



Synthesis of Photoluminescent Carbon Dots from Rice for Metal Sensing and Dye Degradation

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ABSTRACT:

Carbon dots (CDs), commonly known as carbon quantum dots (CQDs), constitute an innovative category of zero-dimensional photoluminescent nano carbons, generally measuring less than 20 nm in diameter. CDs exhibit high selectivity in the detection of metal ions, small molecules, biopolymers, and various food components. These nanoparticles offer significant advantages owing to their superior biocompatibility, robust photoluminescent characteristics, and eco-friendly attributes, rendering them widely used in sensors, fluorescent inks, bio-imaging, and drug delivery systems. Initial investigations of rice types indicated the existence of carbon dots (CDs), leading to the formulation of this study aimed at synthesizing and characterizing CDs obtained from rice, as well as examining their utility in metal ion detection and dye degradation. The rice sample was air-dried at ambient temperature for two days, pulverized into a powder, filtered, and the filtrate was centrifuged at 4000 rpm before to lyophilization to get solid CDs. UV-visible spectroscopy was utilized to verify the effective production of the carbon dots. The manufactured CDs were examined using Scanning Electron Microscopy (SEM), Fourier Transform Infrared Spectroscopy (FTIR), X-ray Diffraction Analysis (XRD), and Photoluminescence (PL) Spectroscopy. These CDs were then applied in metal ion sensing and dye degradation, with the study primarily utilizing UV-vis and Photoluminescence spectroscopic techniques. The limits of detection and quantification for Fe³⁺ were 8.57 μM and 28.57 μM, respectively, while for Mn⁷⁺, they were 7.2 μM and 24.34 μM. The degradation percentages of Congo red, methyl orange, and methylene blue dyes were 80%, 95%, and 80%, respectively. Thus, the study successfully demonstrated the effective use of rice-derived CDs for metal ion detection and dye degradation.

INTRODUCTION

Nanoparticles have garnered significant attention since their discovery by Michael Faraday in 1857 during his investigation of thin gold films (Faraday, 1857). Nanoparticles are particles having size less than 10 nm

(Ahmad et al., 2024; Mumtaz et al., 2024). Their exceptionally high surface area-to-volume ratio distinguishes them from bulk materials, enabling unique properties (Christian, et al., 2008; Imran & Alsayeqh, 2022; Shahmoradi, et al., 2023). This extensive surface area facilitates the easy interaction and binding of target



molecules to the active surface sites (Lahiff, et al., 2010). Consequently, the surface of nanoscale materials plays a pivotal role in defining their distinct characteristics (Hamad & Salman, 2023; Christian et al., 2008). Nanoparticles, typically ranging from 1 to 100 nanometers (nm) in size, exhibit specific properties and functions that have captivated researchers' interest (Lahiff et al., 2010; Lalena, et al., 2020). Materials such as gold, carbon, and platinum have been used to synthesize nanoscale particles (de Dios & Díaz-García, 2010).

Carbon dots (CDs) are a novel class of photoluminescent nanocarbons, generally less than 20 nm in size (Sun et al., 2006), though some particles have been observed to reach up to 60 nm (Liu, Wu, et al., 2011). CDs are typically composed of nanocrystallites or sp² carbon, with oxygen or nitrogen-containing groups integrated into amorphous nanostructures (F. Yuan et al., 2016). These materials hold significant promise for various applications, particularly in fields involving toxic organic dyes and semiconductor quantum dots (QDs). This is due to their outstanding photostability, water solubility, and environmental friendliness, ease of synthesis, strong fluorescence, and optical properties. Carbon dots (CDs) have shown excellent biocompatibility for biomedical applications *in vivo*, including cell imaging, cancer treatment, drug and gene delivery, and disease detection, with several studies reporting no toxicity in various cell lines (Peng et al., 2017).

Rice samples contain many nutrients including amylose content (Faisal et al., 2023). The rice samples underwent a boiling process in water for a duration of 40 minutes to facilitate the synthesis of carbon dots (CDs). The boiled rice underwent a process of air-drying at ambient temperature for a duration of two days, subsequently being ground into a powdered form. Following this, 1.0 g of the powdered rice was combined with 10 mL of water and agitated for a duration of 40 minutes. The mixture underwent a filtration process, followed by centrifugation of the filtrate at a rotational speed of 4000 revolutions per minute. The resultant product underwent lyophilization to yield solid carbon dots. The successful synthesis of the carbon dots (CDs) was confirmed through UV

spectroscopy, which revealed a peak in the range of 270–280 nm, thereby indicating their presence.

Carbon dots (CDs) with a well-defined crystalline core exhibit photoluminescence (PL) primarily related to their size (Li et al., 2010). However, in most cases, the quantum confinement effect is not the dominant factor; instead, PL emission is largely governed by surface states and defects. The quantum yield (QY) of CDs varies based on the production methods and surface chemistries employed (Gan, et al., 2013). To date, the highest reported QY for CDs in solution is around 90%. Various mechanisms have been proposed to explain their photoluminescence properties, including the influence of surface functional groups, aromatic structures, size-related effects, carbon excitons, edge defects, free zigzag sites, and emissive traps. However, the precise source of photoluminescence in carbon dots (CDs) is still not fully understood (Wang et al., 2017).

Toxicity and biocompatibility are crucial concerns for the biological application of CDs. Most CDs demonstrate negligible toxicity at low concentrations (e.g., 10 µg/mL) and can degrade or be eliminated from the organism without significant harm (Nurunnabi et al., 2013). Nonetheless, their toxicity is influenced by factors such as concentration, molecular structure, and particle size. Higher concentrations may induce cytotoxic effects and interfere with organ function (Roy et al., 2015). CDs can also be phototoxic when exposed to light, due to the generation of reactive oxygen species (ROS) (Qin et al., 2015). In some instances, CDs may induce apoptosis and autophagy in cells upon photoexcitation, which holds potential for applications in photodynamic therapy (Chandra, et al., 2014).

In many instances, the core structure and surface functional groups of carbon dots (CDs) work together synergistically to influence various properties such as photoluminescence and photoinduced electron transport. These two components are often interdependent, but there are specific situations where one may play a more dominant role than the other. For example, the dispersibility of CDs is largely determined by their surface characteristics rather than the composition of the core. This is because the surface can be easily tailored through the introduction of different



functional groups, providing significant control over how well the CDs disperse in various solvents (Kang & Lee, 2019).

While the surface is key for properties like dispersibility, the carbon-like core of CDs plays a crucial role in ensuring their low toxicity, which sets them apart from traditional aromatic organic molecules. The presence of this carbon-based core enables CDs to maintain the desirable attributes of carbon materials, such as biocompatibility and stability, while also incorporating the versatile functional characteristics typical of organic compounds. This unique combination allows for a high degree of flexibility in modifying the chemical structure, physical properties, and functionalities of CDs, making them highly adaptable to a wide range of applications, from bioimaging to sensing and drug delivery (Kang & Lee, 2019). This adaptability is a key feature that drives the growing interest in CDs for both research and practical applications across multiple fields.

MATERIALS AND METHOD

Sample Collection

Rice samples were procured from a local market and thoroughly washed with distilled water to eliminate any possible contaminants.

Synthesis of CDs

The rice samples underwent a boiling process in water for a duration of 40 minutes, which facilitated the synthesis of carbon dots (CDs). The boiled rice underwent a process of air-drying at ambient temperature for a duration of two days, after which it was subsequently ground into a powdered form. Following this, 1.0 g of the powdered rice was combined with 10 mL of water and subjected to agitation for a duration of 40 minutes. The mixture underwent a filtration process, followed by the centrifugation of the resulting filtrate at a rotational speed of 4000 revolutions per minute. The resultant product underwent lyophilization to yield solid carbon dots. The successful synthesis of the carbon dots (CDs) was confirmed through UV spectroscopy, which

revealed a peak in the range of 270–280 nm, thereby indicating their presence.



a

b

Fig 01: A boiled rice

Characterization of CDs

The synthesized carbon dots (CDs) underwent extensive characterization using various analytical methods. Scanning Electron Microscopy (SEM) was employed to examine the surface morphology, while X-ray Diffraction (XRD) was used to determine the crystalline structure. Fourier Transform Infrared (FTIR) spectroscopy was applied to identify functional groups on the surface of the carbon dots. Additionally, UV-Visible (UV-Vis) and Photoluminescence (PL) spectroscopies were used to analyze the electronic structure and optical properties of the CDs.

Utilization of Carbon Dots for Metal Detection and Dye Degradation

The synthesized carbon dots were utilized for the detection of metals, specifically manganese and iron, with the analytical procedures conducted through UV-Vis and photoluminescence spectroscopies. Various concentrations of metal solutions ranging from 0.5 to 100 μM , each in a volume of 1 mL, were combined with diluted carbon dots, consisting of 3 mL of carbon dots and 10 mL of distilled water, at an optimal pH level. The mixtures underwent shaking for a duration of five minutes, subsequent to which UV-Vis and PL spectra were documented (Bu et al., 2016).



The catalytic activity of the synthesized carbon dots (CDs) was investigated in relation to model pollutants, specifically Congo red, methylene blue, and methyl orange dyes, as outlined in the existing literature (Zaib, Akhtar, Maqsood, & Shahzadi, 2021). In summary, a volume of 5 mL of the CDs solution was combined with 5 mL of the dye solution at the optimal pH level. The reaction mixture was situated within a sealed enclosure that contained a 100 W tungsten light source. Samples of 3 mL were collected at intervals of 10 minutes, and the degradation of the dye was assessed through the application of UV-Vis and PL spectrophotometry techniques.

RESULTS AND DISCUSSION

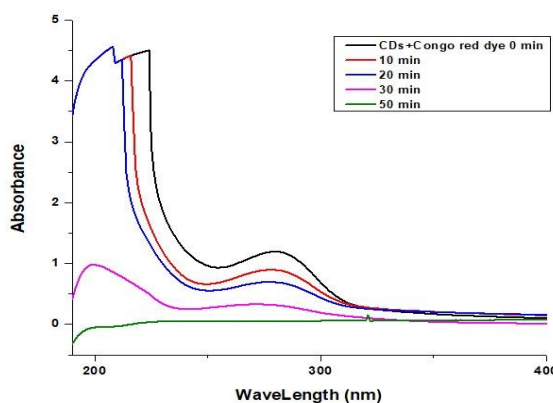
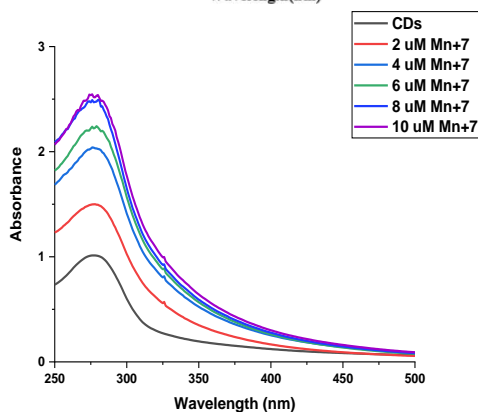
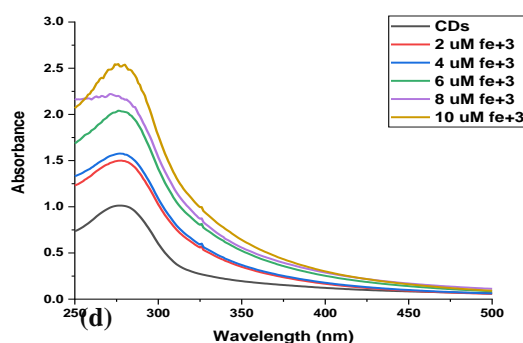
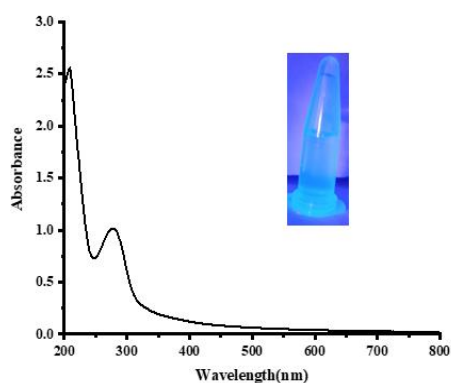
UV-Vis Spectral Analysis of CDs

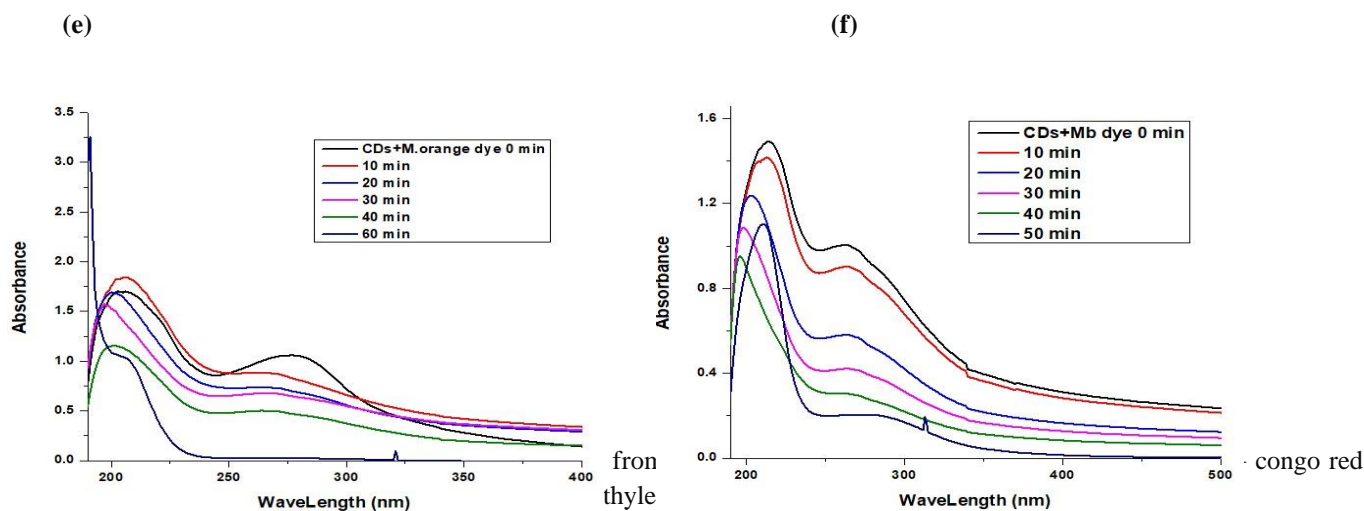
Ultraviolet-visible (UV-Vis) absorption spectrophotometry was employed to identify the characteristic peaks that confirmed the successful synthesis of carbon dots (CDs). Typically, the optical absorption spectra of CDs exhibit an extended tail from the ultraviolet (UV) region into the visible range. In the

visible spectrum, ranging between 400 and 800 nanometers, there is no significant absorption detected. However, in the ultraviolet region, spanning from 200 to 400 nanometers, notable absorption is observed, as illustrated in Figure 2 (a). Studies by R. Zhang and Chen (2014) and Zhao et al. (2015) demonstrated that fluorescent CDs in aqueous solutions present a prominent absorption peak near 280 nm, which is attributed to the π - π^* transitions of conjugated C=C bonds (C-C=C-C=C). When exposed to a UV lamp at 365 nm, CDs exhibit a strong blue fluorescence, with the peak excitation occurring around 340 nm, as depicted in the inset of Figure 2 (a) (W. Lu et al., 2015).

Furthermore, the absorption spectra of CDs in the presence of metal ions reveal a gradual increase in absorption intensity as the concentration of metal ions rises, as shown in Figures 2 (b) and (c) (Pu et al., 2021). This progressive enhancement in absorption intensity suggests an interaction between the CDs and the metal ions. Conversely, as displayed in Figures 2(d), (e), and (f), the absorption bands of CDs containing dyes gradually diminish over time, indicating the degradation of the dyes (Zaib, et al., 2021).

(a)





FTIR analysis

In the region of 400-4000 cm^{-1} , FTIR analysis was performed. The findings are illustrated in figure 4.2. According to Zhu et al. (2013), the stretching vibrations of OH and NH are responsible for the significant absorption peak that can be seen in Figure 3 at 3400 cm^{-1} .

According to Cui et al. (2015) and Zhang et al. (2016), the 2800 cm^{-1} band is caused by stretching vibrations, specifically C-H stretching vibrations. According to Wang et al. (2018) and Yuan et al. (2016), the stretching vibrations of C = O double bonds reach their

highest point at approximately 1600 cm^{-1} . It has been demonstrated by Rodrigues et al. (2015) that the presence of absorbance bands at 1250 cm^{-1} on the surfaces of CDs is indicative of the presence of C-H bending vibrations. According to Ahmed, Saifullah, Ahmad, Swami, and Ikram (2016), the peaks at 1000 cm^{-1} in the spectrum are representative of C-CO connections, also known as -C-O bonds. Due to the presence of these functional groups, CDs exhibit intense fluorescence when exposed to ultraviolet light. This is because functional groups are present. CDs are generated as a result of the observation of these various functional groups on their surface, which provided evidence that CDs are formed.

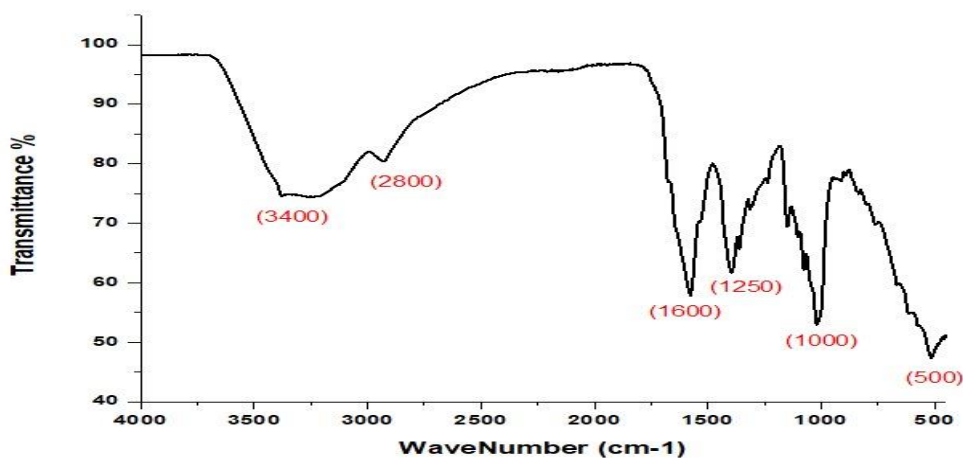


Fig 03; FTIR spectrum of synthesized CDs



4.3 XRD analysis

The X-ray diffraction (XRD) pattern of the synthesized carbon dots (CDs) derived from rice reveals the presence of two prominent peaks located at 2θ values of 29.54° , 31.34° , 40.85° , 54.54° , and 55.33° . These peaks are associated with Miller indices of 101, 100, 111, 101, and 100, respectively, and correspond to d-spacing values of 3.45 Å, 4.21 Å, 5.23 Å, 3.67 Å, and 8.14 Å, as

reported by Liu et al. (2015). The observed shift of these CDs can be attributed to the disordered characteristics of the sp^2 layer, which is a consequence of the high defect density within the carbon structure. This stands in contrast to the typical peak (002) associated with graphite structure, which is found at 26.5° (Miao et al., 2018; Vikneswaran, Ramesh, & Yahya, 2014). The packing factor signifies a fundamental cubic arrangement.

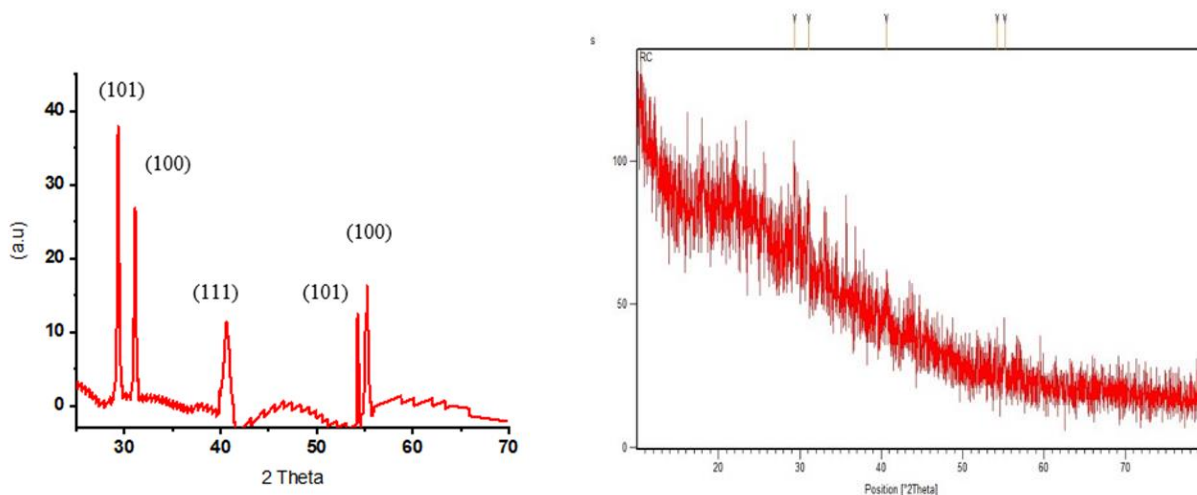


Fig 04: XRD pattern of synthesized CDs from rice

SEM analysis

The scanning electron microscopy (SEM) images demonstrated that the carbon dots (CDs) exhibited a spherical morphology, characterized by an average particle diameter of 2.79 nm, with a size distribution spanning from 1.5 nm to 5.5 nm. The CDs displayed distinctly defined lattice fringes, characterized by an average interplanar spacing of 0.34 nm, which closely corresponds to the 0.33 nm lattice spacing observed in the (001) diffraction facets of graphite. This observation indicates that the CDs exhibit a structure akin to that of graphite (Miao et al., 2018).

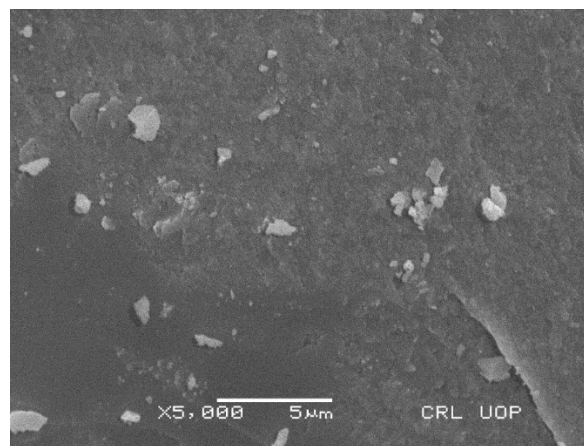


Fig 05: SEM image of synthesized CDs from rice

Effect of different metal ions solutions on CDs

To assess the impact of various cations on the fluorescence behavior of carbon dots (CDs), Pb^{2+} , Cu^{2+} , Cr^{3+} , Ni^{2+} , Co^{2+} , Fe^{3+} , and Mn^{7+} were chosen as



representative examples. As shown in Figure 4.5, the presence of Fe^{3+} and Mn^{7+} significantly reduced the fluorescence emission of carbon dots synthesized from rice, while the effects of the other cations were comparatively minimal. This indicates that the CDs demonstrate selective responsiveness to Fe^{3+} and Mn^{7+} ions in a cation-rich environment. Even under challenging conditions, the carbon dots maintain pronounced selectivity for Fe^{3+} and Mn^{7+} ions.

By incorporating the FT-IR analysis results, which identified hydroxyl, carboxyl, and amine groups on the surface of the CDs, it is inferred that these functional groups form stable complexes with Fe^{3+} and Mn^{7+} ions via carboxyl interactions. This binding results in fluorescence quenching, likely due to photo-induced electron transfer (PET) mechanisms, contributing to the selective recognition of Fe^{3+} and Mn^{7+} ions by the CDs, as highlighted in the 2017 study by Hu, Gong, Liu, and Choi.



Fig 06: CDs, CDs+ Pb^{2+} , CDs+ Cu^{2+} , CDs+ Cr^{3+} , CDs+ Ni^{2+} , CDs+ Co^{2+} , CDs+ Fe^{+3} , CDs+ Mn^{7+}

PL spectroscopy of CDs at different excitation

Another optical technique that substantiates the production of CDs is fluorescence spectroscopy, particularly through its unique emission analysis. The data presented in Figure 4 illustrates that the emission spectra of carbon dots are significantly affected by different excitation wavelengths. Upon excitation of the aqueous solution of rice-derived carbon dots at wavelengths spanning from 330 to 470 nm, a notable shift of the emission peaks toward longer wavelengths was observed. Concurrently, the fluorescence intensities

exhibited an initial increase followed by a subsequent decrease. The peak fluorescence was recorded at an excitation wavelength of 370 nm. This behavior suggests that CDs exhibit a property that is dependent on the level of excitation. As a result, in order to enhance the analysis of the fluctuations in carbon dot (CD) emission during investigations related to metal ion sensing and dye degradation, the CDs were subjected to excitation at a wavelength of 370 nm, which corresponds to the peak intensity of their fluorescence (Başoğlu, Ocak, &Gümrukçüoğlu, 2020). Another optical technique that substantiates the production of



CDs is fluorescence spectroscopy, particularly through its unique emission analysis. The emission spectra of carbon dots, as illustrated in Figure 4, exhibit a dependence on the variation of excitation wavelengths. Upon excitation of the aqueous solution of rice-derived carbon dots at wavelengths spanning from 330 to 470 nm, a notable shift of the emission peaks toward longer wavelengths was observed. Concurrently, the fluorescence intensities exhibited an initial increase followed by a subsequent decrease. The peak fluorescence was recorded at an excitation wavelength of 370 nm. This behavior suggests that CDs exhibit a property that is contingent upon the level of excitation. As a result, in order to enhance the analysis of the fluctuations in carbon dot (CD) emission during investigations related to metal ion sensing and dye degradation, the CDs were subjected to excitation at a wavelength of 370 nm, which corresponds to the peak intensity of their fluorescence (Başoğlu, Ocak, & Gümrükçüoğlu, 2020).

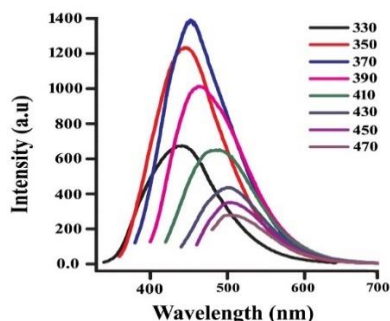


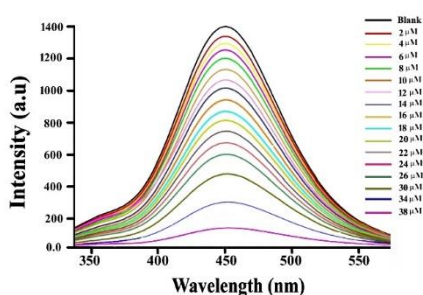
Fig 07: PL spectroscopy of synthesized CDs

Quantitative Study of CDs for Metal Detection and Dye Degradation Based on PL

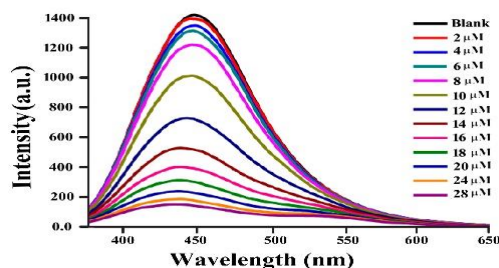
The investigation into the sensitivity of CDs for the detection of Fe^{3+} and Mn^{7+} ions was conducted utilizing Tris-HCl buffer solutions maintained at a pH of 7.0. The concentrations of Fe^{3+} and Mn^{7+} varied from 0 to 38 μM and 0 to 28 μM , respectively (Lu & Zhou, 2019). The data presented in Fig. 4.7 (a) and (b) demonstrate a notable decrease in the emission intensity of the carbon dots (CDs) as the concentration of Fe^{3+} ions is increased, thereby suggesting the presence of a quenching effect. A robust linear correlation was identified between the variable $(F_0 - F)/F_0$ and the concentration of Fe^{3+} , specifically within the concentration range of 10–22 μM , yielding a coefficient of determination (R^2) of 0.977. In the case of Mn^{7+} , the linear relationship was established within the concentration range of 8–16 μM , also resulting in a R^2 value of 0.977. Utilizing the three times standard deviation rule ($\text{LOD} = 3\sigma/s$), the limit of detection (LOD) for Fe^{3+} was ascertained to be 8.57 μM , whereas the limit of quantification (LOQ) was established at 28.57 μM . The limits of detection (LOD) and quantification (LOQ) for Mn^{7+} were determined to be 7.2 μM and 24.34 μM , respectively.

The research further demonstrated effective dye degradation over a specified duration, revealing a robust linear correlation for the degradation processes of methylene blue, methyl orange, and Congo red. The R^2 value for methylene blue was determined to be 0.98 within the time interval of 0 to 25 minutes. The R^2 value for methyl orange was determined to be 0.96 within the time interval of 4 to 22 minutes, whereas for Congo red, the R^2 value was recorded at 0.89 over the same time range of 4 to 22 minutes.

(a)



(b)



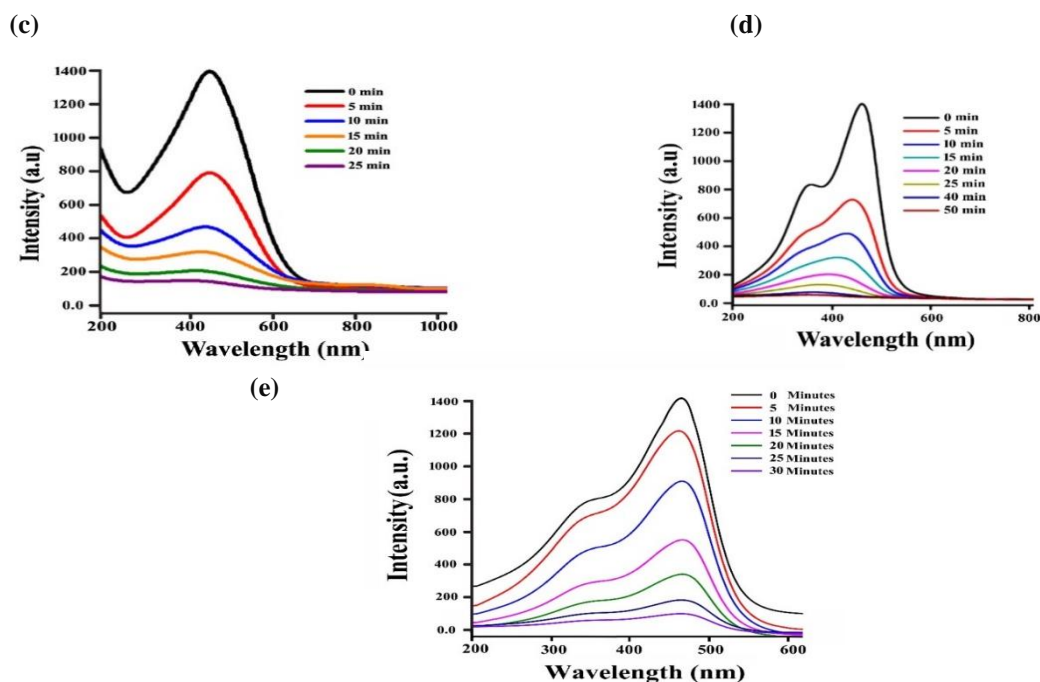
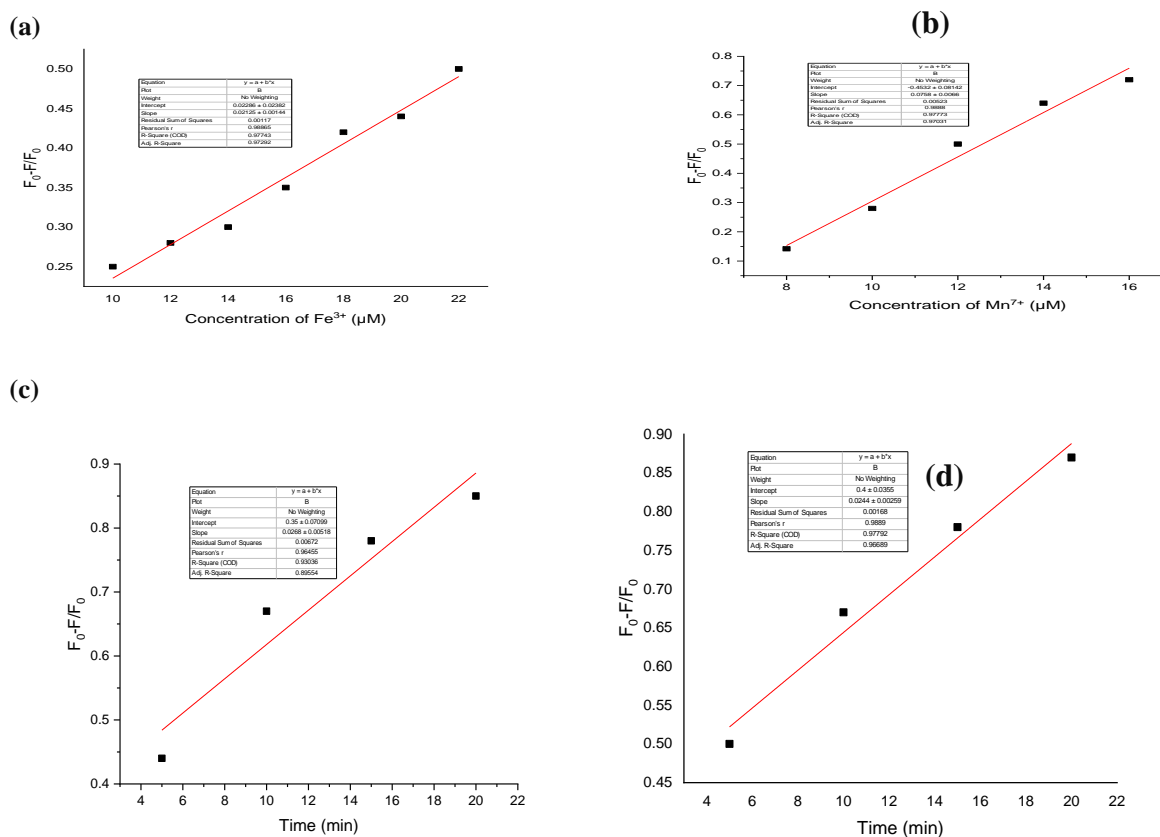


Fig 08: (a) The photoluminescence intensity of carbon dots in the presence of Fe^{3+} ions.(b) Photoluminescence intensity in the presence of Mn^{7+} ions.(c) The photoluminescence intensity of carbon dots with Congo red dye.(d) The photoluminescence intensity of carbon dots with methyl orange.(e) The photoluminescence intensity of carbon dots with methylene blue.



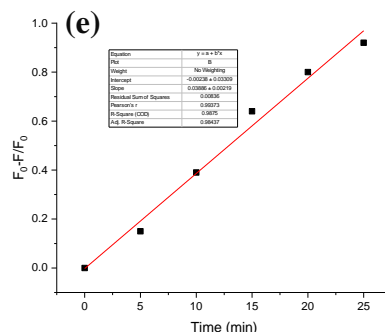


Fig 09: (a) The linear range associated with different concentrations of Fe^{3+} ions.(b) The linear range associated with different concentrations of Mn^{7+} ions.(c) The linear range observed during the degradation of Congo Red dye.(d) The linear range observed during the degradation of Methyl Orange dye.(e) The linear range observed during the degradation of Methylene Blue dye.

CONCLUSION

The introduction of oxygen- and nitrogen-containing functional groups onto the surface of carbon dots (CDs) has significantly improved their photoluminescence properties. These functional groups play a crucial role in enabling the carbon dots to selectively and sensitively detect Fe^{3+} ions. This detection mechanism is facilitated by the formation of stable ground-state complexes between the carbon dots and the Fe^{3+} ions, which promotes interaction and results in fluorescence quenching. The carbon dots were synthesized using a hydrothermal approach, which is known for its environmentally friendly nature, cost-effectiveness, and high efficiency. This method is particularly advantageous due to its straightforward setup, requiring minimal instrumentation, and its ability to produce results in a relatively short time frame. Moreover, these carbon dots have exhibited considerable promise as fluorescent sensors, demonstrating exceptional sensitivity in the detection of Fe^{3+} and Mn^{7+} ions. This highlights their practical potential in applications involving the sensing of metal ions, where their ability to accurately and efficiently detect these ions can be utilized for various analytical and environmental monitoring purposes.

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