of MB.

CQDs-based materials	Source	Method	Degradation achieved	Reference
TiO ₂ -wsCQDs	Lemon peel	Hydrothermal	80.0% after 120 min	[58]
TNTs/CQDs	Citric acid	Hydrothermal	91.3% after 20 min	[65]
TiO ₂ /NP-CQDs	Citric acid	Thermal treatment	40.0% after 20 min	[66]
CQDZ	Ammonium citrate	Thermal treatment	95.0% after 60 min	[57]
PVA/CQDs	D-glucose	Solution casting method	97.0% after 40 min	[67]
g-C ₃ N ₄ /NCQD	Citric acid	Hydrothermal	91.2% after 180 min	[60]
ZnO/C-dots	Glucose and copra oil	Solvothermal	96.0% after 30 min	[68]
ZnO-CDs	Grounded coffee	Hydrothermal	97.0% after 100 min	[69]
AuNPs/ZnO/CQDs	D-lactose	Hydrothermal	98.6% after 50 min	[70]
CQDs	Chicken feather	Hydrothermal	95.0% after 100 min	Present study

4. Conclusion

The current research work was performed to produce the carbon quantum dots from the waste source i.e., chicken feathers. Since the chicken feathers are an all-natural and biodegradable source, the CQDs are environmentally friendly and pose no serious harmful effect to the environment. Their nonmetallic and chemically inert nature along with their ease of synthesis by hydrothermal process makes them an ideal candidate for photodegradation of MB dye. UV-Vis and FTIR spectroscopic investigation confirmed the synthesis of CQDs due to the presence of various functional moieties. The size and shape of CQDs were analyzed using SEM and XRD studies. The EDS analysis confirmed the formation of CQDs due to the presence of carbon as a major element. The kinetic studies revealed that the degradation of MB follows pseudo first-order reaction. The value of rate constant, k was found to be $3.15 \times 10^{-2} \cdot \text{min}^{-1}$. This value of k makes CQDs synthesized from chicken feathers as a potential candidate to degrade MB pretty faster. In addition, the stability studies were made to determine the reusability of synthesized CQDs. The photodegradation efficiency of these nanomaterials remained excellent for five cycles. The obtained XRD results of CQDs before and after their application in the degradation studies exhibited no significant difference, which again indicates an appreciable stability of these carbon-based nanostructures.

Data Availability

The data that support the findings of this study are available in this article.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

Authors' Contributions

All the authors have read the manuscript and gave their approval.

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