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Synthesis and Optical Characterization of N-Doped Carbon Dots Derived from Citric Acid

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ABSTRACT

Carbon dots (CDs) derived from citric acid have gained significant attention because of their excellent photoluminescence properties and low toxicity. However, the optimization of nitrogen doping concentration to improve fluorescence efficiency remains insufficiently explored. In this study, CDs and nitrogen-doped carbon dots (N-CDs) were synthesized hydrothermally from citric acid using ethylenediamine (EDA) at concentrations of 5%, 10%, and 15% (v/v). The influence of EDA concentration on the optical properties and fluorescence efficiency of N-CDs was systematically investigated. FTIR analysis confirmed the presence of O–H/N–H, C=O, C–O, and C–N functional groups, indicating successful nitrogen incorporation into the carbon structure. UV–Vis spectra exhibited characteristic absorption peaks corresponding to π→π* and n→π* transitions, while PL analysis revealed blue emission in the range of 445–447 nm. Nitrogen doping enhanced fluorescence intensity and quantum yield through improved surface passivation and reduced non-radiative recombination. Among the synthesized samples, N-CDs with 10% EDA showed the highest quantum yield of 43.98%, indicating the optimum doping concentration. In contrast, excessive EDA addition at 15% slightly decreased optical performance because of possible defect formation and fluorescence quenching effects.

Keywords: Carbon Dots, Citric Acid, Ethylenediamine, Hydrothermal, Nitrogen Doping

ABSTRAK

Carbon dots (CDs) berbasis asam sitrat telah menarik perhatian besar karena memiliki sifat fotoluminesensi yang sangat baik dan toksisitas yang rendah. Namun, optimasi konsentrasi doping nitrogen untuk meningkatkan efisiensi fluoresensi masih belum banyak dikaji. Pada penelitian ini, CDs dan nitrogen-doped carbon dots (N-CDs) disintesis menggunakan metode hidrotermal dari asam sitrat dengan penambahan etilendiamina (EDA) pada konsentrasi 5%, 10%, dan 15% (v/v). Pengaruh konsentrasi EDA terhadap sifat optik dan efisiensi fluoresensi N-CDs dikaji secara sistematis. Analisis FTIR mengonfirmasi keberadaan gugus fungsi O–H/N–H, C=O, C–O, dan C–N yang menunjukkan keberhasilan inkorporasi nitrogen ke dalam struktur karbon. Spektrum UV–Vis memperlihatkan puncak serapan khas yang berkaitan dengan transisi π→π* dan n→π*, sedangkan analisis PL menunjukkan emisi biru pada rentang 445–447 nm. Doping nitrogen meningkatkan intensitas fluoresensi dan quantum yield melalui peningkatan pasivasi permukaan serta penurunan rekombinasi nonradiatif. Di antara seluruh sampel, N-CDs dengan 10% EDA menunjukkan quantum yield tertinggi sebesar 43,98% yang menandakan konsentrasi doping optimum. Sebaliknya, penambahan EDA berlebih pada 15% sedikit menurunkan kinerja optik akibat kemungkinan terbentuknya cacat permukaan dan efek fluorescence quenching.

Kata kunci: Carbon Dots, Asam Sitrat, Etilendiamina, Hidrotermal, Doping Nitrogen

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1. Introduction

Advances in nanotechnology over the past few decades have spurred the exploration of new nanocarbon materials with superior optical and chemical properties [1]. One material that has attracted attention is carbon dots (CDs), which are zero-dimensional carbon nanoparticles generally less than 10 nm in size [2] with a sp^2/sp^3 hybridized spherical structure that exhibits strong photoluminescence, high chemical stability, good biocompatibility, high quantum yield, and low toxicity [3]. These properties make CDs highly promising for various applications, such as sensors, bioimaging, optoelectronics, and photocatalysis [4].

CDs consist of a carbon core surrounded by surface functional groups, where the core is responsible for their stability, while the surface groups allow for chemical modification for specific applications [5]. CDs can be synthesized through various methods, generally classified into top-down and bottom-up approaches. Top-down methods can be performed via arc discharge, laser ablation, and chemical oxidation. These methods are costly, require sophisticated equipment, and produce hazardous waste such as strong acids. Bottom-up methods are preferred because they are simpler and more cost-effective. Bottom-up methods can be performed using microwave, pyrolysis, and hydrothermal techniques.

The hydrothermal method is widely used because it offers a simple, environmentally friendly, and rapid synthesis process that requires low energy and allows for better control over the structure and optical properties of the material. These advantages support the application of CDs in the fields of renewable energy and environmental remediation [6]. In this method, simple organic precursors such as citric acid are often used because they readily undergo carbonization and possess functional groups that support the formation of carbon cores.

Citric acid is a highly popular carbon precursor in CD synthesis due to its environmentally friendly nature, low cost, and tendency to undergo dehydration and polymerization during the heating process [7], [8]. Additionally, its chemical structure, rich in carboxyl groups, facilitates the formation of carbon cores as well as surface groups that play a crucial role in photoluminescence properties [9]. However, pure CDs often have limitations in quantum efficiency and emission stability, so modification strategies are needed to improve their performance [10]. Carbon dots derived from citric acid emit blue light [11].

One of the most effective approaches is heteroatom doping, particularly with nitrogen, to produce nitrogen-doped carbon dots (N-CDs). Nitrogen doping can increase electron density, introduce new energy levels, and improve the optical and electronic properties of CDs, such as increased fluorescence intensity and quantum efficiency. Nitrogen also plays a role in enhancing charge transfer capabilities, which is crucial in photocatalytic applications.

Ethylenediamine (EDA) is a commonly used nitrogen source in the synthesis of N-CDs because it contains two reactive amine groups that facilitate nitrogen incorporation into the carbon framework [12]. When combined with citric acid under hydrothermal conditions, condensation and carbonization reactions produce nitrogen-containing surface groups and molecular fluorophores that contribute to strong blue luminescence. Previous studies reported that citric acid–EDA systems can generate highly fluorescent CDs with improved optical properties and high quantum yields due to enhanced surface passivation and modified electronic states [13], [14]. Nevertheless, most previous studies mainly focused on synthesis optimization or application performance, whereas systematic evaluation of how different EDA concentrations influence fluorescence intensity and quantum yield remains limited.

The synthesis of CDs from citric acid and ethylenediamine generally involves dehydration, polymerization, and carbonization steps. In the initial stage, fluorophore molecules form, which subsequently develop into a conjugated carbon structure. As the temperature and reaction time increase, a transformation occurs from the molecular structure into a carbon core with sp^2 domains that play a role in the material optical properties. Therefore, synthesis parameters such as precursor ratio, temperature, and reaction time significantly determine the final properties of the resulting CDs [15]. Nitrogen doping can modify the electronic structure of CDs by introducing additional energy levels, increasing electron density, and improving surface passivation, which ultimately leads to enhanced photoluminescence intensity and quantum yield. Previous studies have reported that citric acid–EDA systems can produce highly luminescent CDs with significantly improved optical properties.

In addition, the optimum EDA concentration for balancing nitrogen incorporation and fluorescence efficiency has not been fully clarified. Excessive nitrogen precursor addition may introduce additional surface defects that promote non-radiative recombination and reduce fluorescence performance. Therefore, understanding the influence of EDA concentration on the optical characteristics of CDs is important for designing highly luminescent N-CDs.

Based on these considerations, this study aims to synthesize CDs and nitrogen-doped CDs using citric acid and different EDA concentrations of 5%, 10%, and 15% (v/v), selected to represent low, moderate, and high nitrogen precursor concentrations. The optical properties of the resulting materials were systematically investigated using FTIR, UV–Vis, and photoluminescence analyses to determine the optimum nitrogen doping concentration for improving fluorescence efficiency.

2. Materials and Methods

2.1. Materials

The materials used in this study included citric acid monohydrate (Merck, ≥99.5%), ethylenediamine (Merck, 99%), and distilled water. All chemicals were used without further purification. The instruments used included glassware (Pyrex), an analytical balance (O'hauss), an autoclave (Biobase BKM P-18 (A)), a centrifuge (MPW MED Instrument), a UV lamp (Alonefire), an oven (Memmert), an FTIR spectrometer (Shimadzu IR Prestige 21), a UV-Vis spectrophotometer (Shimadzu), and a photoluminescence (PL) spectrometer (Horiba FL 1000).

2.2. Synthesis of Carbon Dots

Carbon dots (CDs) were synthesized using a hydrothermal method. First, 4 g of citric acid was dissolved in 40 mL of distilled water under magnetic stirring until a homogeneous solution was obtained. The solution was transferred into a Teflon-lined stainless-steel autoclave and heated at 200 °C for 5 h. After naturally cooling to room temperature, the obtained brown solution was centrifuged at 4000 rpm for 30 min to remove large carbon particles. The supernatant was subsequently filtered using Whatman No. 42 filter paper, followed by a 0.22 µm nylon syringe filter to remove unreacted precursors and large aggregates. For the synthesis of nitrogen-doped carbon dots (N-CDs), ethylenediamine (EDA) was added to the precursor solution before hydrothermal treatment with concentrations of 5%, 10%, and 15% (v/v), corresponding to 2 mL, 4 mL, and 6 mL of EDA, respectively. The resulting samples were labeled as N-CDs 5%, N-CDs 10%, and N-CDs 15%. resulted in clear and brown CDs and N-CDs solutions [8]. UV light testing at a wavelength of 365 nm showed that CDs and N-CDs exhibit blue fluorescence. The figure shows that the UV reflection from 10% N-CDs has a brighter blue color compared to CDs. The synthesis steps are shown in Figure 1.

Figure 1. Schematic illustration of N-CDs synthesis

2.3. Characterization

The fluorescence behavior of CDs and N-CDs solutions was initially observed under a 365 nm UV lamp. FTIR analysis was performed using a Shimadzu IR Prestige-21 spectrometer in the wavenumber range of 4500–500 cm^{-1} to identify functional groups. UV–Vis absorption spectra were recorded using a Shimadzu UV-1700 spectrophotometer within the wavelength range of 200–800 nm. Photoluminescence (PL) measurements were carried out using a Horiba FL 1000 spectrometer with an excitation wavelength of 370 nm and emission range of 400–900 nm. The quantum yield (QY) of the samples was determined comparatively using quinine sulfate in 0.54 M H_2SO_4 as the reference standard with a known quantum yield (QYref) of 0.54 [16]. The quantum yield was calculated using Equation (1):

$$QY = QYref\left(\frac{I}{Iref}\right)\left(\frac{Aref}{A}\right)\left(\frac{n}{nref}\right)^2 \qquad (1)$$

where QY is the quantum yield of the sample, QYref is the quantum yield of the reference, I and I_{ref} are the integrated fluorescence intensities of the sample and reference, A and A_{ref} are the absorbance values at the excitation wavelength, and η and η_{ref} are the refractive indices of the sample and reference solutions, respectively.

3. Results and Discussion

3.1. Fourier Transform Infrared Spectroscopy (FTIR) Analysis

The FTIR spectra of CDs and N-CDs synthesized using different EDA concentrations are shown in Figure 2, while the corresponding functional groups are summarized in Table 1. All samples exhibit a broad absorption band in the region of 3500-3200 cm^{-1}, which is attributed to overlapping O–H and N–H stretching vibrations. The broadness of this peak indicates the presence of hydroxyl and amine functional groups on the carbon dot surface, contributing to the hydrophilic nature and dispersion stability of the materials.

A strong absorption peak observed around 1653-1635 cm^{-1} is associated with C=O stretching vibrations of carbonyl or amide groups. The oxygen-containing functional groups and conjugated carbon structures are commonly reported in citric acid-derived CDs [14]. The absorption peaks in the range of 1222-1016 cm^{-1} correspond to C–O stretching vibrations originating from alcohol, ether, or carboxyl groups on the carbon dot surface. These oxygen-containing groups are important because they contribute to surface reactivity and influence the optical properties of CDs.

After EDA addition, new peaks appeared around 1330-1328 cm^{-1}, corresponding to C–N stretching vibrations, confirming successful nitrogen incorporation into the carbon structure. Compared with undoped CDs, the intensity of nitrogen-related peaks became more pronounced in N-CDs samples, particularly at 10% and 15% EDA concentrations, indicating increased nitrogen functionalization. Similar FTIR features have been reported in previous studies on citric acid–EDA-derived N-CDs, where nitrogen doping produced amide and amine groups that improved fluorescence performance through surface passivation [17].

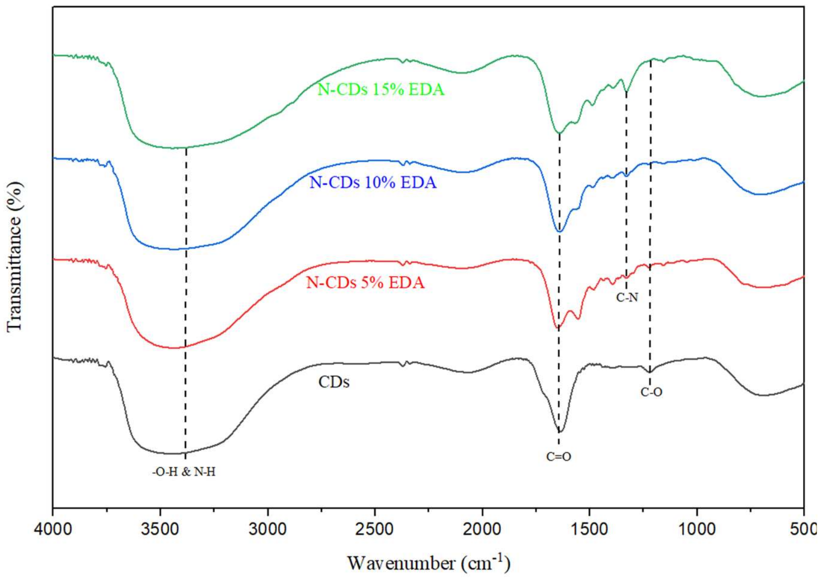

Figure 2. FTIR spectra of CDs, N-CDs 5% EDA, N-CDs 10% EDA, and N-CDs 15% EDA

Table 1. FTIR spectra of CDs, N-CDs 5% EDA, N-CDs 10% EDA, and N-CDs 15% EDA

Functional Group	Wavenumber (cm^{-1})			
	CDs	N-CDs 5% EDA	N-CDs 10% EDA	N-CDs 15% EDA
O-H/N-H	3444	3444	3446	3442
C=O	1635	1653	1639	1643
C-O	1222	1047	1016	1157
C-N	-	1330	1330	1328

The FTIR results demonstrate that nitrogen doping successfully modified the surface chemistry of the CDs through the formation of nitrogen-containing functional groups while maintaining oxygen-containing groups that are beneficial for optical activity.

3.2. UV-Vis Analysis

UV-Vis spectroscopy was performed to identify a material's ability to absorb light at various wavelengths. Figure 3 shows the UV–Vis absorption spectra of CDs and N-CDs synthesized using different EDA concentrations. The undoped CDs exhibit two characteristic absorption peaks centered around 289 nm and approximately 390 nm. The absorption band near 289 nm is attributed to π→π* electronic transitions, whereas the absorption around 390 nm originates from n→π* transitions of carbonyl-related surface states.

After nitrogen doping, slight changes in the absorption peak position and intensity were observed. The N-CDs 5% sample exhibited absorption peaks at approximately 294 nm and 391 nm, indicating that nitrogen incorporation slightly modified the surface electronic states. For N-CDs 10%, the absorption peak shifted to around 392 nm with higher absorption intensity, suggesting improved surface passivation and stronger interaction between nitrogen-containing groups and the carbon core [18].

Interestingly, the N-CDs 15% sample showed a shift toward a lower wavelength (approximately 385 nm), indicating a slight blue shift rather than a continuous red shift. This behavior suggests that excessive EDA addition may alter the surface state distribution and increase structural disorder or defect density, which can influence the optical transition behavior. Therefore, the optical enhancement does not increase linearly with EDA concentration.

Nitrogen doping is known to modify the surface electronic environment of CDs by introducing nitrogen-containing functional groups such as amide and amine groups, which influence electron transition pathways and fluorescence behavior [14]. Similar UV–Vis characteristics for citric acid–EDA-derived N-CDs have also been reported in previous studies, where nitrogen incorporation enhanced absorption intensity and improved photoluminescence properties.

Overall, the UV–Vis results confirm that EDA concentration significantly affects the optical absorption characteristics of CDs through modification of surface states and electronic interactions rather than drastic structural changes in the carbon core.

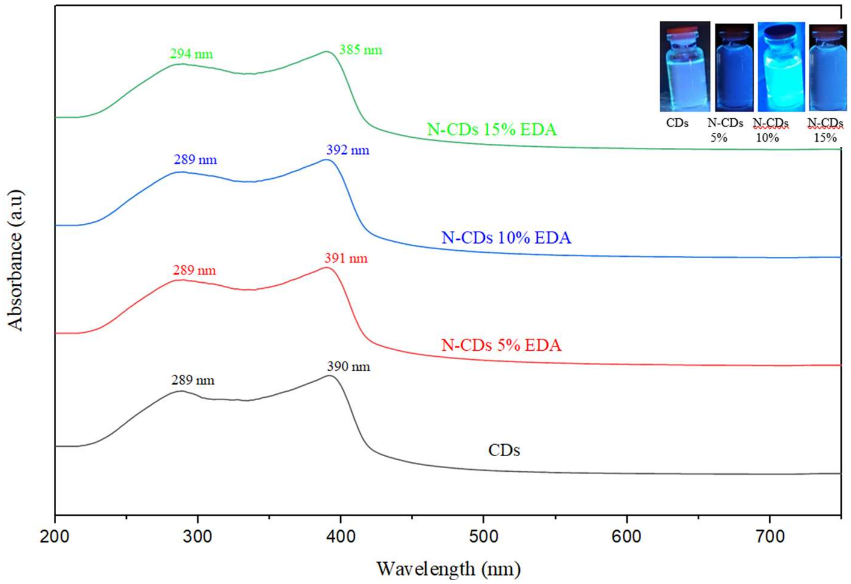

Figure 3. UV-Vis spectra of CDs, N-CDs 5% EDA, N-CDs 10% EDA, and N-CDs 15% EDA

3.3 Photoluminescence (PL) Analysis

The photoluminescence spectra of CDs and N-CDs are presented in Figure 4. The undoped CDs exhibit a blue emission peak centered at approximately 445 nm. After nitrogen doping using EDA, the emission peak slightly shifted to 446–447 nm, accompanied by a significant increase in fluorescence intensity.

The enhancement of fluorescence intensity in N-CDs is associated with the incorporation of nitrogen-containing functional groups that improve surface passivation and suppress non-radiative recombination pathways. Nitrogen atoms can introduce additional surface states that facilitate radiative electron–hole recombination, thereby improving fluorescence efficiency [19]. The slight emission shift observed after nitrogen doping indicates modification of surface electronic states without significantly changing the carbon core structure.

Among all samples, N-CDs 10% exhibited the highest fluorescence intensity and quantum yield. This result suggests that 10% EDA provides an optimum balance between nitrogen incorporation and surface defect passivation. At this concentration, nitrogen doping effectively improves surface electronic interactions while minimizing non-radiative defects.

In contrast, although N-CDs 15% still showed relatively high fluorescence intensity, their quantum yield slightly decreased compared with N-CDs 10%. This phenomenon may be attributed to excessive nitrogen precursor addition that introduces additional surface defects or aggregation effects, which promote non-radiative recombination and fluorescence quenching. Therefore, increasing EDA concentration beyond the optimum level does not necessarily improve fluorescence performance.

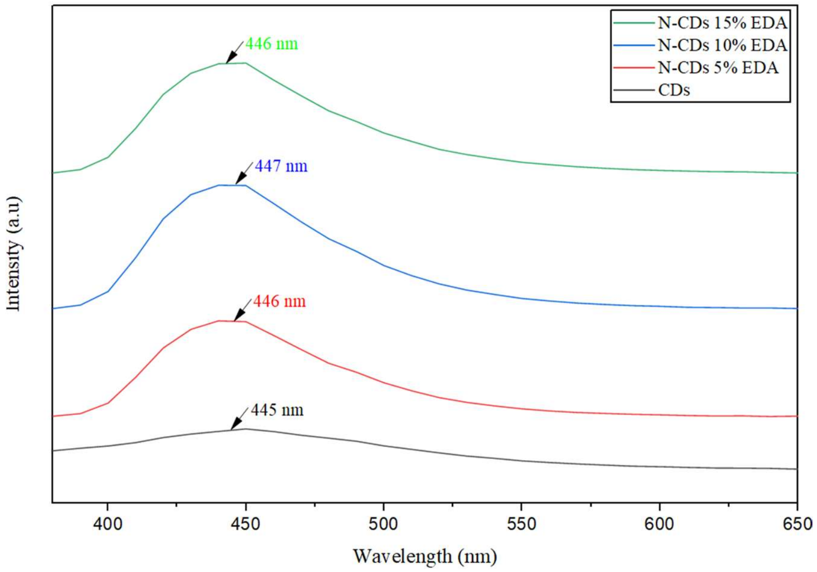

Figure 4. PL spectra of CDs, N-CDs 5% EDA, N-CDs 10% EDA and N-CDs 15% EDA

Table 2. QY Values of CDs, N-CDs 5% EDA, N-CDs 10% EDA and N-CDs 15% EDA

No	Materials	λexcitation	λemission	QY (%)
1	CDs	370 nm	445 nm	30.28%
2	NCDs 5% EDA	370 nm	446 nm	38.01%
3	NCDs 10% EDA	370 nm	447 nm	43.98%
4	NCDs 15% EDA	370 nm	446 nm	43.07%

Table 2 summarizes the quantum yield values of all samples. The undoped CDs exhibited a quantum yield of 30.28%, while N-CDs 5%, N-CDs 10%, and N-CDs 15% showed values of 38.01%, 43.98%, and 43.07%, respectively. The substantial increase in quantum yield after nitrogen doping confirms the important role of EDA in improving fluorescence efficiency.

Compared with previous studies, the QY values obtained in this work are comparable to those reported for citric acid–EDA-derived N-CDs synthesized using hydrothermal methods, which generally range between 30–50% depending on synthesis conditions and precursor composition [16]. This indicates that the present synthesis strategy successfully produced highly luminescent N-CDs with competitive optical performance.

Table 3. Comparison of Quantum Yield (QY) Values of nitrogen-doped carbon dots (N-CDs)

No.	Precursor	Doping Element	Synthesis Method	QY (%)	Reference
1	Citric acid + Urea	Nitrogen (N)	Hydrothermal microwave-assisted	19%	[20]
2	Biomassa tempurung kemiri + EDA	Nitrogen (N)	Hydrothermal	27%	[21]
3	Citric acid + L-phenylalanine	Nitrogen (N)	Hydrothermal	36.5%	[22]
4	Citric acid + EDA	Nitrogen (N)	Hydrothermal	43.98	This work

Table 3 presents a comparison of the quantum yield (QY) values of nitrogen-doped carbon dots (N-CDs) reported in several previous studies with the results obtained in this work. The comparison demonstrates that the QY of the N-CDs synthesized in this study (43.98%) is relatively higher than those reported in other hydrothermal synthesis systems using different nitrogen precursors. N-CDs synthesized from citric acid and urea showed a QY of 19%, while N-CDs derived from candlenut shell biomass and EDA exhibited a QY of 27%. In another study, citric acid combined with L-phenylalanine produced N-CDs with a QY of 36.5%. The higher QY obtained in this work indicates that the use of EDA as a nitrogen source effectively improved fluorescence efficiency through enhanced surface passivation and modification of surface electronic states. In addition, the optimized EDA concentration (10%) provided a balance between nitrogen incorporation and suppression of non-radiative recombination, resulting in stronger photoluminescence performance. These findings confirm that precursor selection and nitrogen doping concentration play important roles in determining the optical properties and fluorescence efficiency of carbon dots.

Conclusion

Carbon dots (CDs) and nitrogen-doped carbon dots (N-CDs) were successfully synthesized from citric acid using a hydrothermal method with different ethylenediamine (EDA) concentrations. FTIR analysis confirmed the formation of oxygen-containing and nitrogen-containing functional groups, indicating successful nitrogen incorporation into the carbon structure. UV–Vis and photoluminescence analyses demonstrated that nitrogen doping significantly influenced the optical properties of the materials by modifying the surface electronic states and improving fluorescence efficiency. Among all samples, N-CDs synthesized with 10% EDA exhibited the best optical performance, showing the highest fluorescence intensity and quantum yield. This result indicates that moderate nitrogen doping provides optimum surface passivation and minimizes non-radiative recombination. In contrast, excessive EDA concentration slightly reduced fluorescence performance due to possible defect formation and fluorescence quenching effects. Overall, this study demonstrates that controlled nitrogen doping is an effective strategy for tuning the optical characteristics of citric acid-derived carbon dots. The synthesized N-CDs show promising potential for future applications in sensing, bioimaging, and photocatalysis.

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