



Research Article

Carica papaya-Derived Carbon Nanodots for the Detection of Fe (III) Ions

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Abstract

Carbon dots (CDs) possess distinctive optical and electronic properties as well as dimensions smaller than 10 nm, making them a unique category of carbon-based nanomaterials. They have been widely utilized across various domains including sensors, photocatalysis, biomedicine, and optoelectronics. This study investigates the use of a one-step hydrothermal synthetic approach to produce nanocarbon dots derived from *Carica papaya* seeds. Through the application of sophisticated characterization methods, the structural properties of the carbon nanoparticles were verified. These techniques included UV-visible absorption spectroscopy, fluorescence spectroscopy, Fourier transform infrared spectroscopy, and high-resolution transmission electron microscopy (HR-TEM). The photoluminescence emission of carbon dots (CDs) has been found to depend on excitation, as determined by photoluminescence (PL) spectroscopy. This study has explored the interaction between various metal ions and the photoluminescent properties of CDs, revealing a particularly noteworthy interaction with Fe (III) ions. The Stern-Volmer equation is utilized to examine the extinction mechanism linked with the sensing capability of carbon dots, resulting in the establishment of a recognition threshold of 0.36 μM . The existence of surface functional groups, which enable the formation of complexes with Fe (III) ions is a primary factor contributing to the sensing capabilities observed. This paper explores the fabrication and advancement of environmentally friendly sensor systems for detecting metal ions in biomedical and environmental contexts.

Keywords: Carbon nanodots, *Carica papaya*, CDs, Green synthesis, Fe (III) ion detection, Fluorescence sensing

1 Introduction

Carbon dots (CDs), also known as fluorescent carbon nanoparticles, represent the latest addition to the nanocarbon category, showcasing distinctive optical and chemical properties. The significant interest in these intriguing nanoparticles is warranted due to their remarkable biocompatibility, fluorescence characteristics, water solubility, chemical stability, and thermal robustness [1]–[4]. CDs have garnered considerable

attention owing to their rapid emergence across diverse fields including bioimaging, bio- and chemosensing, photocatalysis, and biolabeling. This rapid adoption can be attributed to their initial and prominent reports [5]–[8]. Given the prevailing environmental context, there is a strong recommendation for the advancement of eco-friendly synthesis approaches. The accessibility of natural resources facilitates the production and synthesis of carbon dots, thereby contributing to environmental welfare [9], [10]. The

utilization of environmentally friendly solvents in the synthesis of carbon dots closely adheres to the principles of green chemistry [11]. In the context of effective property control, the ability to modify carbon sources proves to be advantageous. Among numerous synthesis methods, the hydrothermal approach distinguishes itself for its effectiveness, providing the chance to incorporate surface functional groups like amino, hydroxyl, or carboxylic groups. This method facilitates the bonding of CDs with particular biological or environmental target molecules, making it highly applicable [12]–[14].

CDs have been reported to sense metal ions, such as heavy metal ions such as Iron (III), Mercury (II), Copper (II), Chromium (VI), Lead (II), Arsenic (III), Cobalt (II), Aluminum (III), Silver (I), and Gold (III), which are prominent environmental pollutants [15]. CDs derived from lotus roots [16], bamboo leaves [17], and honey [18] have been reported for the detection of Hg (II). Sources includes Tulsi leaves [19], and potato dextrose agar [20] were developed for the detection of Pb (II). Radhakrishnan *et. al.*, used edible cactus derived CDs for the detection of As (III) while Zao *et al.*, developed CDs based sensors for Co (III) from algal sources [21], [22]. Papaya waste pulp has been used for label free detection of Cr in water [23]. Reports on the future perspectives on CDs as optoelectronic devices are also reviewed previously [24].

Within the environment, ferric ion Fe (III) stands as a pivotal and irreplaceable metal ion. Its abundance is noteworthy. Operating significantly, these ions uphold numerous biological processes. Numerous reports demonstrate the effectiveness of CDs sourced from various origins in sensing environmentally and biologically relevant metal ions and biomolecules through both turn-off and turn-on mechanisms. Excessive intake of heavy metal ions poses significant health risks due to their non-degradability and bio-cumulative effects. Among these metal ions, ferric ions Fe (III) hold particular importance from both biological and environmental perspectives. The presence of an excessive amount of Fe (III) ions can pose threats to ecological sustainability and human health. Altering the optimal levels of ferric ions has the potential to disrupt cellular processes, potentially leading to Conditions such as Alzheimer's disease, Parkinson's disease, heart failure, inflammation, and hemochromatosis [25]–[27]. Consequently, the precise and selective identification of Fe (III) ions holds significant importance within biological applications.

This research paper discusses the synthesis of carbon dots derived from *Carica papaya* seeds, using

a straightforward, environmentally friendly, and cost-effective hydrothermal synthesis technique. Papaya being an abundant and renewable source can be considered as a sustainable source for the synthesis of CDs. Utilizing papaya, which is often considered a low-cost agricultural product, reduces the overall cost of synthesizing carbon nanodots compared to traditional methods that may rely on expensive precursors. Additionally, it put forward advantages, such as cost effectiveness, straight forward-greener preparation methods, high versatility in surface functionalization, etc. owing to which utilization of papaya can potentially lead to a high yield of carbon nanodots, enhancing the efficiency of the production process [28]. The CDs developed from papaya seeds evinced selectivity towards Fe (III) ions, highlighting their sensitivity with an impressive detection limit of 0.36 μM . This emphasizes their potential not only in biological applications but also in environmental technologies related to water, particularly for detecting ferric ions. To the best of our knowledge, the application of carbon dots obtained from *Carica papaya* seed extract for ferric ion detection has not been investigated previously.

2 Experimental

2.1 Materials and Methods

Carica papaya was acquired from a vegetable market in Malleshwaram, Bangalore, Karnataka, India. The following chemicals were procured from Sigma Aldrich: Copper sulfate pentahydrate, Cobaltous sulfate heptahydrate, Ferric chloride, Mercuric chloride, Sodium chloride, Zinc nitrate, Manganous sulfate monohydrate, Nickel sulfate, Magnesium sulfate, and Ferrous sulfate. Analytical-grade chemicals were utilized in this study in their as-purchased state. In all experimental procedures, double distilled water (DDW) was consistently used.

2.2 Synthesis of Carbon dots (CDs)

The papaya seeds underwent multiple washes with double distilled water (DDW) before being finely ground to obtain the extract. After meticulous filtration, a solution with a faint light brown color was obtained. The solution was subsequently transferred into a Teflon-lined autoclave after filtration. The process was conducted at a stable temperature of 180 °C and left to progress for a duration of 12 h. After the allotted reaction time, the solution was allowed to cool

to room temperature. Purification of the solution containing brown-colored carbon dots was accomplished through a series of purification procedures including filtration using Whatman filter paper, followed by multiple rounds of centrifugation utilizing both water and ethanol.

2.3 Detection of Fe (III) ions

The concentration of the dried CDs was adjusted to 10 mg mL⁻¹. In the experimental procedure, 3 mL of the carbon dots (CDs) solution was combined with a fixed volume of 300 μ L, containing various metal ion solutions (1 mL each), individually. These mixtures were then allowed to incubate for a duration of 10 min. Subsequently, fluorescence measurements were performed on all samples using an excitation wavelength of 380 nm.

2.4 Material characterization techniques

UV-visible absorption spectra were acquired using a UV-visible spectrophotometer (Specord 210 plus, Analytic Jena) featuring adjustable spectral resolution and a dual detection system, which includes cooling capabilities. Analysis using high-resolution transmission electron microscopy involved depositing a sample onto a copper grid coated with carbon. The analysis was performed utilizing an electron microscope (Thermofisher TALOS F200S G2) operated with a field-emission gun (FEG) at 200 kV, along with a CMOS camera boasting a resolution of 4K \times 4K pixels. Additionally, an in-column energy dispersive X-ray spectroscopy (EDS) detector was employed. The Fourier transform infrared spectrum (FTIR) was generated using a Perkin Elmer Spectrum 1000 spectrometer with a resolution of 1.00 cm⁻¹, operating across a spectral range from 350 to 4000 cm⁻¹. For photoluminescence (PL) measurements, a spectrofluorimeter (HITACHI F 2700) was employed. During the recordings, a uniform slit width of 5 nm was upheld, and the scanning rate for all measurements was established at 1500 nm per min.

3 Results and Discussion

3.1 UV-Vis absorption spectral analysis

The carbon dots (CDs) displayed their highest optical absorption peaks at wavelengths of 206, 211, and 216 nm, with a slight extension into the visible spectrum [28]. These three peaks are attributed to the $n-\pi^*$

transition of the $-C=O$ bond, which arises from the carboxylic and amide moieties present in CDs, accounting for the initial peak while the $\pi-\pi^*$ transitions of the $-C=C$ bonds in the carbon dots core are responsible for the second and third peaks. The spectral analysis reveals that the synthesized CDs absorb well in the UV region with their tail extending well beyond the wavelengths of the visible region. This information is a key parameter for understanding its electronic character and its application in photovoltaics. The blue shift occurred in the UV spectrum, compared with a few reports (with maximum absorption being observed at around 283 nm) also provides a chance of the formation of smaller sized CDs due to quantum confinement. The observations are similar to the previous reports and this supports the formation of CDs from papaya seeds via the hydrothermal method [29], [30]. When suspended in a liquid medium, the CDs appeared brown under sunlight exposure, and when illuminated with a UV lamp emitting at a wavelength of 350 nm, they emitted green luminescence, as depicted in Figure 1.

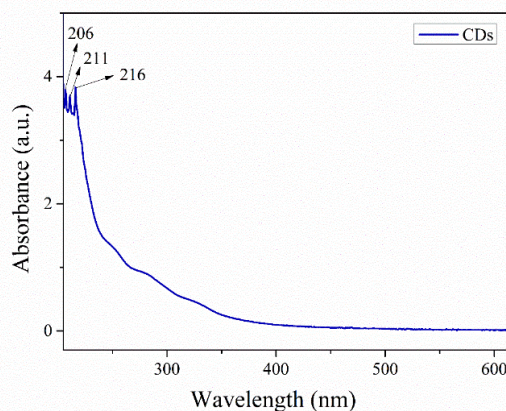


Figure 1: CDs spectra of UV-visible absorption.

3.2 High-Resolution Transmission Electron Microscopy Analysis (HR-TEM)

HRTEM captured images (Figure 2(a)–(c)) of carbon nanodots synthesized through the hydrothermal method, offering definitive proof of the formation of nanodots. The lattice spacing observed in the TEM image portrayed in Figure 2(b) corresponds to 0.31 nm, suggesting the presence of a graphitic carbon structure [31]. The graphitization of carbon precursors can occur in green synthesis if the reaction time is prolonged. Extended reaction times can allow for the development of more ordered carbon structures,

including graphitic carbon. Using carbon-rich precursors, such as certain fruits (papaya, for instance), biomass, or other organic materials with high carbon content can also enhance the likelihood of forming graphitic structures. Solvents that facilitate carbon-carbon bond formation, like water in hydrothermal synthesis as well as pH conditions may influence graphitic carbon formation [32], [33]. A particle size histogram (Figure 2(d)) was constructed based on TEM images, incorporating data from 50 nanoparticles. Examining this histogram reveals an average distribution of particle sizes around 5 nm.

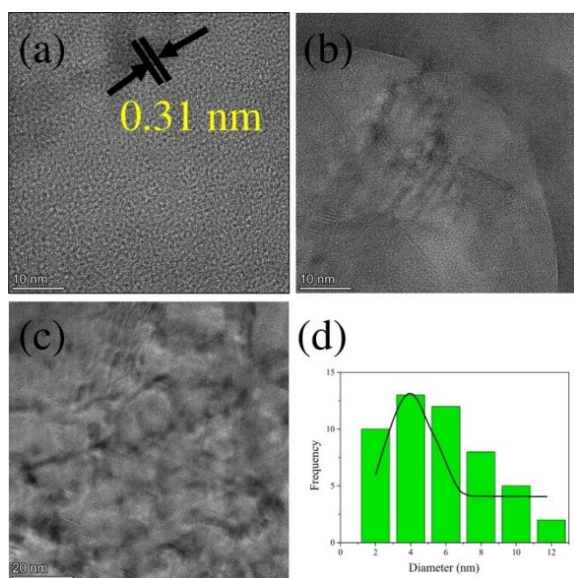


Figure 2: Images of carbon nanodots taken with a high-resolution transmission electron microscope (HRTEM) (a) Image showing lattice fringes of CDs at 10 nm magnification, CDs particles at varying magnifications (b) 10 nm, and (c) 20 nm. (d) A histogram showing the calculated particle sizes from TEM images.

3.3 Fourier Transform Infrared (FT-IR) analysis

Figure 3 presents the vibrational spectra of the green carbon dots, highlighting a prominent band within the 3400–3100 cm^{-1} range. This specific band is assigned to stretching vibrations linked to either $-\text{OH}$ or $-\text{NH}$ groups. The existence of a nitrile group is verified by a faint band detected within the range of 2200 to 2000 cm^{-1} . The presence of a carbonyl group within an

amide linkage is indicated by the emergence of a band at 1640 cm^{-1} . Moreover, the presence of planar bending vibrations associated with $-\text{CH}$ groups is evident from the emergence of bands falling within the range of 600–523 cm^{-1} . Thus, the comprehensive analysis of the vibrational spectra of carbon dots (CDs) reveals the presence of functional groups including $-\text{OH}$, $-\text{CO}$, and NH_2 within the synthesized CDs [34]–[36].

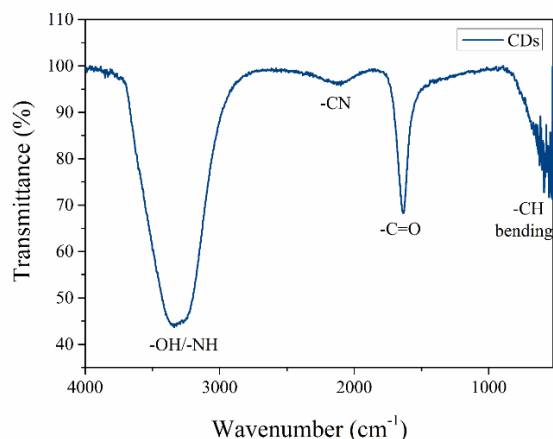


Figure 3: FTIR absorption spectrum of CDs

3.4 Photoluminescence Spectral (PL) analysis

To investigate the optical properties of carbon dots (CDs) synthesized using environmentally friendly methods, we performed photoluminescence analysis with an excitation wavelength of 320 nm, resulting in an emission peak centered at 380 nm. Additionally, a detailed examination of the fluorescence properties revealed that CDs exhibited the distinctive phenomenon known as “excitation-dependent emission” [37]–[40]. As shown in Figure 4, varying the excitation wavelength from 320 nm to 400 nm caused the emission peak to shift from 380 nm to 420 nm. Remarkably, the highest emission intensity was observed at an excitation wavelength of 380 nm and the details of the investigation are mentioned in Table 1. This discovery supports the synthesized CD ability to exhibit a diverse range of emission colors, including blue, green, and red, by simply adjusting the excitation wavelength [41]–[44]. This observation may be attributed to the optical selection of specific surface defect states near the Fermi level of nanodots, as confirmed by our FTIR spectroscopic analysis.

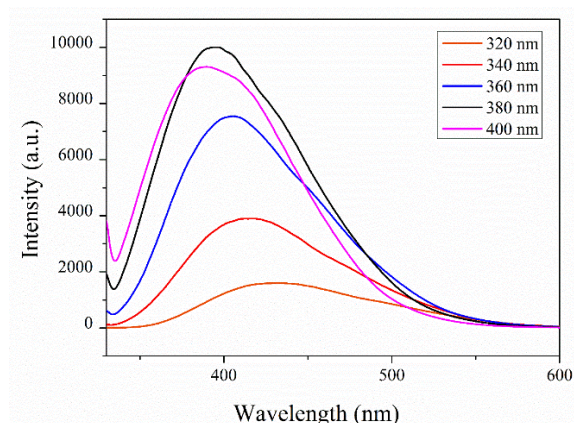


Figure 4: Photoluminescence spectra obtained for carbon dots excited at 320–400 nm wavelengths.

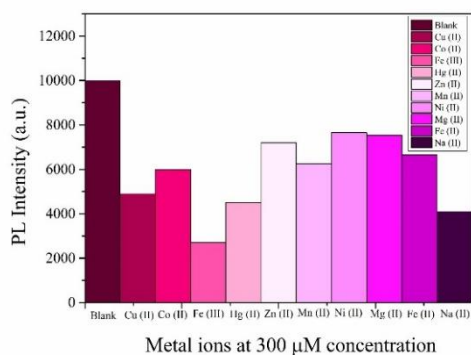


Figure 5: Interaction of metal ions under investigation at a concentration of 300 μM with the photoluminescence of CDs.

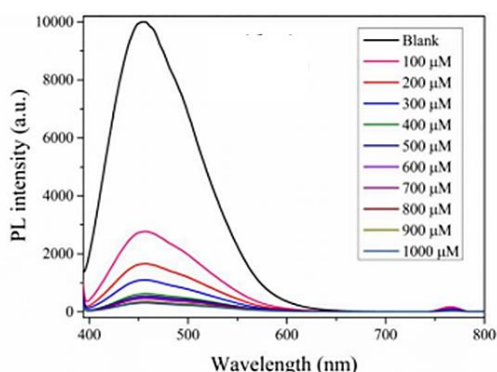


Figure 6: Fe (III) metal ions quenching the PL of CDs across the concentrations ranging from 100 to 1000 μM compared with a blank.

Table 1: The variation of emission wavelength with respect to the change in excitation wavelength of CDs.

Sl. No.	Excitation Wavelength (nm)	Wavelength at which Maximum Emission is Observed (nm)
1	320	380
2	340	389
3	360	401
4	380	412
5	400	420

3.5 Detection of Fe (III) ions

Fourier-transform infrared spectroscopy was employed to verify the existence of oxygen-containing functional groups on the surface of carbon nanodots. Following this, an examination was carried out to evaluate the influence of different metal ions on the photoluminescence of these carbon dots. Photoluminescence (PL) analysis was employed to examine the interactions between different metal ions and the photoluminescent properties of the carbon dots. The experiment involved the use of metal ions at concentrations of 300 μM, including Cu (II), Co (II), Fe (III), Hg (II), Zn (II), Mn (II), Ni (II), Mg (II), Fe (II), and Na (I) [45]–[47].

The concentration of carbon dots (CDs) remained constant throughout the experiment. In the control experiment, no metal ions were introduced. Figure 5 illustrates how different metal ions influence the photoluminescence intensity of carbon dots when present at a concentration of 300 μM [48]. Under the same experimental conditions, a control without any metal ions was also conducted. It is evident from Figure 5 that the impact of various metal ions other than Fe (III) ions on the photoluminescence intensity of CDs under excitation at 380 nm is negligible. The specificity for Fe (III) ions was demonstrated by a significant reduction in the photoluminescence emission peak intensity of the CDs compared to other metal ions [49]–[51].

This study aimed to investigate how different concentrations of Fe (III) ions affect the fluorescence quenching exhibited by carbon dots. The concentrations of Fe (III) ions were systematically adjusted across a range from 0 to 1000 μM, as illustrated in Figure 6. The figure shows that as the concentration of Fe (III) added to CDs increases, the intensity of fluorescence emission of CDs decreases exponentially. Figure 6 illustrates a graph with a blank (control), representing the baseline fluorescence intensity of the sample without any external influences, such as Fe (III) ions. This blank graph serves as a reference point for comparison to analyze

the effect of introducing Fe (III) ions on fluorescence intensity. With respect to blank intensity value, it is evident that the increase in Fe (III) concentration from 0–1000 μM results in the quenching of the emission of CDs.

The Stern-Volmer relationship was utilized to investigate the mechanism underlying fluorescence quenching and the corresponding plot can be seen in Figure 7 [52]. The R^2 value computed is found to be 0.9769 suggesting that the PL quenching does not strictly follow a static mechanism. Therefore, this observation implies that dynamic quenching plays a significant role in this sensor system and should not be completely disregarded [53]. The presence of oxygen-containing functional groups likely contributes to the observed fluorescence quenching, primarily due to their effective interaction with Fe (III) ions, as depicted in Figure 7.

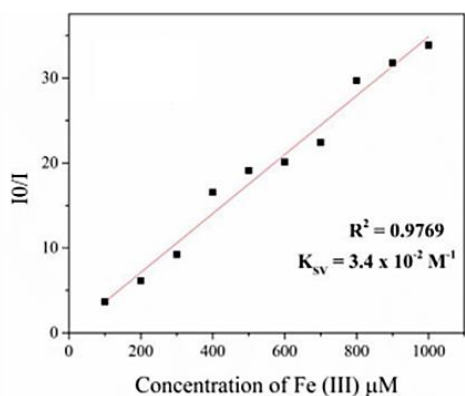


Figure 7: Stern-Volmer plot illustrating the correlation between the quenching constant and the concentration of the quencher.

The concentration of Fe (III) ions detected in this study is observed to be below the recommended guideline limit established by the World Health Organization, which is 5.36 μM . Achieving such a low detection limit as 0.36 μM for dynamic fluorescence quenching caused by Fe (III) ions necessitates carefully selecting a highly sensitive fluorophore, optimizing excitation and emission wavelengths, controlling environmental variables, creating calibration curves, reducing noise, maintaining quality control, preparing samples meticulously, and validating the method. This research suggests the potential for developing environmentally friendly sensors using carbon dots (CDs), which can be realized through thorough optimization and validation efforts [54].

4 Conclusions

To summarize, this work utilized a one-step process to synthesize carbon dots (CDs) using an environmentally friendly source, specifically the seed extract of *Carica papaya*. The size and the existence of distinctive surface functional groups on the carbon dots were validated through high-resolution transmission electron microscopy (HRTEM) and Fourier-transform infrared (FTIR) spectroscopy. To elucidate their optical properties, UV-visible absorption and photoluminescence analyses were conducted. These carbon dots exhibited favorable solubility in water. Additionally, their potential for detecting Fe (III) metal ions is investigated and found that they can sense and identify Fe (III) ions with a specific limit of detection (LOD) of 0.36 μM . The Stern-Volmer relationship was applicable for Fe (III) ion concentrations ranging from 0 to 1000 μM , with an R^2 value of 0.9769, indicating that the proposed sensor system involves both dynamic and static quenching processes. Any deviation from the ideal concentration of Fe (III) ions in the human body could disrupt various cellular functions, potentially leading to illnesses. Iron contamination in water sources can affect human health and the environment. Reliable sensors are needed for detecting and monitoring Fe (III) ions in drinking water as well as in effluent. Monitoring iron level is essential in food production and pharmaceutical formulations to ensure safety and to be in compliance with regulatory standards. Therefore, our work holds equal significance in biomedical, environmental monitoring and food/pharmaceutical industries.

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Author Contributions

G.P.: writing an original draft, conceptualization, research design, data analysis, investigation, reviewing, and editing; N.K.: investigation, research design, data analysis, methodology, reviewing, and editing; G.M.M.: research design, reviewing, and data

analysis; S.S.P.N.: conceptualization, research design and reviewing. All authors have read and agreed to the published version of the manuscript.

Conflicts of Interest

The authors declare no conflict of interest.

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