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Research Article

Optimization of Nitrogen-Doped Carbon Dots from Tea Waste (*Camellia sinensis*) and Its Application for Metal Ion Sensing

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ABSTRACT
Carbon dots (CDs) were synthesized from tea waste extract (*Camellia sinensis*) as a carbon source via microwave irradiation, with the addition of urea as a nitrogen-doping agent. This study aimed to evaluate the effects of synthesis time and urea mass on the optical properties of CDs and their potential for metal ion sensing. CDs were synthesized with varying irradiation times and urea concentrations. CDs were characterized using a UV-Vis spectrophotometer, a photoluminescent spectrophotometer, and Fourier Transform Infrared (FTIR) spectroscopy to evaluate their optical properties and functional groups. The results showed that the optimal reaction conditions for tea waste extract-based CDs were 10 minutes of irradiation and 0.5 g of urea, yielding the highest fluorescence intensity. The FTIR spectra confirmed the presence of the main functional groups of hydroxyl (–OH) and carbonyl (C=O), which contribute to the stability and formation of CDs. Metal-ion sensing tests with CDs showed selectivity for Fe(III) with a detection limit of 5.91 mM.

Keywords: Carbon Dots, Tea Waste, Metal Ion Sensing

1. INTRODUCTION

Heavy metal pollution is a major environmental concern. Several metal ions, such as lead (Pb^{2+}), cadmium (Cd^{2+}), mercury (Hg^{2+}), chromium (Cr^{6+}), and iron (Fe^{3+}), are recognized as toxic and pose a threat to aquatic ecosystems and human health [1–2]. The detection of metal ions is important to control their presence in polluted environments. Various analytical methods, such as atomic absorption spectroscopy (AAS), inductively coupled plasma optical emission spectrometry (ICP OES), and flow injection analysis (FIA), have been widely used [3]. Although they have good sensitivity, these methods have several drawbacks, including high costs, complex sample preparation, and the need for special equipment and expertise. Therefore, fluorescence sensors have become one of the alternative methods widely developed in the detection of heavy metals. These sensors offer several advantages, such as

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simplicity and high sensitivity, enabling them to detect analytes at very low nanomolar concentrations [4].

Carbon dots (CDs) are one of the materials that can be applied as fluorescence sensors. Some of the interesting properties of CDs include their nano-scale size and good light resistance, which allows them to maintain a stable fluorescence signal. CDs are also known for their good compatibility, making them suitable for use in biological environments without causing harmful reactions [5–7]. In the synthesis of CDs, photoluminescence (PL) is an important aspect that can generally be controlled by particle size and surface conditions. The quality and optical properties of CDs are affected by various synthesis parameters, such as reaction media, carbon precursors, and dopant types [8].

There are two common methods used in CDs synthesis, which are the top-down and bottom-up approaches. The top-down approach involves breaking down larger carbon structures through several processes, such as chemical and electrochemical oxidation. Meanwhile, the bottom-up approach involves transforming carbon structures into CDs of the required size, typically via multiple treatments such as hydrothermal, solvothermal, ultrasonic, pyrolysis, and microwave irradiation [9]. The bottom-up approach is generally easier and allows the production of CDs with well-defined sizes and shapes. In the bottom-up approach, microwave irradiation is considered a suitable alternative for synthesis because it can provide sufficient energy to break chemical bonds in the precursor, achieve homogeneous heating in a shorter time, and produce CDs with a uniform particle-size distribution [8]. The use of natural materials rich in functional groups is expected to yield CDs with a high heteroatom content, thereby improving their optical properties [10].

One of the natural materials with potential as a precursor in CDs synthesis is tea waste. Tea (*Camellia sinensis*) is one of the most abundant agricultural products in Indonesia [11]. The high levels of tea production and consumption result in significant tea waste, most of which is not utilized. Tea waste is an abundant source of carbon, making it a potential precursor for CDs synthesis. The presence of natural phenolic compounds in tea, such as polyphenols with aromatic rings and phenolic hydroxyl groups, can enhance the optical properties of CDs. In tea waste, the total phenolic content has been reported to reach 172.42 mg GAE/g [12–13].

The usefulness of tea-based CDs is further demonstrated by their use in various applications, including as a heavy-metal ion sensor [14]. In addition to encouraging the

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utilization of waste, the use of tea waste for CDs synthesis is also expected to make it a potential material for nanotechnology and environmental applications. In contrast to previous studies, this work focuses on the systematic optimization of synthesis parameters, particularly microwave irradiation time and nitrogen doping level, and their influence on the optical properties and sensing performance of carbon dots derived from tea waste. This approach provides a deeper understanding of the relationship between synthesis conditions and functional performance, which is essential for the rational design of efficient and sustainable fluorescence sensors.

2. EXPERIMENTAL SECTION

2.1. Materials

The materials used include tea waste (*Camellia sinensis*), urea (CH_4N_2O), cadmium (II) chloride ($Cd(Cl)_2$), silver nitrate ($AgNO_3$), cobalt (II) nitrate ($Co(NO_3)_2$), copper (II) nitrate ($Cu(NO_3)_2$), iron (III) nitrate ($Fe(NO_3)_3$), manganese (II) nitrate ($Mn(NO_3)_2$), nickel (II) nitrate ($Ni(NO_3)_2$), lead (II) nitrate ($Pb(NO_3)_2$), and aquades.

2.2. Instrumentation

Synthesis of carbon dots was performed using a microwave (SAMSUNG MS20A3010AL/SE). The resulting carbon dots were characterized using a UV lamp, a UV-Vis spectrophotometer (PG Instruments), a Photoluminescence (Go Direct Fluorescence/UV-Vis Spectrophotometer), and an FTIR (Perkin Elmer Spotlight 200).

2.3. Procedure

2.3.1. Preparation and Extraction of Tea Waste

Tea waste was dried in an oven at 60 ℃ for 5 hours, then 5 g was extracted in 150 mL of water. Extraction was carried out on a hotplate at 70 ℃ for 30 minutes, then filtered using filter paper to obtain tea waste extract.

2.3.2. Synthesis of Carbon Dots (CDs)

A total of 10 mL of tea waste extract and urea at various masses (0, 0.5, 1.0, 1.5, and 2.0 g) were irradiated in a microwave (380 W) for 5, 10, 15, 20, 25, and 30 minutes. The black solid obtained from the reaction weighed 0.02 grams and was added to 20 mL of water. The mixture was stirred, then centrifuged to remove insoluble particles. The supernatant obtained was filtered using a 0.22 µm syringe filter [15–16]. The obtained CDs were then characterized.

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2.3.3. Characterization of Carbon Dots (CDs)

The formation of CDs was confirmed visually by fluorescence under a 365 nm UV lamp. Characterization was performed using a UV-Vis and Photoluminescence spectrophotometer to analyze the optical properties of CDs. An FTIR spectrophotometer was used to identify the functional groups of CDs.

2.3.4. Metal Ion Sensing

Metal ion solutions (Ag^{+}, Cd^{2+}, Co^{2+}, Cu^{2+}, Fe^{3+}, Mn^{2+}, Ni^{2+}, and Pb^{2+}) were prepared at a concentration of 10 mM each. Then, 0.5 mL of CDs was added to 0.5 mL of each metal ion solution. The change in fluorescence intensity was monitored under a 365 nm UV lamp and a PL spectrophotometer to assess the quenching effect of CDs upon the addition of metal ions. The selectivity and sensitivity of CDs were determined based on the results of observing the quenching effect and the most significant change in PL intensity compared to the control (aquades) [17].

3. RESULTS AND DISCUSSION

3.1. Effect of Irradiation Time

Irradiation time variations were performed to determine the optimal reaction time for the synthesis of CDs from a tea waste extract. Each synthesized sample was named based on the reaction time used: CDs-5, CDs-10, CDs-15, CDs-20, CDs-25, and CDs-30 for reaction times of 5, 10, 15, 20, 25, and 30 minutes, respectively. Each sample was synthesized by adding 1 g of urea as a nitrogen-doping source. The tea waste extract showed a brownish color, while the microwave-irradiated sample appeared nearly transparent under daylight (Figure 1a) and exhibited a color change, emitting bluish-green light under UV light (Figure 1b). When photons excite electrons in the CDs, the electrons transition to a higher energy level and subsequently return to the ground state, releasing excess energy as light through a process known as photoluminescence (PL) [18].

An initial evaluation was conducted by observing the fluorescence intensity of the synthesized CDs. Based on Figure 2, the PL spectrum shows that tea waste extract does not fluoresce. Fluorescence appeared in the CDs and its intensity gradually increased with irradiation time, reaching its peak at 10 minutes of irradiation. This indicates that a heating duration of 10 minutes through irradiation is the most effective reaction time to facilitate

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carbonization and the formation of the CDs luminescent centers. In the early stages of synthesis (CDs-5), the thermal energy generated by microwave irradiation is insufficient to form stable aromatic carbon cores.

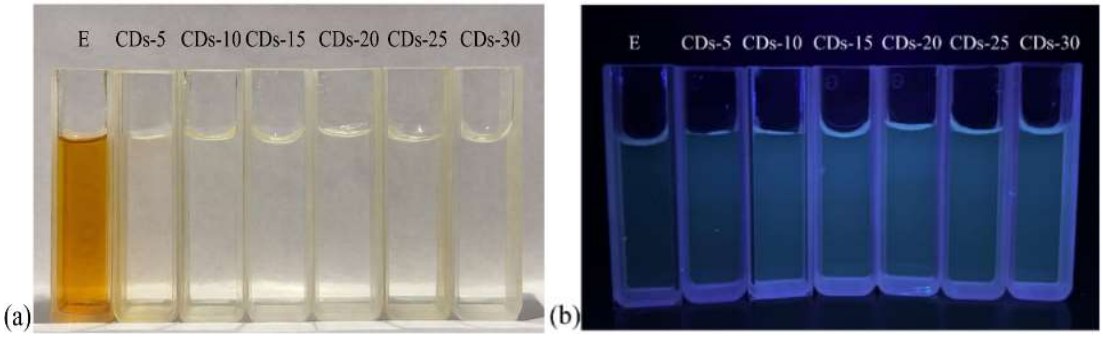

Figure 1. Samples of tea waste extract (E) and CDs under (a) daylight and (b) UV light.

The tea waste extract precursors exist as molecular fragments lacking a conjugated sp^2 structure, thus failing to form effective emission centers. This condition is characterized by low fluorescence intensity. Meanwhile, a significant increase in fluorescence intensity was obtained in CDs-10, indicating that the optimum synthesis conditions had been achieved. At this duration, the formation of carbon cores and surface passivation occurred in a balanced way. The microwave energy was sufficient to promote the aromatization and carbonization of organic fragments into sp^2 domains. This enabled efficient radiative electron-hole recombination, resulting in maximum fluorescence emission. The optimal synthesis time increases the frequency of emission trap formation due to changes in carbon core hybridization and π domains on the CDs surface [19].

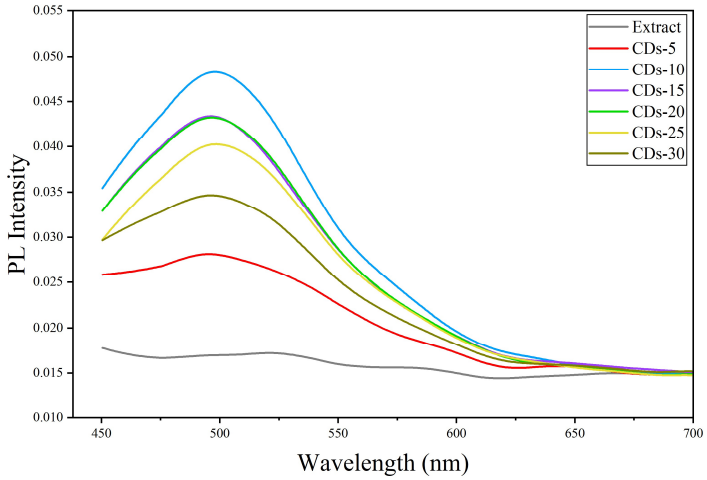

Figure 2. Emission spectra of CDs with various synthesis times.

However, a synthesis time of more than 10 minutes led to a gradual decrease in fluorescence intensity. This phenomenon is associated with excessive carbonization and

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particle growth, which tend to form larger aggregates, thereby quenching fluorescence. In addition, excessive heating can damage the surface passivation layer, leading to degradation of surface functional groups that are supposed to produce light emission. The loss of these active groups reduces the number of emission centers, thereby decreasing fluorescence intensity [20].

The optical properties of the CDs were also assessed by absorbance measurements at 200–500 nm. CDs generally have one or more absorption bands between 220–270 nm and 280–350 nm, which vary significantly depending on the synthesis process and the precursors used [21]. Based on the UV-Vis spectrum (Figure 3), all samples showed spectra similar to one another, with sharp absorption peaks at 220 nm and weak absorptions at 260–290 nm and ~337 nm. The sharp peak at ~220 nm indicates the presence of π→π* electronic transitions from C=C bonds representing sp^2 aromatic domains. Weak absorption around 260–290 nm and ~337 nm is related to n→π* transitions from carboxyl C=O bonds on the CDs surface [18,22].

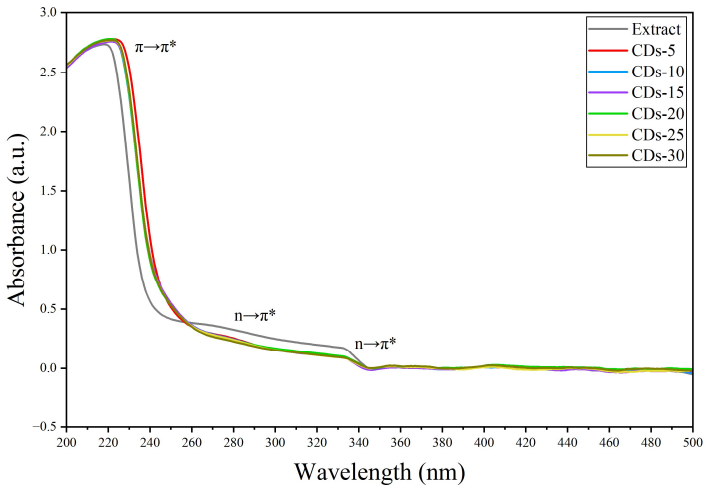

Figure 3. Absorbance spectra of CDs with various synthesis times.

The results of the FTIR analysis for the tea waste extract sample and the CDs-10 are shown in Figure 4. The FTIR spectrum of tea waste shows main absorption bands at 3327 cm^{-1}, 2916–2851 cm^{-1}, 1734 cm^{-1}, 1619 cm^{-1}, 1456 cm^{-1}, 1034 $cm^{-1,}$ which are associated with O–H/N–H, C–H, C=O, C=C, N–H/C–N, and C–O stretching vibrations, respectively. In the CDs sample, some of these absorption bands are still observed but have shifted and increased in intensity, particularly at 3282 cm^{-1} associated with O–H/N–H stretching vibrations, and at 1724 cm^{-1} and 1644 cm^{-1} associated with C=O and C=C stretching, respectively. In addition, in the CDs sample, clearer bands appear at 1566 cm^{-1} and 1413 cm^{-1}, which are related to N–H

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vibrations and C–N stretching. Other bands at 982 cm^{-1} to 655 cm^{-1} are associated with C–O strain vibration [14]. This is consistent with other studies on tea waste-based CD synthesis, which also exhibit these absorption bands [23].

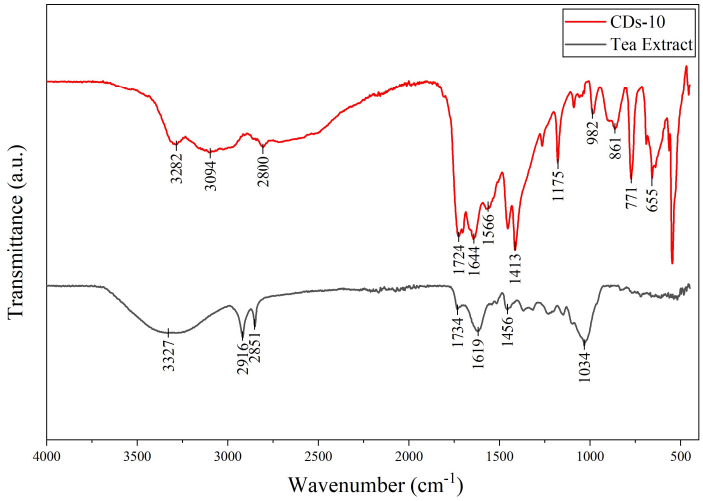

Figure 4. IR spectra of tea waste extract and CDs-10.

3.2. Effect of Urea Mass

Urea is used as a nitrogen-doping source in CDs. Therefore, the amount of urea used in the reaction affects the optical properties of the CDs formed. In this study, the urea doping mass was varied to analyze its effect on changes in the optical properties of CDs. CDs were synthesized without urea and with urea at 0.5, 1.0, and 2.0 g. The CDs obtained were then named CDs-U0, CDs-U0.5, CDs-U1.0, and CDs-U2.0. The CDs sample without urea addition showed a yellowish-brown color similar to the color of the initial extract. Meanwhile, the CDs samples with urea addition tended to appear colorless under daylight and emitted a blue-green color under UV irradiation (Figure 5).

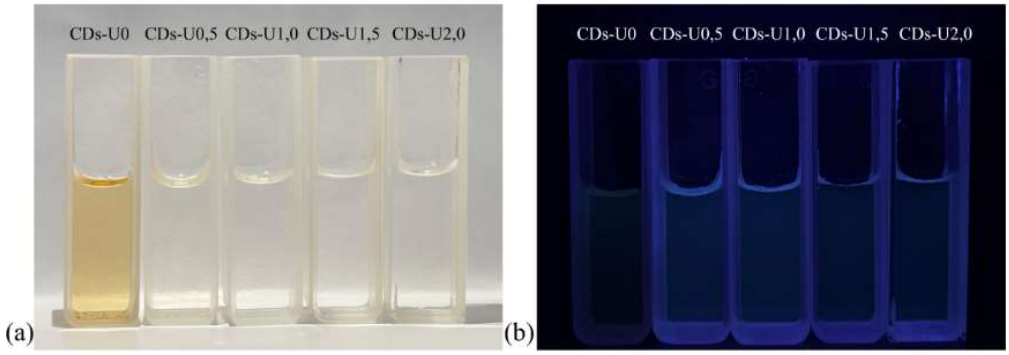

Figure 5. CD samples with urea mass variations under (a) daylight and (b) UV light.

Urea contains two amine groups (-NH_2) connected by a carbonyl functional group (-C=O). In addition to being a source of nitrogen, urea can act as a passivation agent, thereby

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increasing CDs fluorescence. During microwave heating, urea decomposes, and its nitrogen atoms participate in condensation and carbonization reactions, forming nitrogen-containing functional groups, such as amines and amides, on the surface of CDs [24]. Figure 6 shows the effect of urea addition on fluorescence intensity. Samples without urea addition (CDs-U0) showed the lowest PL intensity. The CDs samples then experienced an increase in PL intensity as the urea mass increased, reaching a peak at a urea mass of 0.5 grams (CDs-U0.5), which then decreased as the added urea mass increased. This is similar to other studies that observed a decrease in PL intensity with excessive urea doping, which is assumed to result from aggregation or self-quenching [25]. Urea can interact with epoxy and carboxyl groups to form amides and alcohols, creating double emission traps between π→π* transitions on the carbon core, thereby increasing fluorescence intensity. However, excess urea mass can also oxidize sp^2 domains, thereby increasing non-radiative recombination and decreasing fluorescence intensity [26].

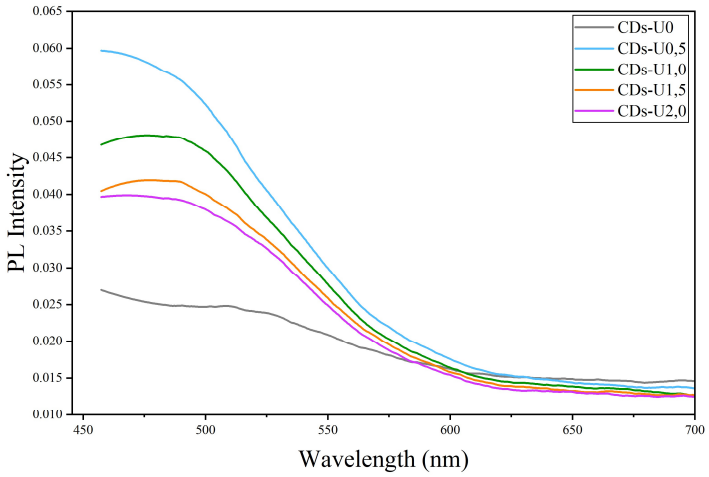

Figure 6. Emission spectra of CDs with varying urea mass.

The changes in the optical properties of CDs due to the addition of urea were also confirmed by the absorbance spectra shown in Figure 7. All samples exhibit a sharp peak at 220 nm, associated with the π→π* transition of the C=C bond. Meanwhile, the peak at around 275 nm indicates the n→π* transition of the C=O bond. There is a significant difference in the wave number, which shows that CDs samples without urea have a higher absorption peak than CDs samples with urea addition. This peak then decreases as the urea mass increases. This may occur because excess urea can interfere with the polymerization and carbonization processes of the carbon precursor, inhibiting sp^2 aromatic domain growth and leading to differences in the absorption peak. Excessively high urea concentrations can cause

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aggregation or surface defect formation, thereby reducing effective optical absorption and resulting in decreased absorbance [25].

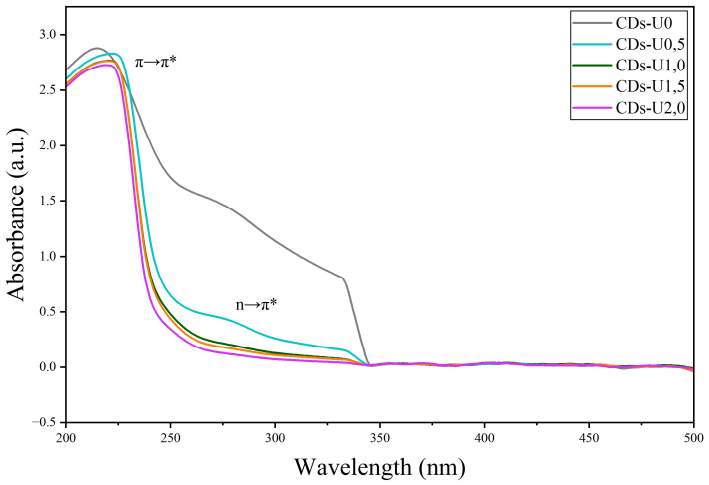

Figure 7. Absorbance spectrum of CDs with various urea mass variations.

Functional group analysis by FTIR of CD samples without urea showed main absorption bands at 3232 cm^{-1}, 2946 cm^{-1}, 1690–1606 cm^{-1}, 1538 cm^{-1}, 1448 cm^{-1}, 1340 cm^{-1}, 1230 cm^{-1}, 1174 cm^{-1}, 1033 cm^{-1}, 762 cm^{-1}, and 605 cm^{-1}. Meanwhile, the CDs sample with urea addition showed absorption bands at 3279 cm^{-1}, 2804 cm^{-1}, 1724 cm^{-1}, 1649 cm^{-1}, 1570 cm^{-1}, 1411 cm^{-1}, 1178 cm^{-1}, 773 cm^{-1}, 655 cm^{-1}, and 547 cm^{-1}. In both samples (Figure 8), the broad bands in the 3200–3400 cm^{-1} region are associated with O–H and N–H stretching vibrations, indicating the presence of carboxyl and amine groups on the CDs surface. The bands at 2946 cm^{-1} and 2804 cm^{-1} are related to C–H stretching vibrations, while the absorptions at 1724 cm^{-1} and 1690–1606 cm^{-1} are associated with C=O stretching vibrations, indicating the presence of carbonyl groups and conjugated structures in the carbon framework.

Furthermore, the bands at 1570–1538 cm^{-1} and 1411–1448 cm^{-1} are associated with C=C and C–N vibrations, indicating the presence of nitrogen-containing groups, especially in CDs-U0.5, which underwent urea addition. The bands in the 1230–1033 cm^{-1} and 1178 cm^{-1} regions are attributed to C–O–C, C–OH, and C–N stretching vibrations, while the bands at 773–547 cm^{-1} are related to C–H and C–O in accordance with the characteristics of carbon dots rich in oxygen and nitrogen groups [3,27]. Overall, the spectra of the CDs-U0.5 and CDs-U0 samples show several significant differences, indicating that the addition of urea successfully alters the nitrogen content on the CDs surface, as reported in other nitrogen-doped CDs studies [28].

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Based on the optimization results obtained, a urea doping mass of 0.5 g and a synthesis time of 10 minutes were selected as the optimal parameters for the synthesis of tea waste-based CDs and for further studies on their application in metal ion sensing.

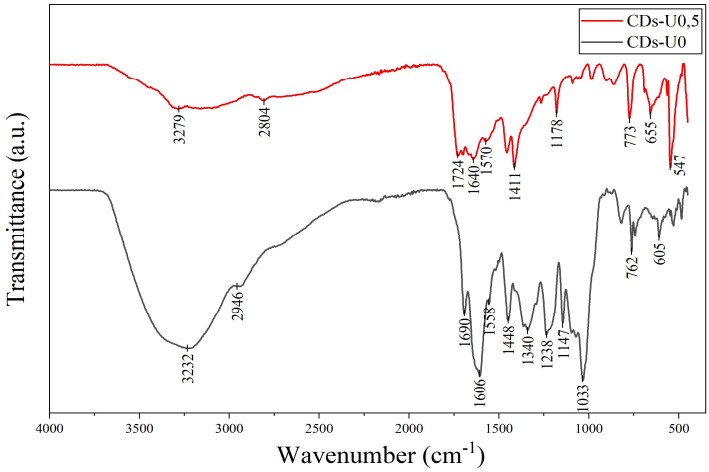

Figure 8. IR spectra of CDs without doping and with nitrogen doping.

Although the optical and spectroscopic analyses confirm the successful formation of carbon dots, it is acknowledged that further structural characterization, such as Transmission Electron Microscopy (TEM) and X-ray Diffraction (XRD), would provide more detailed information on particle size distribution, morphology, and crystallinity. Future work will focus on employing these techniques to obtain a more comprehensive understanding of the structural properties of the synthesized CDs.

3.3. Metal Ion Sensing

The potential of tea waste extract-based CDs as metal-ion sensors was tested against various metal ions, including Ag^{+}, Cd^{2+}, Co^{2+}, Cu^{2+}, Fe^{3+}, Mn^{2+}, Ni^{2+}, and Pb^{2+}, each at 10 mM. This test was conducted to evaluate the fluorescence intensity response of the CDs toward each metal ion. The selectivity of CDs toward various metal ions was evaluated by visual observation and analysis of the quenching response ratio (Figure 9).

Visually, each metal ion had a different effect on changes in CDs fluorescence intensity (Figure 9a). The strongest quenching response was observed in the CDs sample with the addition of Fe^{3+} ions. This observation was confirmed by the quenching ratio (F_0/F) diagram in Figure 9b, where F_0 and F represent the fluorescence intensities of the CDs in the absence and presence of metal ions, respectively. Based on the diagram, the F_0/F ratio for Fe^{3+} ions was the highest, indicating the strongest quenching response among the metal ions.

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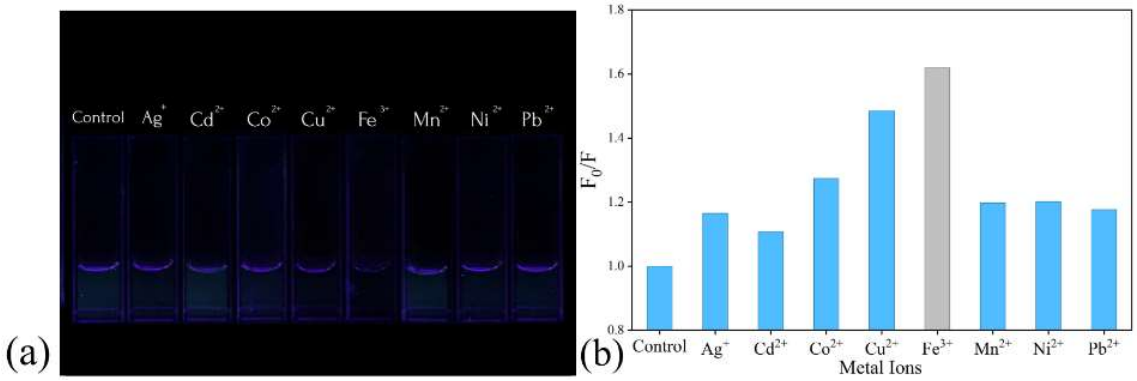

Figure 9. CDs selectivity test via (a) observation under UV light and (b) quenching ratio F_0/F against the addition of Ag^+, Cd^{2+}, Co^{2+}, Cu^{2+}, Fe^{3+}, Mn^{2+}, Ni^{2+}, and Pb^{2+} metal ions.

The PL intensity measurement results in Figure 10 show that the CDs with the addition of aquades (control) exhibited the highest fluorescence intensity. Meanwhile, the addition of Fe^{3+} ions produced the strongest quenching effect on the CD fluorescence, resulting in the greatest decrease in intensity. In contrast, the addition of Cd^{2+} ions resulted in a relatively small decrease in fluorescence intensity, so that the CDs continued to fluoresce at an intensity close to that of the control. The other metal ions showed decreases in intensity that were not significantly different from each other. These insignificant changes in intensity indicate that the CDs do not show strong selectivity for these metal ions, but rather are selective for Fe^{3+}.

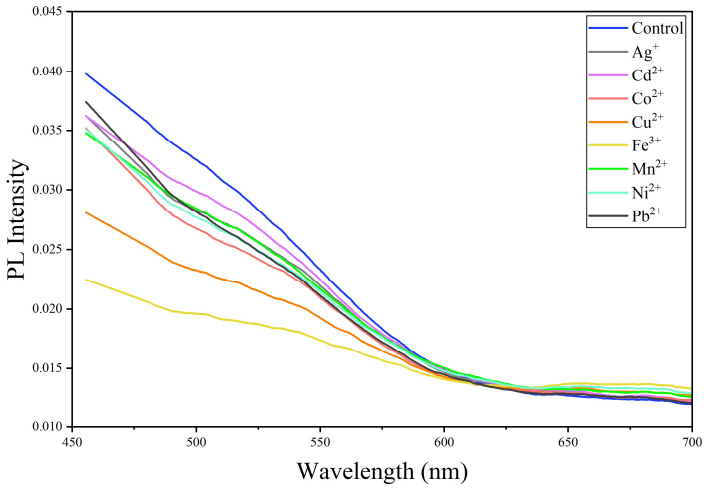

Figure 10. Emission spectra of CDs before and after the addition of various metal ions.

The quenching effect occurs when CDs electrons return to their ground state without emission. Metal ion detection by CDs involves major quenching mechanisms, such as complex formation or coordination, the Inner Filter Effect (IFE), Light-Induced Electron Transfer (LET), ion binding, and aggregation [29]. The surfaces of CDs contain functional groups containing oxygen (–OH and C=O) and nitrogen ($–NH_2$ and –CN) that can donate lone pairs of electrons

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to form coordination complexes with metal ions. More specifically, in tea waste-based CDs, quenching by Fe^{3+} metal ions is associated with LET interactions between functional groups on the CDs' surfaces and the metal ions. The hydroxyl (–OH) functional groups on the surface of CDs that interact with Fe^{3+} facilitate non-radiative electron transfer from the excited state, thereby quenching fluorescence [14]. The presence of hydroxyl (–OH) groups on the surface of CDs has been confirmed by FTIR spectra.

Furthermore, the sensitivity of CDs toward Fe^{3+} ions was evaluated by determining the limit of detection (LOD). Based on the measurement results (Figure 11), the relationship between fluorescence intensity and Fe^{3+} concentration produced a linear equation, y = 0.04082x−0.01929, with a correlation coefficient R^2 = 0.9622, where y represents $(F_0\text{-}F)/F_0$ and x is the Fe^{3+} concentration. From this linear relationship, the LOD was 5.91 mM. This indicates that the synthesized CDs sensor can detect the presence of Fe^{3+} ions in water at a minimum concentration of 5.91 mM.

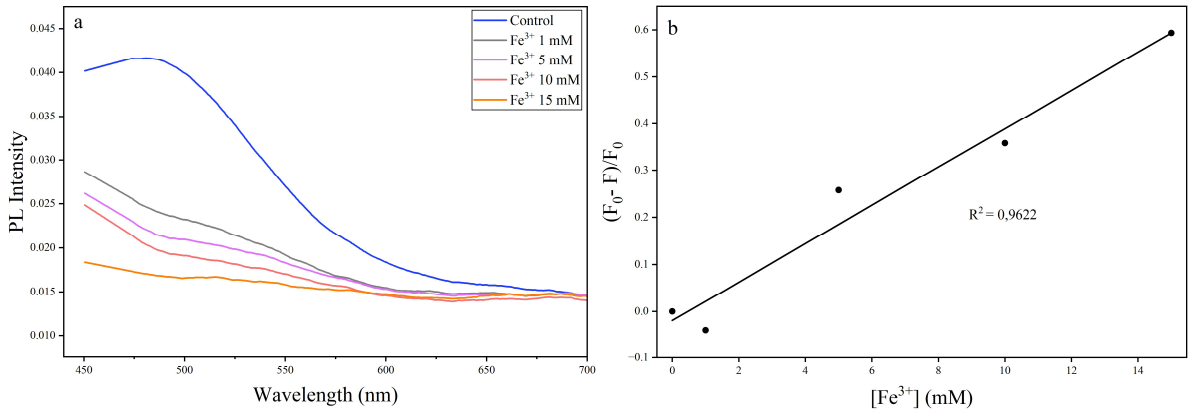

Figure 11. (a) CDs emission spectra with the addition of Fe^{3+} ions at various concentrations and (b) linearity of $(F_0\text{-}F)/F_0$ against Fe^{3+} ion concentration.

The obtained limit of detection (LOD) of 5.91 mM for Fe^{3+} is higher than those reported in many recent studies on carbon dots, where LOD values are commonly in the µM or even nM range. For instance, several biomass-derived nitrogen-doped carbon dots have demonstrated detection limits in the micromolar range, owing to optimized surface passivation and enhanced electron-transfer efficiency [30-32]. This difference in sensitivity can be attributed to variations in precursor composition, synthesis conditions, and the absence of further surface engineering or signal amplification strategies in the present study.

It is important to emphasize that the CDs developed in this work were synthesized via a simple, rapid, and environmentally friendly microwave-assisted method using tea waste as a

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renewable carbon source, without additional chemical modification or complex post-treatment. While this approach may yield lower sensitivity than more advanced systems, it offers significant advantages in sustainability, cost-effectiveness, and ease of preparation. Moreover, the synthesized CDs exhibit clear selectivity toward Fe^{3+} ions, indicating their potential applicability for preliminary or qualitative detection in aqueous systems.

4. CONCLUSION

The optimum conditions for the synthesis of CDs from tea waste extract using the microwave method were determined to be 10 minutes of irradiation and the addition of 0.5 g of urea, which produced CDs with the highest fluorescence intensity. The resulting CDs have high selectivity towards Fe^{3+} ions. This is indicated by the largest decrease in fluorescence intensity among other tested metal ions. Although the detection limit is relatively moderate, this study highlights a simple, rapid, and environmentally friendly approach for producing nitrogen-doped carbon dots from biomass waste with good selectivity toward Fe^{3+} ions. Further improvements in sensitivity and validation in real sample matrices, such as environmental water samples, will be explored in future studies to enhance practical applicability.

AUTHOR CONTRIBUTIONS

AEP and MA collaborated on the design and implementation of the experiment. AEP carried out the experimental work and drafted the manuscript, while MA supervised and reviewed the manuscript and edited the final version. All authors have reviewed and approved the final version of the manuscript.

CONFLICT OF INTEREST

The authors have no competing interests to declare relevant to this article's content.

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