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Eco-friendly synthesis of Carbon Quantum Dots from rice husk and their potential applications in dicot plants

Tran Duc Loc, Pham Thi Bao Uyen, Tran Quang Vu, Nguyen Van Hai, Nguyen Van Phong, Pham Viet Hung, Vo Cong Anh, Ve Quoc Linh, Dao Le Minh Tuan, Do Thanh Tien*

ABSTRACT

Carbon quantum dots (CQDs) were synthesized from organic precursors, specifically rice husks, using a simple low-temperature hydrothermal process. This process did not involve surface passivation agents, oxidizing agents, or inorganic salts. Advanced analytical techniques such as X-ray diffraction (XRD), transmission electron microscopy (TEM), photoluminescence (PL), photoluminescence excitation (PLE), and Commission Internationale de l'Éclairage (CIE) were employed to investigate the structure, particle size, and optical properties of the CQDs. The positive effects of CQDs on the growth and development of green bean sprouts, morning glory, and mustard greens are detected in preliminary application trials. These findings will be the useful orientation for the application of CQDs in different crops.

Keywords: Carbon quantum dot, luminescent, cultivation

1. INTRODUCTION

By 2050, the world population is expected to reach ten billion people. To guarantee food security, food production must be increased by at least 50% by the middle of the 21st century. The growth rate of global agriculture output has decreased because the Green Revolution is reaching its biological limits (Dorling, 2013). In general, 90% of plant dry matter comes from photosynthesis. Therefore, photosynthesis became a primary research subject in improving plant productivity. Phases of photosynthesis include light energy absorption, electron transfer, photosynthesis, and carbon assimilation. These phases mainly occur in chloroplast. In photosynthesis, chlorophyll is the light-harvesting molecule. It converts light energy to electrical energy, causing electron transfer and the

generation of ATP (Adenosine Triphosphate - Energy storage molecule) (Rabinowitch, 1949).

These ATP molecules provide energy for CO₂ fixation, where biomass is generated. Light in the solar radiation spectrum's blue, red, and far-red regions is considered necessary for photosynthesis, but light in the UV and green regions is not (Liu and Van-Iersel, 2021). Significantly, Ozone depletion, caused by the discharge of toxic substances into the environment, increases UV intensity on the earth's surface. Therefore, harnessing UV light for photosynthesis could reduce the negative impact of UV on the Earth and improve crop productivity. In addition, photosynthesis could only harness less than 10% of solar energy. Therefore, the improvement of photosynthetic efficiency has become an important research issue. Additionally, the development of artificial photosynthesis systems has become a trending research direction (Lewis, 2016).

Rice husk accounts for 20% of the weight of a rice seed (Glushankova et al., 2018). Therefore, in Vietnam, 8.8 million tons of waste rice husk are discharged into the environment annually, causing severe environmental pollution (Tam and Linh, 2023). SiO₂ and C account for 13 - 15% and 18 - 20% of rice husk weight respectively, organic compounds and trace elements account for the rest of the rice seed's weight. Recently, research on utilizing rice husks to improve agricultural output has been conducted. However, most studies focus solely on developing nano SiO₂ from rice husks (Moosa and Saddam, 2017; Nayak and Datta, 2021; Nourbakhsh et al., 2011; Tran et al., 2021). Notably, carbon, organic compounds, and trace elements, which make up 75% of rice husks, have not been fully exploited.

Recently, many scientists have focused on synthesizing carbon quantum dots (CQDs) from various biomass sources, such as palm shells, oyster shells, bagasse, and cornstarch. These CQDs have demonstrated potential applications in bioimaging, drug delivery, pollutant detection, and wastewater treatment (Abu et al., 2023; Kong et al., 2024). Several methods are commonly used for CQDs synthesis, including hydrothermal Jia et al., (2012), microwave Manikandan and Lee, (2022), and electrochemical approaches (Mondal et al., 2016). Typical precursors for CQDs production include glucose, sucrose, glycol, glycerol, citric acid Patel et al., (2019), and ascorbic acid Jia et al., (2012), often combined with strong, toxic, and expensive reducing agents.

However, scaling up the production of biocompatible CQDs for practical applications remains a significant challenge. To address this issue, material-based approaches that avoid the use of harmful and costly byproducts in CQDs synthesis need to be explored. In this study, we aim to fabricate CQDs from rice husks, a low-cost and eco-friendly biomass source, and evaluate their effects on the growth of three dicot plants: bean sprouts, mustard greens, and morning glories. Furthermore, the study seeks to determine the optimal CQDs solution concentration for each plant species.

2. EXPERIMENT

Materials

Rice husks were collected from a local area in Hue City, Vietnam. Double distilled water was prepared using an Aquatron A4000D distillation system at the Faculty of Engineering and Food Technology, University of Agriculture and Forestry, Hue University.

Material fabrication

Firstly, impurities were eliminated by washing and rinsing rice husks with double-distilled water. After that, 5 grams of the cleaned rice husks were mixed with 80 milliliters of double-distilled water to prepare a reagent mixture. Next, three reagent mixture samples, labeled as CQD1, CQD2, and CQD3, were poured into Teflon-lined autoclave reactors to undergo hydrothermal reactions for 4 hours at 170°C, 200°C, and 230°C, respectively (Figure 1). The temperature range of 170°C to 230°C was selected based on a synthesis of references from previous studies on the hydrothermal fabrication of CQD solutions (Nammahachak et al., 2022; Wu et al., 2015; Peng and Travas-Sejdic, 2009; Bourlinos et al., 2012; Wang et al., 2016; Kong et al., 2024).

The resulting solution after heating was coarse-filtered using filter paper, producing a brownish-yellow liquid. It was then centrifuged for 10 minutes at a speed of 10,000 rpm to remove larger particles. No additional chemicals were used during this fabrication process. The CQDs were separated from the samples by vacuum filtration and centrifugation. Finally, the CQD samples were kept in a dark environment for the following experiments.



Figure 1 The fabrication procedure of Carbon quantum dots

Measurement systems used in the article

A D8 Advance X-ray diffractometer (Bruker, Germany) over a range of $2\theta = 10 - 70^\circ$ was used to study the material's crystal structure. The material's Photoluminescent spectra (PL spectra) were taken by a specialized FS5 Spectrofluorometer (Edinburgh Inc, England) was used to take the material's PL spectra. CIExyz 1931 software was used to determine the color coordinate of the PL spectra. The absorption spectrum of the material was recorded using a GENESYS 10S UV-Vis spectrophotometer (Thermo Scientific, USA). The particle size and morphology of the CQDs were examined using a JEOL JEM-1400 transmission electron microscope (JEOL, Japan) operating at an acceleration voltage of 100 kV.

3. RESULTS AND DISCUSSIONS

Morphology and structure of the Carbon quantum dots solutions

Figure 2 shows the XRD spectra of the CQD1, CQD2, and CQD3 samples. The spectra indicate that all the CQD samples exhibit an amorphous structure, which is consistent with the known properties of carbon quantum dot solutions (Kumar et al., 2018; Li et al., 2012; Park et al., 2014). Figure 3 shows the TEM images of the CQD1, CQD2, and CQD3 samples. The particle sizes of the carbon quantum dots in CQD1, CQD2, and CQD3 were 7.61 ± 2.27 nm, 6.35 ± 2.18 nm, and 8.75 ± 2.36 nm, respectively.

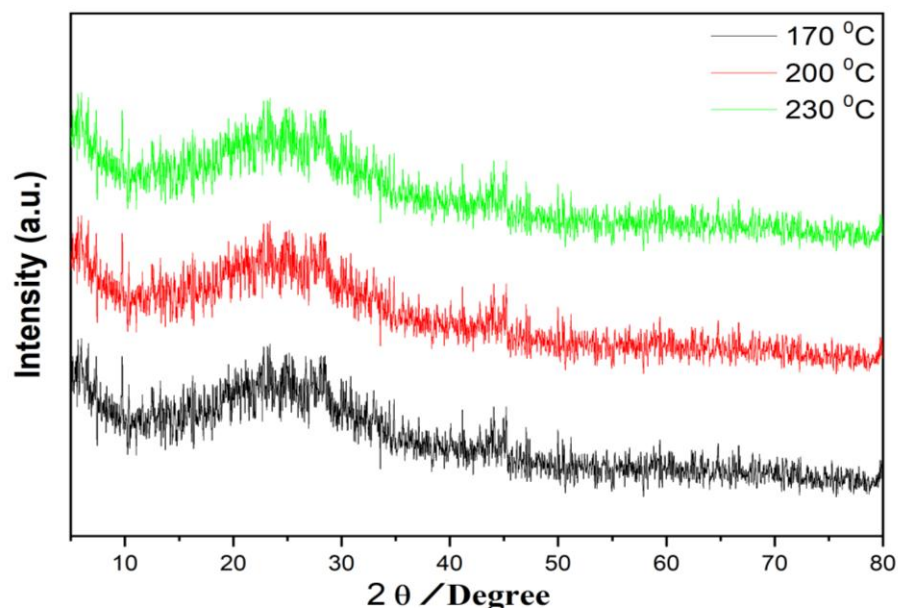
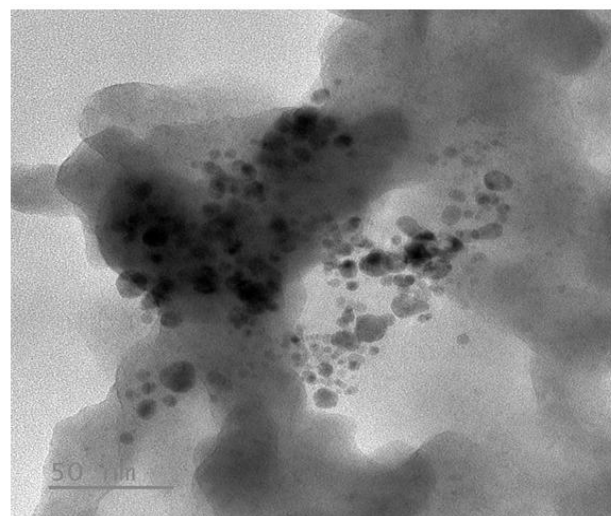
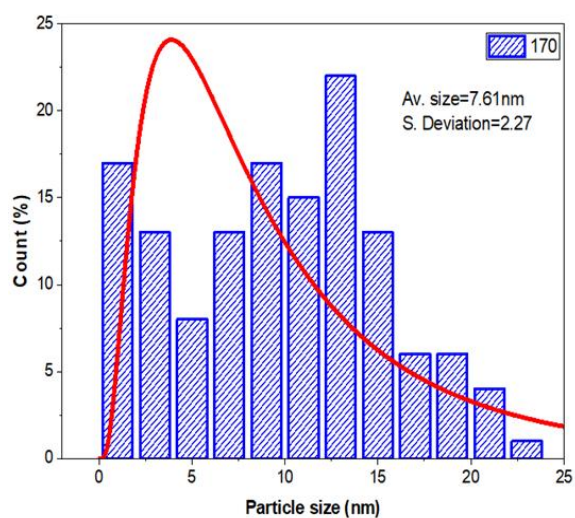
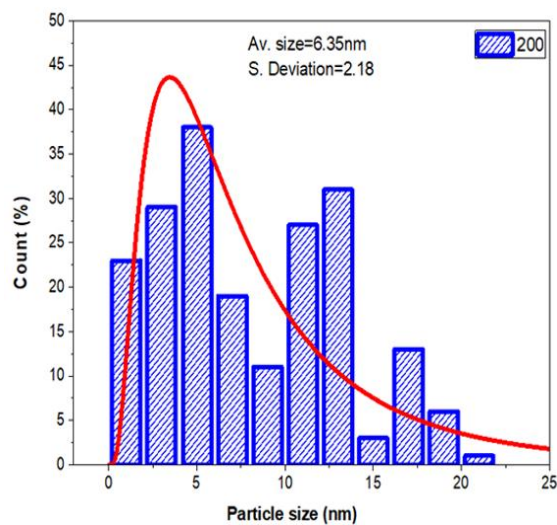


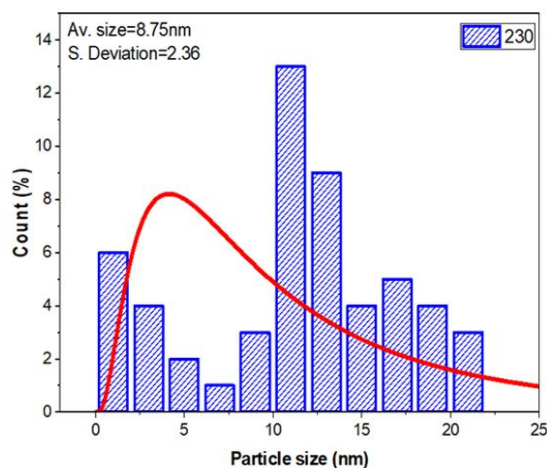
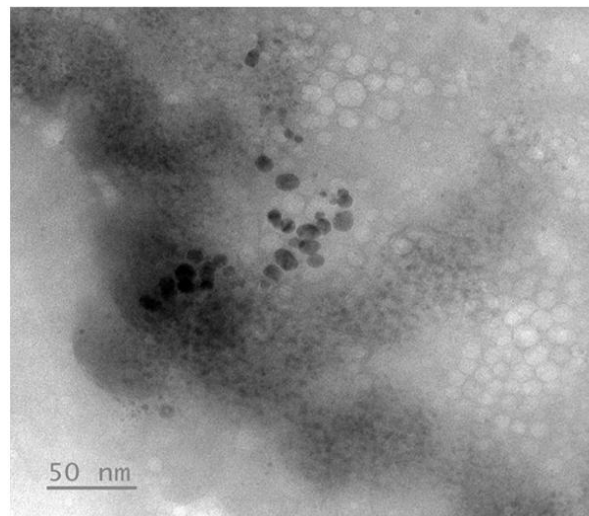
Figure 2 XRD spectra of CQD1, CQD2, and CQD3, fabricated at hydrothermal temperatures of 170°C, 200°C, and 230°C, respectively.



(a)



(b)



(c)



Figure 3 TEM images of the CQD1 (a), CQD2 (b), and CQD3 (c) samples, fabricated at hydrothermal temperatures of 170°C, 200°C, and 230°C, respectively.

By analyzing the XRD phase structure of the material and measuring TEM to determine particle size, we identified the optimal heating temperature to be 200°C for obtaining CQD solutions with the smallest particle size. This result aligns well with previous studies by other research groups that synthesized CQDs using hydrothermal (Nammahachak et al., 2022; Wu et al., 2015; Peng and Travas-Sejdic, 2009; Bourlinos et al., 2012; Wang et al., 2016; Kong et al., 2024). The optimal hydrothermal temperature typically ranges from 180 to 260°C, depending on the initially prepared material. To control particle size and shape, appropriate temperature should be taken into consideration as a vital factor.

Indeed, excessively high temperatures cause overreaction, structure destruction of the quantum dot, and the efficiency reduction of luminescence. Additionally, undesirable product transformations could have happened at extremely high temperatures because the bonds in the carbon structure may be completely broken. Conversely, overly low temperatures may excessively slow hydrolysis, resulting in incomplete reactions (Kong et al., 2024; Nazibudin et al., 2023). In our CQD fabrication process, a temperature of 200°C was sufficient to break the bonds within the complex organic molecules of the biomass (rice husks) without requiring additional toxic chemicals. This process releases carbon atoms, facilitating the formation of carbon quantum dots.

Luminescent characteristics of Carbon quantum dots solutions

Photoluminescent spectra and CIE color coordinate of the carbon quantum dot solution under different excitation wavelength

The luminescent properties of the synthesized carbon quantum dots were examined by studying the photoluminescence (PL) spectra and their corresponding color coordinates (Leonidovna, 2018). The color coordinates (x , y) of the CQD solutions were derived from the PL spectra excited at various wavelengths. The investigated results of the luminescence spectrum changing according to the excitation wavelength showed that when increasing the excitation radiation from 300 nm to 430 nm, the obtained luminescence spectrum had a maximum shift from 389 nm to 503 nm (Figure 4).

Additionally, the PL intensity diminished progressively due to the weak interaction between the CQDs and the excitation light source (Zhu et al., 2012). The shift in CIE color coordinates with varying excitation wavelengths is demonstrated in (Figure 5). As the excitation wavelength increased, the CIE color coordinates of the CQD solutions transitioned from blue to green. The photoluminescence mechanism can be attributed to photon reabsorption, the quantum confinement effect, and surface defects (Zuo et al., 2015). As the excitation wavelength changes, emission trap centers on the surface become dominant. Notably, smaller carbon particles have a higher surface-to-volume ratio, resulting in a greater number of trap centers, which enhances the excitation of more electrons.

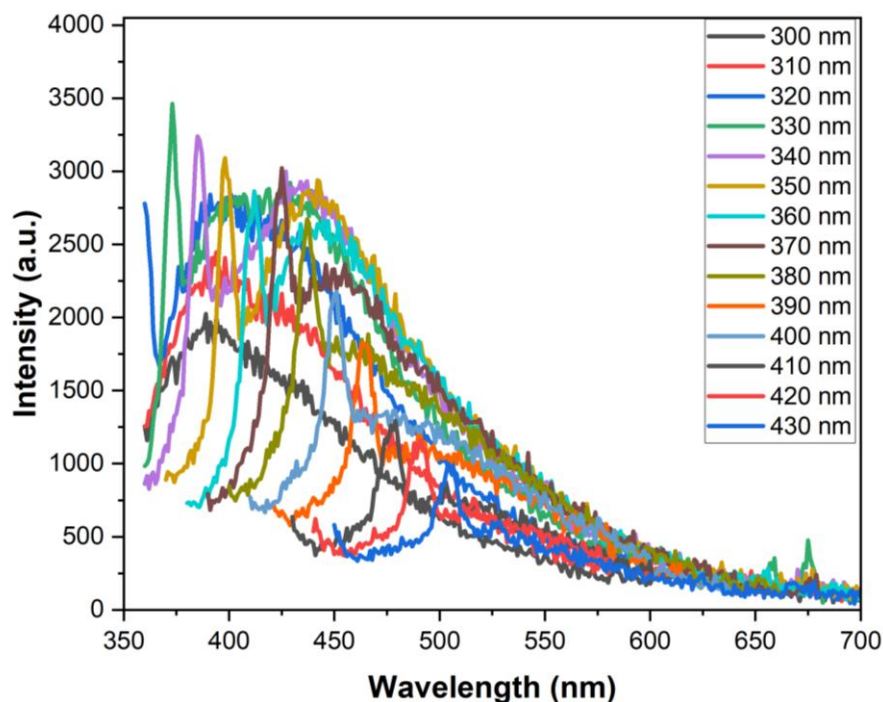


Figure 4 Photoluminescent spectra of carbon quantum dot solutions, excited by different wavelengths.

The analysis of photoluminescence spectra and CIE color coordinates for CQD solutions revealed a remarkable phenomenon: strong absorption of ultraviolet (UV) radiation and the emission of green light, which is beneficial for plant photosynthesis. It is well known that blue, red, and far-red light within the solar spectrum are crucial for plant photosynthesis, whereas UV and green light are less significant for plants. Notably, UV radiation on Earth has increased in recent years due to the depletion of the ozone layer caused by the release of harmful chemicals into the environment. Thus, the harmful impact of UV radiation could be mitigated and the photosynthesis efficiency of plants may be enhanced if UV light was converted into blue, red, and far-red light. As a result, the crop production may be improved. The study results on the influence of carbon quantum dots on the growth of several crops are discussed in application of the CQD solution to a few crops.

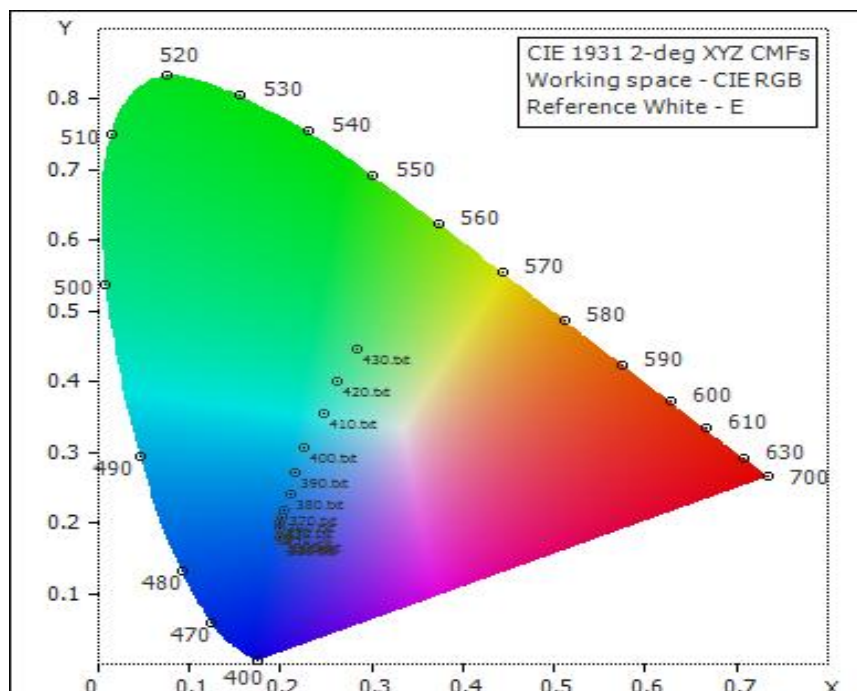


Figure 5 CIE color coordinates of carbon quantum dot solutions, excited by different wavelengths.

Effects of concentration on the luminescent characteristics of carbon quantum dots solutions

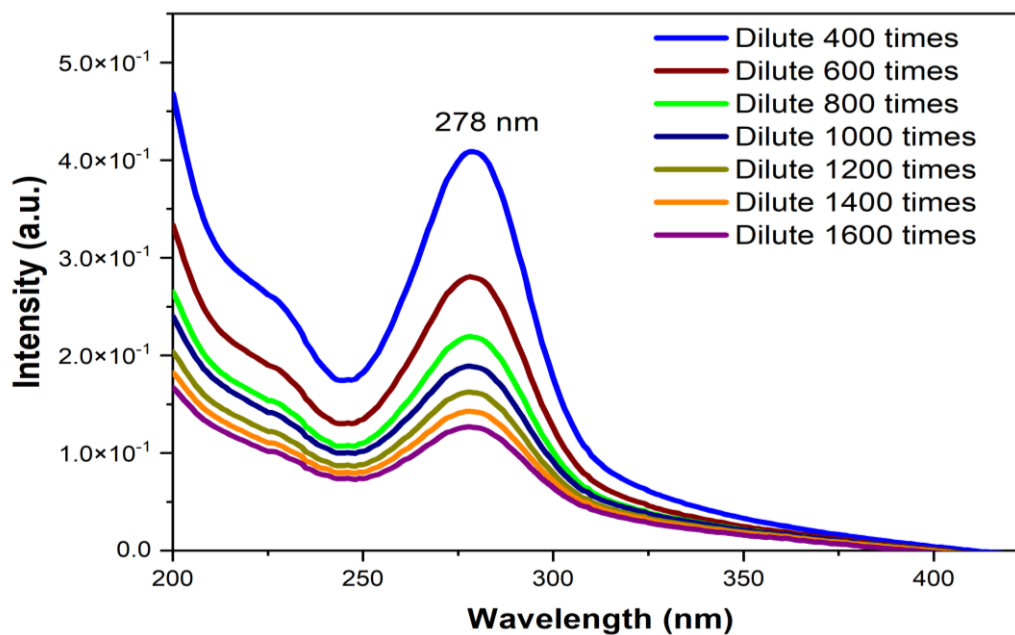


Figure 6 Absorption spectra of carbon quantum dot solutions at different concentrations

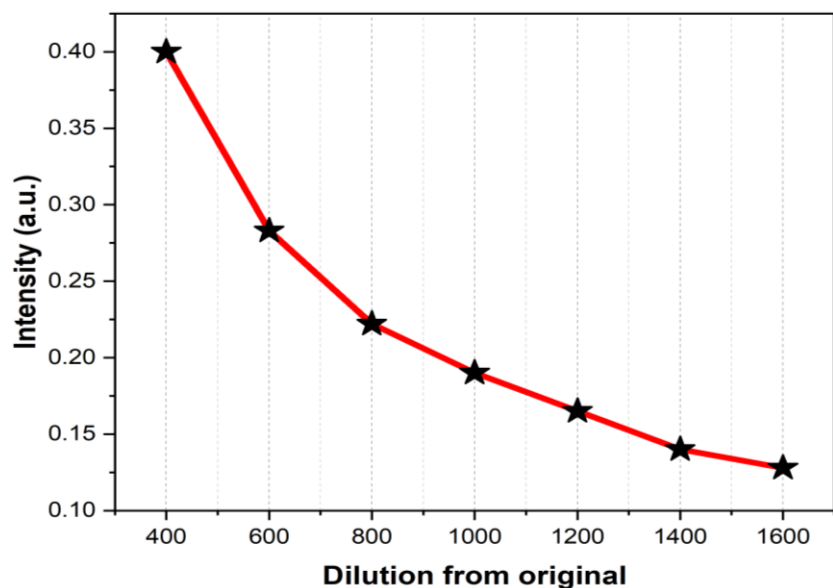


Figure 7 Dependence of absorption intensity and concentration of CQD solutions.

To further investigate the luminescent characteristics of the CQD material, absorption spectra and photoluminescence spectra of the diluted carbon quantum dot solutions were recorded. All the absorption spectra exhibit a broad peak with a maximum at 278 nm, extending into the visible region (Figure 6), with characteristic peaks of the $n \rightarrow \pi^*$ transition of the C = O bond (Mohammed et al., 2023). Figure 7 shows the dependence of the intensity of the peaks at 278 nm on the dilute concentration of the carbon quantum dot solutions. It shows that the intensity decreased as the concentration of carbon quantum dots decreased.

Figure 8 shows the PL spectrum of the diluted CQD solutions, excited by a wavelength of 340 nm. All these spectra exhibit a broad band with a maximum of 436 nm and a narrow line at 387 nm. As the concentration of CQD was reduced, the luminescent intensity decreased, but the spectrum profile remained unchanged. This observation indicates that the luminescent intensity depends on the concentration of CQD.

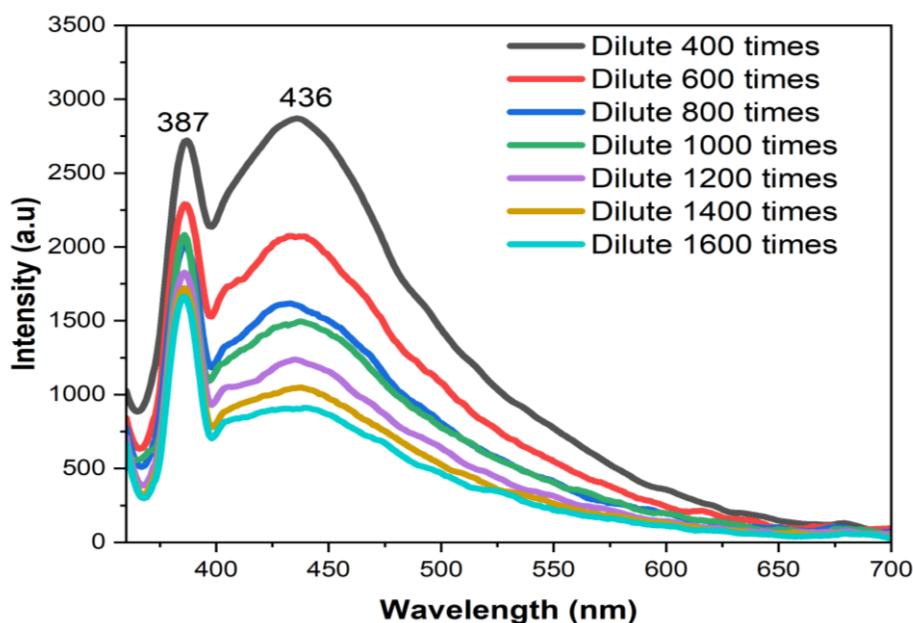


Figure 8 Luminescent spectra of CQD solutions at different concentrations, excited by a wavelength of 340 nm

Application of the CQD solution to a few crops

To investigate the effect of CQD solutions on the growth of crops, CQD solution was applied to a few crops including green bean sprouts, morning glory, and green mustard. After collecting experimental data for each type of crop, we applied the Kruskal-Wallis statistical method to compare the indicators of stem length, root length, and total mass across the experimental treatments. The P-value with less than 0.05 indicated a significant difference between the experimental groups. This result confirmed the effectiveness of the CQD formulations. The Kruskal-Wallis statistical method is a non-parametric test used to compare the medians of more than two independent groups (McKight and Najab, 2010; Ostertagova et al., 2014; Lelwala et al., 2024; Wani and Nagaraj, 2022; Meléndez et al., 2024).

Application of CQD solution to green bean sprout

In the experiments, five samples containing 8 grams of green bean seeds were planted in freshwater or CQD solutions at concentrations ranging from 100 mg/L to 250 mg/L, with an interval of 50 mg/L. Figure 9 illustrates the effect of these CQD solutions on the growth of green bean sprouts over four days.



Figure 9 The growth of green bean sprouts planted in freshwater and those grown in CQD solutions with different concentrations for four days: (a) 0 mg/L; (b) 100 mg/L; (c) 150 mg/L; (d) 200 mg/L; (e) 250 mg/L.

Table 1 The effects of research formulations on the growth of stem length, root length, and total mass of the green bean.

Samples	Stem length (cm)	Roots length (cm)	Total mass (g)
Freshwater	6.40a ± 0.59	3.43a ± 0.48	57.38a ± 4.19
CQD - 100mg/L	8.42b ± 0.67	4.23b ± 0.74	65.71b ± 6.80
CQD - 150mg/L	8.51b ± 0.70	4.40bc ± 0.71	67.63b ± 8.32
CQD - 200mg/L	9.40c ± 0.92	4.71c ± 0.74	70.20b ± 10.45
CQD - 250mg/L	9.11c ± 0.83	4.51bc ± 0.68	69.11b ± 10.03
P – value (Kruskal Wallis)	0.000	0.000	0.000

Note: a, b, and c indicate the levels of difference between formulations at a significance level of 0.05; formulations with the same letter do not differ.

The effects of the experimental formulations on the growth of bean sprouts were clearly reflected in the measurements of stem length, root length, and total mass. The Kruskal-Wallis analysis ($P < 0.05$) revealed significant differences among the experimental groups. For stem length, the Freshwater control group exhibited the lowest average (6.40 ± 0.59 cm), which was significantly different from the CQD-treated groups. At CQD concentrations of 100 mg/L and 150 mg/L, stem length increased to 8.42 ± 0.67 cm and 8.51 ± 0.70 cm, respectively, while the highest value was observed at 200 mg/L (9.40 ± 0.92 cm), exceeding those of the other CQD concentrations. Similarly, root length in the Freshwater group (3.43 ± 0.48 cm) was significantly shorter than in the CQD-treated groups. At CQD concentrations of 100 mg/L and 150 mg/L, root length increased to 4.23 ± 0.74 cm and 4.40 ± 0.71 cm, respectively (Table 1).

The longest root length was recorded at 200 mg/L (4.71 ± 0.74 cm), although it was not significantly different from that observed at 250 mg/L (4.51 ± 0.68 cm). A similar trend was observed for total mass. The Freshwater group exhibited the lowest biomass (57.38 ± 4.19 g), while samples treated with CQD concentrations of 100 mg/L and 150 mg/L reached 65.71 ± 6.80 g and 67.63 ± 8.32 g, respectively. The highest biomass was recorded at 200 mg/L CQD (70.20 ± 10.45 g), which, however, was not significantly different from the biomass at 250 mg/L (69.11 ± 10.03 g). Overall, the growth of bean sprouts was significantly enhanced due to the addition of CQDs. The optimized dosage of CQD was 200 mg/L and this confirmation is reflected through the superior growth in stem length, root length, and total mass. The CQD concentration of 250 mg/L did not obtain any further significant improvements.

Application of CQD solution to morning glory plants

The effect of CQD concentration on the growth of morning glory plants after 11 days of cultivation is shown in (Table 2). In this experiment, 4 grams of morning glory seeds were used in each experimental group. The concentration of CQD solution ranged from 100 mg/L to 400 mg/L, with an interval of 100 mg/L.



Figure 10 The 11-day growth of morning glories cultivated at different concentrations of CQD solution: (a) 0 mg/L; (b) 100 mg/L; (c) 200 mg/L; (d) 300 mg/L; (e) 400 mg/L.

Table 2 The effects of research formulations on the growth of stem length, root length, and total mass of the morning glory.

Samples	Stem length (mm)	Roots length (mm)	Total mass (g)
Freshwater	$65.32a \pm 6.08$	$40.24a \pm 6.15$	$32.22a \pm 5.67$
CQD - 100mg/L	$70.12a \pm 6.14$	$43.52ac \pm 6.70$	$39.52b \pm 5.52$
CQD - 200mg/L	$85.40b \pm 7.11$	$49.64b \pm 7.28$	$41.18bc \pm 5.07$
CQD - 300mg/L	$98.90c \pm 7.00$	$54.94d \pm 6.54$	$45.54c \pm 6.82$
CQD - 400mg/L	$78.82b \pm 7.51$	$47.24bc \pm 7.51$	$40.56b \pm 5.42$
P – value (Kruskal Wallis)	0.000	0.000	0.000

Note: a, b, c, and d indicate the levels of difference between formulations at a significance level of 0.05; formulations with the same letter do not differ.

The study investigating the effects of experimental formulations on the growth of morning glory plants highlighted significant differences among the test groups in stem length, root length, and total mass. Kruskal-Wallis analysis ($P < 0.05$) verified that CQD treatments significantly enhanced the growth metrics of morning glory plants compared to the Freshwater control group. In terms of stem length, the Freshwater formulation recorded the lowest average value (65.32 ± 6.08 cm), while CQD treatments, especially at a concentration of 300 mg/L, achieved the highest result (98.90 ± 7.00 cm). Concentrations of 200 mg/L (85.40 ± 7.11 cm) and 400 mg/L (78.82 ± 7.51 cm) also demonstrated marked improvements over the Freshwater group.

Similarly, root length was minimal in the Freshwater group (40.24 ± 6.15 cm) but peaked at 54.94 ± 6.54 cm with the 300 mg/L CQD concentration, outperforming other treatments. A comparable trend was observed for total mass. The Freshwater group achieved the lowest value (32.22 ± 5.67 g), whereas the 300 mg/L CQD treatment resulted in the highest biomass (45.54 ± 6.82 g). The 200 mg/L concentration obtained a slightly lower value of (41.18 ± 5.07 g). When the CQD concentration was up to 400 mg/L, the additional improvement cannot be detected when the biomass was (40.56 ± 5.42 g). Consequently, the CQD concentration of 300 mg/L was suggested as the optimal dosage for the best biomass improvement. In conclusion, among different dosage of CQDs, it is recommended that 300 mg/L was the most suitable concentration that promote the significant growth of morning glory plants.

Application of CQD solution to green mustards

The impact of CQD concentration on the growth of green mustard after 10 days of cultivation is summarized in (Table 3). In this study, 1 gram of green mustard seeds was utilized for each trial. The CQD solution concentrations varied from 100 mg/L to 400 mg/L, increasing in 100 mg/L increments. The green mustards were watered twice daily, in the morning and afternoon, with 10 ml of either freshwater or the corresponding CQD solutions.



Figure 11 The 10 - day growth of green mustards cultivated at various concentrations of CQD solution: (a) 0 mg/L; (b) 100 mg/L; (c) 200 mg/L; (d) 300 mg/L; (e) 400 mg/L.

Table 3 The effects of research formulations on the growth of stem length, root length, and total mass of green mustards.

Samples	Stem length (mm)	Roots length (mm)	Total mass (g)
Freshwater	$60.54a \pm 5.43$	$40.12a \pm 5.83$	$5.80a \pm 0.81$
CQD - 100mg/L	$69.52b \pm 8.01$	$41.20a \pm 6.12$	$6.22a \pm 0.66$
CQD - 200mg/L	$70.92b \pm 8.56$	$43.22a \pm 6.07$	$7.60b \pm 0.66$
CQD - 300mg/L	$80.52c \pm 6.53$	$55.22b \pm 8.41$	$9.51c \pm 1.16$
CQD - 400mg/L	$73.12b \pm 5.44$	$42.32a \pm 6.73$	$8.20bc \pm 0.70$

P – value (Kruskal Wallis)	0.000	0.000	0.000
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Note: a, b, c indicate the levels of difference between formulations at a significance level of 0.05; formulations with the same letter do not differ.

Research investigating the effects of different formulations on mustard green growth identified significant variations among experimental groups in terms of stem length, root length, and total biomass. The Kruskal-Wallis analysis ($P < 0.05$) confirmed that CQD-based formulations markedly enhanced plant growth compared to the Freshwater control. Regarding stem length, the Freshwater formulation yielded the lowest value (60.54 ± 5.43 mm), whereas CQD at a concentration of 300 mg/L exhibited the highest value (80.52 ± 6.53 mm), outperforming all other treatments. CQD concentrations of 100 mg/L (69.52 ± 8.01 mm) and 200 mg/L (70.92 ± 8.56 mm) also significantly improved, though they were less effective than the 300 mg/L concentration. For root length, Freshwater again showed the lowest measurement (40.12 ± 5.83 mm), while CQD at 300 mg/L achieved the highest value (55.22 ± 8.41 mm), demonstrating its superior ability to promote root development.

However, CQD concentrations of 100 mg/L and 200 mg/L produced results similar to Freshwater, with no statistically significant differences observed. The impact of CQD on total biomass was equally remarkable. Freshwater produced the lowest biomass (5.80 ± 0.81 g), while CQD at 300 mg/L delivered the highest value (9.51 ± 1.16 g), significantly surpassing all other concentrations including at 400 mg/L. Thus, CQD with 300 mg/L concentration was taken into consideration as the optimal dosage. In conclusion, CQD at a concentration of 300 mg/L was identified as the most effective for enhancing mustard green growth across stem length, root length, and total biomass. This concentration is recommended to maximize mustard plant yields under cultivation conditions.

4. CONCLUSIONS

Carbon quantum dots were successfully created from rice husks through a hydrothermal synthesis process. Their application positively influenced the growth of green bean sprouts, morning glory, and green mustard, as indicated by measurable increases in stem length, root length, and total mass. This study provides a method for synthesizing CQDs from safe, low-cost, and non-toxic organic precursors, making it suitable for future research on interactions between CQDs and other plant species or their potential applications in fields such as biomedicine, environmental science, and optoelectronics. Furthermore, scaling up the production of CQDs from rice husks and exploring potential industrial applications represent promising directions for future research.

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Author Contributions

Conceptualization: Do Thanh Tien and Tran Duc Loc; Investigation: Pham Thi Bao Uyen, Tran Quang Vu, Nguyen Van Hai, Nguyen Van Phong; Methodology: Pham Viet Hung, Vo Cong Anh, Ve Quoc Linh, Dao Le Minh Tuan, Do Thanh Tien; Software: Dao Le Minh Tuan, Do Thanh Tien; Supervision: Do Thanh Tien; Validation: Pham Viet Hung, Vo Cong Anh, Dao Le Minh Tuan, Do Thanh Tien; Visualization: Do Thanh Tien, Ve Quoc Linh, Vo Cong Anh, Dao Le Minh Tuan; Writing – original draft: Do Thanh Tien; Writing – review and editing: Pham Viet Hung, Vo Cong Anh, Ve Quoc Linh. All authors have reviewed and approved the final version of the manuscript.

Ethical Considerations

The ethical guidelines for plants & plant materials are followed in the study for collection & experimentation.

Informed consent

Not applicable.

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Conflict of Interest

The author declares that there are no conflicts of interests.

Data and materials availability

All data associated with this study are present in the paper.

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