

Article

Synthesis of Cellulose-Based Fluorescent Carbon Dots for the Detection of Fe(III) in Aqueous Solutions

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Abstract: The need for eco-friendly, cost-effective, and scalable methods to synthesize carbon quantum dots (CQDs) remains a critical goal in nanotechnology. In this work, nitrogen-doped carbon quantum dots (N-CQDs) were successfully synthesized using cellulose nanocrystals (CNCs) derived from microcrystalline cellulose (MCC) and urea through a rapid one-step microwave-assisted method. The use of renewable cellulose as a precursor aligns with sustainable practices, offering a pathway to transform agricultural waste into valuable nanomaterials. Characterized by TEM, XRD, Raman, XPS, and PL spectroscopy, the N-CQDs demonstrated outstanding optical properties, including strong excitation-dependent fluorescence with an emission maximum at 420 nm. The N-CQDs exhibited exceptional selectivity and sensitivity toward Fe³⁺, achieving a detection limit of 75 nM. Additionally, the pH-dependent fluorescence and stability in diverse conditions highlight the N-CQDs' versatility in environmental monitoring. This study establishes a foundation for using agricultural waste to produce high-performance nanostructures for sensing applications, advancing green nanotechnology and environmental solutions.

Keywords: cellulose; carbon quantum dots; chemosensor; Fe³⁺; fluorescence quenching



Academic Editors: David Cabaleiro and Jacek Fal

Received: 2 December 2024

Revised: 8 January 2025

Accepted: 13 January 2025

Published: 17 January 2025

Citation: Magagula, L.P.; Masemola, C.M.; Motaung, T.E.; Moloto, N.; Langaniso-Dziike, E.C. Synthesis of Cellulose-Based Fluorescent Carbon Dots for the Detection of Fe(III) in Aqueous Solutions. *Processes* **2025**, *13*, 257. <https://doi.org/10.3390/pr13010257>

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1. Introduction

With the increase in industrial and wastewater effluents, water pollution has become a serious threat to human and aquatic life [1]. One of the major concerns is the presence of heavy metal ions from industrial waste such as Fe³⁺, Hg²⁺, Pb²⁺, Cu²⁺, Cr⁶⁺, Zn²⁺, Co²⁺, etc. [2–7]. Ferric ion (Fe³⁺) is the most abundant and essential metal ion in living organisms; it plays crucial roles such as cellular metabolism, oxygen transport, enzyme catalysis, deoxyribonucleic acid (DNA), and ribonucleic acid (RNA) synthesis [8–11]. Although Fe³⁺ plays vital roles in the biological system, excess Fe³⁺ can cause liver damage, kidney failure, cell oxidation, and the annihilation of blood circulation in human body [12,13]. Moreover, high concentrations of Fe³⁺ in water bodies play a role in the primary production of phytoplankton, besides nitrate, phosphate, and silicate which negatively affect the environment and aquatic life [13]. Hence, it is a necessity to develop effective analytical methods for the detection of Fe³⁺ ions and other metal ions.

So far, several approaches for the determination of Fe³⁺ in aqueous solutions have been developed, including plasma optical emission spectroscopy, atomic absorption spectrometry, inductively coupled plasma mass spectrometry, etc. [9,14,15]. However, these methods are complicated, expensive, and require several steps for sample preparation [16].

To overcome these shortcomings, fluorescence quenching has been used for the detection of Fe^{3+} owing to the high sensitivity, great simplicity, easy monitoring, and rapid response [10]. The most widely used fluorescence sensors include organic dyes, semiconductor quantum dots (QDs), fluorescent metal–organic frameworks, and fluorescence metal nanoclusters [1,16–19]. However, there are concerns rising from their photo instability, toxicity, low sensitivity, and environmental unfriendliness, which restrict their real applications [20,21]. Thus, highly efficient, sensitive, photo-stable, and eco-friendly nanostructures are desirable.

Since their discovery in 2004 by Xu et al. during the purification of single-walled carbon nanotube (SWCNT) through preparative electrophoresis, and then via the laser ablation of graphite powder and cement in 2006 by Sun et al., CQDs have gradually become a rising star in the carbon nanomaterials family [22], owing to their unique tunable photoluminescence, size-dependent optical properties, good biocompatibility, facile synthesis, and low toxicity as compared to their counterparts, the semiconductor quantum dots (QDs) [23,24]. Recently, CQDs have been widely used as fluorescence sensors in the detection of Fe^{3+} due to their fluorescence quenching effect, which may originate from the formation of CQD– Fe^{3+} complexes through the interaction of Fe^{3+} with surface functional groups of the CQDs such as phenolic hydroxyl, carboxyl, and amino groups [14]. Different synthesis methods have been developed for the preparation of CQDs such as microwave-assisted synthesis, hydrothermal methods, solid-phase methods, ultrasonic treatment, electrochemical methods, and so on [10,25]. Among these methods, microwave-assisted synthesis is highly desirable because of the simplicity and short synthesis time, which result in homogeneous and fast heating that is beneficial in the synthesis of CQDs [26,27]. Recently, there has been increasing interest in using agricultural waste, such as fruit peels, husks, stalks, and leaves, as precursors for CQDs synthesis. This approach not only provides a sustainable source of carbon for the production of CQDs but also addresses the environmental challenge of waste disposal. By utilizing these abundant agricultural by-products, the synthesis of CQDs becomes more eco-friendly and cost-effective, contributing to the development of greener nanomaterials for various applications, such as Fe^{3+} detection through fluorescence quenching [28].

In this paper, we report a simple, one-step microwave synthesis to prepare nitrogen-doped carbon quantum dots (N-CQDs) from microcrystalline cellulose-derived cellulose nanocrystals and urea. The as-prepared N-CQDs exhibit an excitation-dependent emission behavior with an optimum fluorescence excitation at 340 nm and an emission maximum of 420 nm. These N-CQDs were explored for the detection of Fe^{3+} in solution.

2. Materials and Methods

2.1. Materials and Reagents

Microcrystalline cellulose (MCC), sulfuric acid (H_2SO_4 , 98%), and urea ($\text{CH}_4\text{N}_2\text{O}$) were obtained from Sigma-Aldrich (Johannesburg, South Africa). Metal salts, namely KCl, $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\text{CdCl}_2 \cdot \text{H}_2\text{O}$, $\text{Cu}(\text{NO}_3)_2$, $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, $\text{Ca}(\text{NO}_3)_2$, $\text{Fe}(\text{NO}_3)_2$, $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, and NaNO_2 , were all purchased from Sigma-Aldrich (Johannesburg, South Africa). All the chemicals and reagents were of analytical grade and were used as received without further purification. Deionized water was used for the preparation of aqueous solutions.

2.2. Preparation of Cellulose Nanocrystal (CNCs) from Microcrystalline Cellulose

Microcrystalline cellulose (MCC) was used to prepare cellulose nanocrystals by acid hydrolysis according to previously documented methods [29], with some minor modifications. Briefly, 5 g of MCC was hydrolyzed with 50% of H_2SO_4 (v/v) solution at 45 °C

for 30 min under continuous stirring. This reaction was then quenched by the addition of 10-fold cold distilled water to the reaction mixture, followed by centrifugation at 5000 rpm for 20 min. The suspension was dialyzed against distilled water for several days until a constant pH in the range of 6–7 was obtained, resulting in a colloidal suspension that was sonicated in an ice bath for 1 h to homogenize the generated cellulose nanocrystals before storage in a refrigerator for further use.

2.3. Synthesis of Nitrogen-Doped Carbon Quantum Dots (N-CQDs) from Cellulose Nanocrystals (CNCs)

Figure 1 illustrates the schematic diagram of the synthesis of N-CQDs from CNCs. Briefly, 0.5 g dry powdered CNCs was mixed with 0.35 g of urea and placed in a 35 mL microwave tube with 15 mL of distilled water. To perform the synthesis, the sample was heated from room temperature to 180 °C and maintained at this temperature for 10 min before fast cooling to room temperature. The reaction mixture was centrifuged to remove large particles and impurities and then passed over a 0.22 µm filter membrane to isolate the N-CQDs, which were stored in the refrigerator for further analysis, including characterization and application.

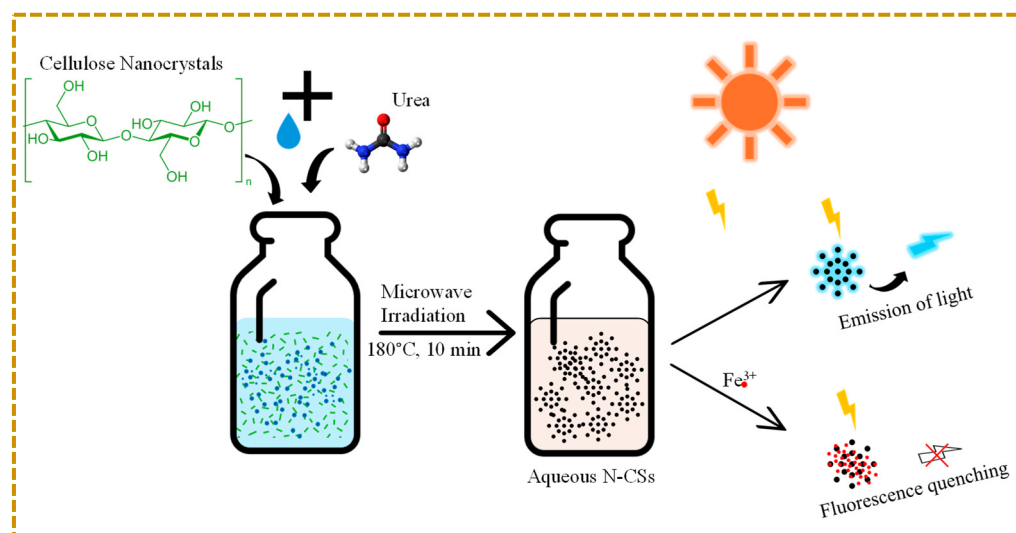


Figure 1. Synthesis of N-CQDs using microwave synthesis and their application in the detection of Fe³⁺ using photoluminescence spectroscopy.

2.4. Detection of Fe³⁺ Using N-CQDs

For the detection of Fe³⁺, N-CQDs (100 µL of the original fluid was diluted with 2 mL of distilled water) were mixed with varying concentrations of ferric nitrate (0–3000 µM) at a 1:1 *v/v* ratio. To study the selectivity of the prepared N-CQDs towards the detection of Fe³⁺ in aqueous solutions, other metal ions with a concentration of 1000 µM were prepared (K⁺, Cd²⁺, Mg²⁺, Zn²⁺, Ni²⁺, Al³⁺, Co²⁺, Na⁺, and Cu²⁺), and examined similarly to Fe³⁺. The fluorescence emission spectra of the above solutions were collected at the optimum excitation wavelength of 340 nm. The pH effect was determined by gradually adding 0.1 M HCl or 0.1 M NaOH to the N-CQDs solution.

2.5. Characterization

The functional groups of N-CQDs were analyzed using a Fourier transform infrared spectroscopy (FT-IR) (Bruker TENSOR 27 FT-IR, Johannesburg, South Africa). The crystalline phases of the prepared materials were studied using powder X-ray diffraction (PXRD) (Bruker D2 phaser equipped with Cu-Kα radiation ($\lambda = 1.5405 \text{ \AA}$), Johannesburg,

South Africa) at an operating voltage of 30 kV and a current of 10 mA. Transmission electron microscopy was used to determine the morphology of the prepared N-CQDs (TEM, FEI Tecnai G2 Spirit, Johannesburg, South Africa) operated at 120 kV. ImageJ software version 1.8.0 was used to determine the particle size distribution from TEM images; more than 150 particles were measured. The fluorescence characteristics of the N-CQDs were analyzed using a photoluminescence (PL) spectrophotometer (Varian Cary 2656 Eclipse EL04103870 fluorescence spectrophotometer, Johannesburg, South Africa). The UV-vis absorption spectra of the N-CQDs were recorded using a spectrophotometer (SPECORD 210 plus UV-vis spectrophotometer, Johannesburg, South Africa). X-ray photoelectron spectroscopy (XPS) data provide surface elemental composition and chemical states of the N-CQDs; these were acquired in an ultra-high vacuum (UHV) chamber equipped with a SPECS PHOIBOS 150 hemispherical electron energy analyzer (Al K_{α} excitation line; $h\nu = 1486.71$ eV) (Pretoria, South Africa). Raman spectroscopy was employed to determine the area and peak positions of the D and G bands of N-CQDs using a Jobin Yvon T6400 micro-Raman spectrometer (Johannesburg, South Africa), which is equipped with a 514.9 nm argon ion laser and a liquid nitrogen-cooled charge-coupled detector (CCD). During the analysis, significant noise was observed in the signal output due to interference from the fluorescent nature of the functional groups present in the N-CQDs. To mitigate this issue, the laser power was increased to 50% for a few seconds, which helped to suppress the fluorescence and leave behind the carbon skeleton of the N-CQDs for clearer analysis.

3. Results

3.1. Properties of the Prepared N-CQDs

Transmission electron microscope (TEM) images (Figure 2a,b) demonstrate that the prepared N-CQDs exhibited spherical particles with diameters ranging from 1 to 13 nm and an average size of 5 ± 2 nm (the diameters of the particles were determined from the TEM images using ImageJ software as shown in Figure 2c). FT-IR spectroscopy was employed to determine the functional groups (Figure 3a). The characteristic absorption peaks of the CNCs at $3046\text{--}3675\text{ cm}^{-1}$ and $2805\text{--}2990\text{ cm}^{-1}$ can be attributed to the O-H and C-H stretching vibrations [30]; these peaks slightly shifted on the N-CQDs appearing around $2735\text{--}3522\text{ cm}^{-1}$, with an additional adsorption band around $3272\text{--}3389\text{ cm}^{-1}$, which is due to N-H stretching vibration [31]. Comparatively, the spectrum of N-CQDs consists of special bands between 1324 cm^{-1} and 1610 cm^{-1} , which correspond to the characteristic stretching vibration of C-N bonds and N-H bonds, respectively, suggesting that nitrogen was successfully incorporated into the carbon matrix during the synthesis. The XRD pattern of the N-CQDs is shown in Figure 3b. These peaks are attributed to the (002) and (100) planes of graphitic carbon, respectively. The broad peak at 23° indicates the presence of highly disordered carbon atoms, while the weak shoulder at 42° reflects the underlying graphitic structure. These results suggest that the N-CQDs possess a predominantly amorphous nature with some degree of a graphitic character [32]. The inset in Figure 3b shows the XRD pattern of the cellulose nanocrystals (CNCs); three peaks appeared at 16° , 22° , and 34° which were assigned to the amorphous and crystalline carbon framework. This indicates that the CNCs were thoroughly carbonized. The optical properties of the as-prepared N-CQDs were studied using both UV-vis and photoluminescence spectroscopy.

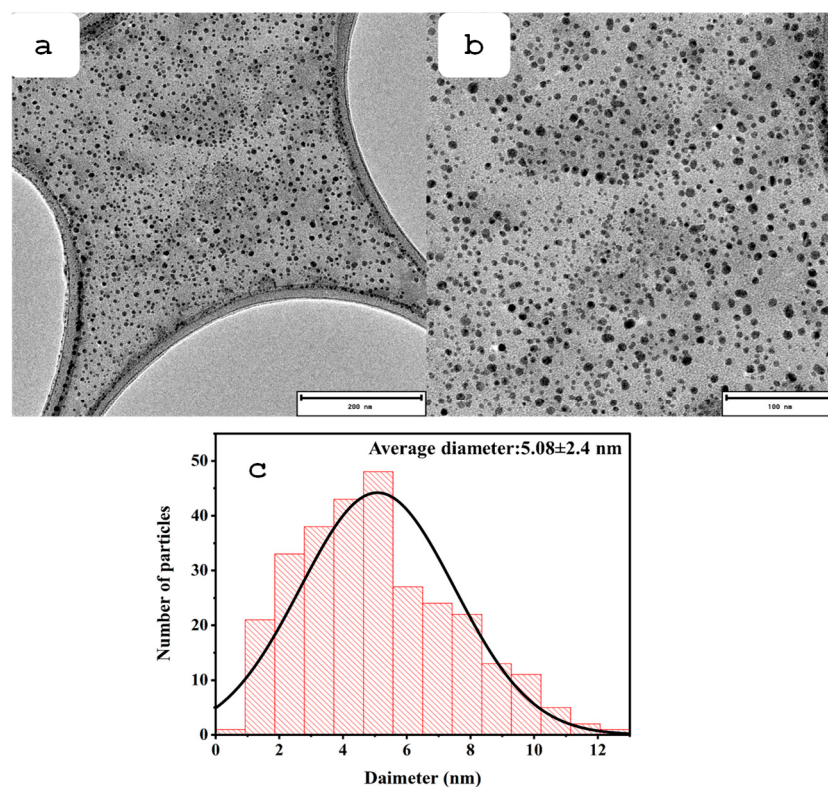


Figure 2. TEM images of the N-CQDs (a,b) and particle size distribution of the N-CQDs (c).

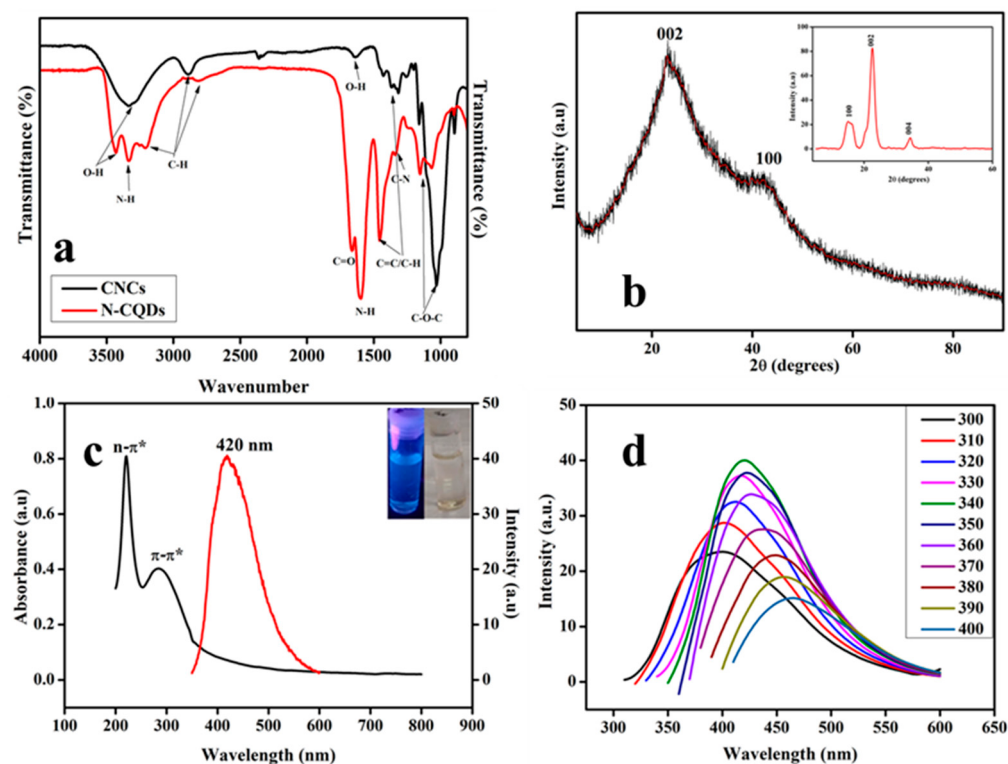


Figure 3. (a) FT-IR of CNCs and the N-CQDs. (b) XRD of the N-CQDs, the inset in (b) shows the XRD of the cellulose nanocrystals. (c) UV-vis absorption spectra of the as-prepared N-CQDs sample in water (black) and fluorescence emission of the N-CQDs at 340 nm excitation wavelength (red); the inset in (c) shows the N-CQDs sample solution in water during daylight and under a UV lamp (365 nm). (d) Fluorescence spectra of N-CQDs obtained from different wavelengths of excitation 300–400 nm (with 10 nm increments starting from 300 nm).

Figure 3c shows the UV-Vis of the N-CQDs; two characteristic peaks at 220 and 285 nm were observed. The peak at 220 nm is attributed to the π - π^* electronic transition of the π -conjugated sp^2 carbon core, while the peak at 285 nm corresponds to the n - π^* electronic transition, arising from the non-bonding orbitals of functional groups attached to the sp^2 hybridized carbon core [33]. Under the UV lamp (365 nm), the N-CQDs exhibited a blue fluorescence (shown in the inset of Figure 3c). Figure 3d shows the excitation-dependent fluorescence emission spectra of N-CQDs obtained at varying excitation wavelengths from 300 nm to 400 nm with an increment of 10 nm, which resulted in an emission redshift [30]. This property may be due to the distribution of different functional groups with different emission traps, the difference in particle size, and multi fluorescence components in N-CQDs [25]. Emission maximum was obtained at 340 nm for the prepared N-CQDs.

The Raman spectrum of the N-CQDs exhibits characteristic carbon peaks centered at 1335 cm^{-1} and 1549 cm^{-1} , corresponding to the D-band and G-band, respectively, as shown in Figure 4. The D-band is associated with sp^3 carbons, arising from structural defects such as dangling bonds and disordered graphite, while the G-band corresponds to sp^2 -hybridized graphitic carbons, representing the vibrations within the graphitic lattice of carbon atoms in a two-dimensional (2D) hexagonal lattice [34]. The area ratio of the D-band to the G-band (A_D/A_G) for the N-CQDs was determined to be 2.9. This high ratio indicates a highly disordered structure, with significant functionalization and incorporation of heteroatoms, which increases the proportion of sp^3 carbons [35]. The Raman analysis highlights that the N-CQDs are highly amorphous, consistent with the XRD results. The D-band reflects the vibrations of carbon atoms with dangling bonds in disordered regions, while the G-band represents the vibrations of sp^2 -bonded carbon atoms. X-ray photoelectron spectroscopy (XPS) was used to investigate the surface chemistry, bonding interactions, and purity of the prepared N-CQDs. The XPS survey spectrum (Figure 5a) identified the presence of carbon (C), oxygen (O), nitrogen (N), and silicon (Si), which are the primary constituents of the material. The high-resolution C 1s spectrum (Figure 5b) reveals four different chemical environments, such as sp^2/sp^3 carbon (C-C, C=C and C-H), C-N/C-OH, C=O/C-O-C, and N-C=O/O-C=O at 284.2, 284.8, 286.0, 287.6, and 288.7 eV, respectively [36]. These results confirm the presence of graphitic carbon and functionalized carbon within the N-CQDs, with sp^3 carbons arising from structural defects such as functional groups and sp^2 -hybridized graphitic carbon contributing to the in-plane structure. This is consistent with the XRD pattern, which shows a broad amorphous peak, indicating a highly disordered carbon phase. The findings align with the FT-IR analysis, which identified various functional groups, and the Raman spectroscopy results, which verified the presence of graphitic structures and structural defects. Similarly, the O 1s spectrum (Figure 5c) deconvolutes into two prominent peaks at 531.6 eV and 533.17 eV, which correspond to C=O and C-O bonds, respectively [37]. The high-resolution N 1s spectrum (Figure 5d) demonstrates peaks at 327.2 eV and 400.2 eV, attributed to organic nitrogen species and cyanide groups, respectively. These nitrogen species, particularly pyridinic and graphitic N, are known to enhance electronic properties and catalytic activity, making the N-CQDs suitable for applications in sensing, energy conversion, and biomedicine [38]. Overall, the XPS results are consistent with the FT-IR analysis, indicating the presence of nitrogen and oxygen on the prepared N-CQDs.

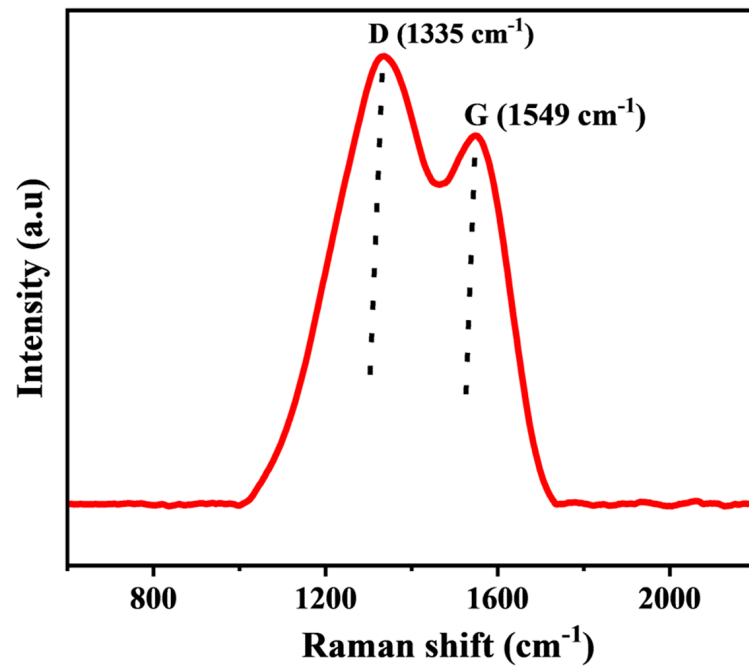


Figure 4. Raman spectrum of the as-synthesized N-CQDs.

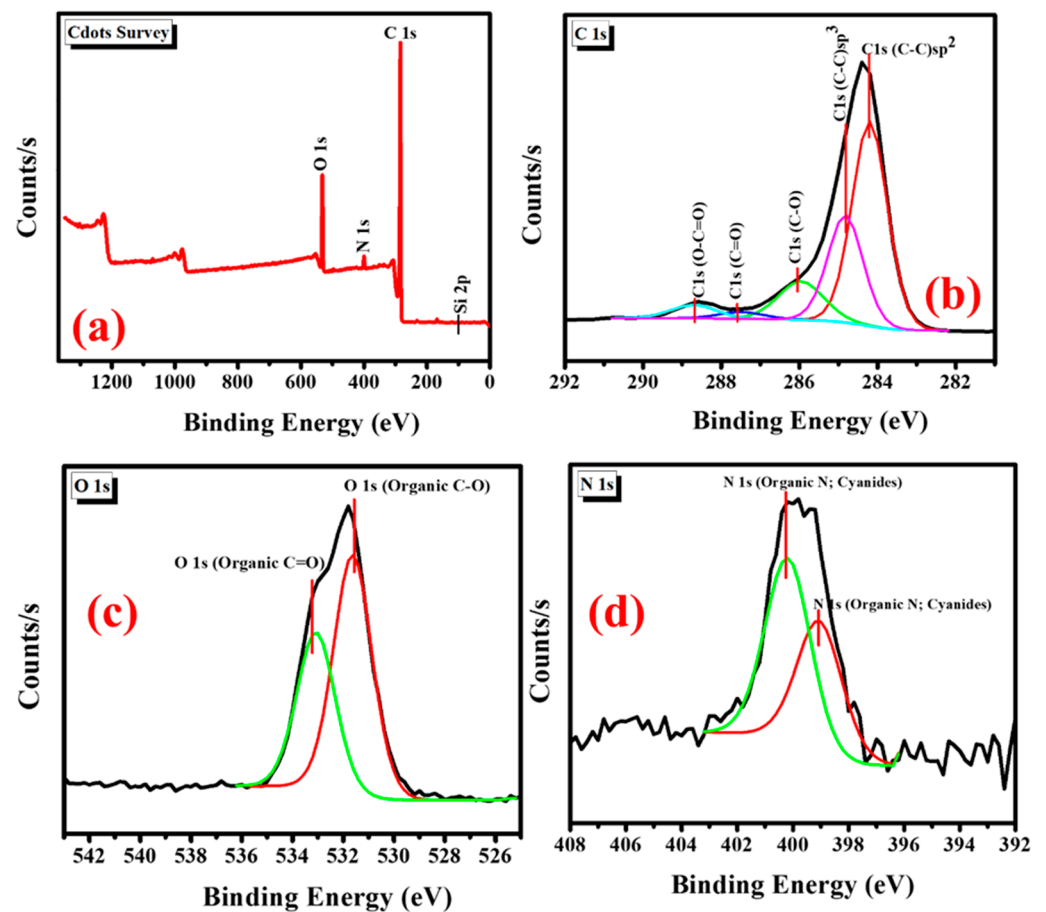


Figure 5. (a) XPS survey spectrum of N-CQDs. (b–d) High resolution spectra of C 1s, O 1s, and N 1s from as synthesized N-CQDs, respectively. The colored peaks in (b–d) show the deconvolution of the C 1s, O 1s, and N 1s peaks, respectively.

3.2. N-CQDs Sensitivity and Selectivity Towards Fe^{3+}

The sensitivity of the N-CQDs towards Fe^{3+} was studied by adding different concentrations of Fe^{3+} (0 μM to 3000 μM) to the N-CQDs suspension and measuring the fluorescence emission intensity at the optimum excitation wavelength of 340 nm. As shown in Figure 6a, the fluorescence intensity of the N-CQDs decreased gradually with the increasing concentration of the Fe^{3+} , indicating that the addition of Fe^{3+} ions can effectively quench the fluorescence emission of the N-CQDs. Based on previous studies, the mechanism of fluorescence quenching of N-CQDs in the presence of Fe^{3+} is caused by the formation of the N-CQDs- Fe^{3+} complexes, which facilitate electron transfers between N-CQDs and Fe^{3+} and which restrict excitation recombination, leading to fluorescence quenching [10,14]. The quenching constant was calculated using the Stern–Volmer equation, $F_0/F - 1 = K_{\text{sv}} [\text{Fe}^{3+}]$, where K_{sv} represents the static Stern–Volmer constant and F_0 and F represent the fluorescence intensities of the N-CQDs in the absence and presence of Fe^{3+} ions, respectively. The Stern–Volmer plots in Figure 6b showed good linearity in the Fe^{3+} range of 0–500 μM , with K_{sv} equal to $0.164 \times 10^4 \text{ M}^{-1}$ and a correlation coefficient (R^2) value of 0.9949. The limit of detection (LOD) was calculated to be 75 nM using the formula $\text{LOD} = 3\sigma/s$ (where σ is the standard deviation and s is the slope of the linear response), which is comparable to previously reported fluorescence detection methods for Fe^{3+} ion detection, as shown in Table 1 [39–41]. UV-Vis was used to further study the mechanism of fluorescence quenching of the N-CQDs in the presence and absence of Fe^{3+} (Figure 6c). After adding 1000 μM Fe^{3+} , the absorption peak at 285 nm disappeared and the spectrum resembled the absorption spectrum of the undiluted Fe^{3+} , indicating Fe^{3+} saturated the surface of N-CQDs, which resulted in a color change from a bright blue to brown color under UV irradiation (the inset of Figure 5c).

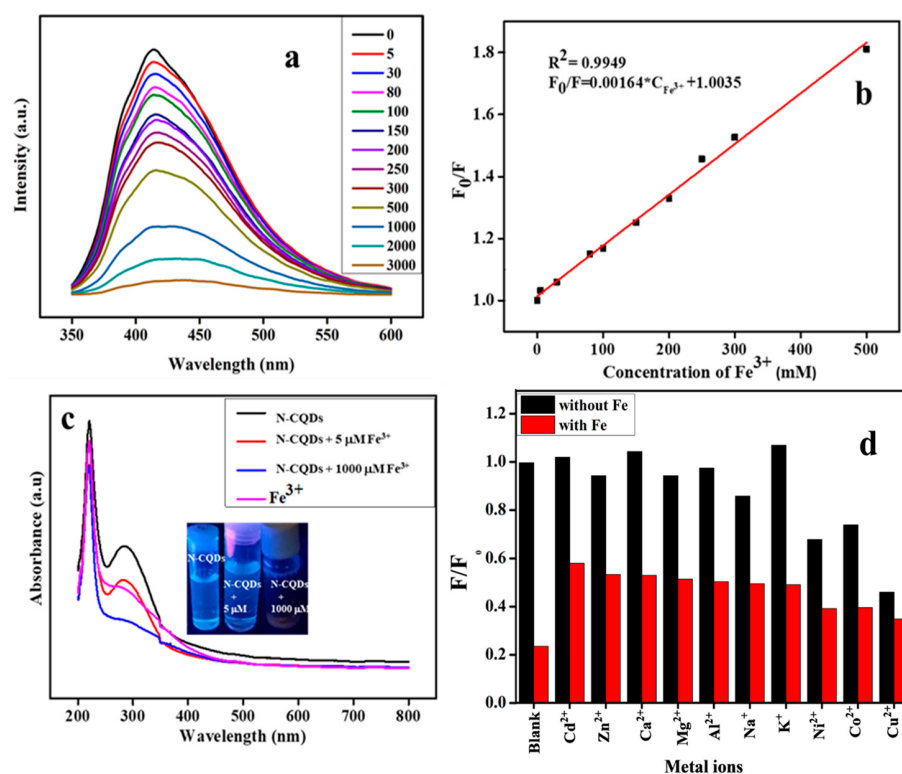


Figure 6. (a) Fluorescence spectra of N-CQDs in different concentrations of Fe^{3+} (5–3000 μM), (b) linear relationship between F_0/F and Fe^{3+} concentration (5–500 μM), (c) UV-Vis spectra of the N-CQDs with and without different concentrations of Fe^{3+} , and (d) changes in the fluorescence intensity ratio (I/I_0) of N-CQDs after the addition of various metal ions. The inset in (c) shows the N-CQDs sample with different concentrations of Fe^{3+} under the UV lamp (365 nm).

Table 1. Comparison of different methods used for the detection of Fe^{3+} .

Detection Method	Sensor Material	Synthesis Method	Average Particle Size (nm)	Linear Range (μM)	LOD (μM)	Reference
Fluorescence	N-CQDs from <i>Morinda coreia</i>	Hydrothermal, 180 °C, 24 h	1.99	0–250	1.32	[39]
Fluorescence	N-CQDs from <i>Crescentia cujete</i> fruit	Hydrothermal, 200 °C, 10 h	4.36	0–250	0.257	[40]
Fluorescence	N-CQDs from <i>Tagetes patula</i> flowers	Hydrothermal, 220 °C, 5 h	5.15	1–4	0.32	[41]
Fluorescence	N-CQDs from cellulose nanocrystals	Microwave, 180 °C, 10 min	5 ± 2	0–500	0.075	This work

To investigate the selectivity of the N-CQDs towards Fe^{3+} , the fluorescence emissions of different metal ions with the potential of interfering with Fe^{3+} (1000 μM of Ca^{2+} , Cd^{2+} , Cu^{2+} , Co^{2+} , Mg^{2+} , Ni^{2+} , Zn^{2+} , Al^{3+} , K^{+} , or Na^{+}) were measured at the optimum excitation wavelength of 340 nm. Figure 6d shows the quenching effect of the different metal ions. It is seen that Fe^{3+} , Cu^{2+} , Ni^{2+} , and Co^{2+} tend to decrease the fluorescent intensity, with Fe^{3+} showing the strongest effect. This is due to the ions having strong binding affinities for the functional groups (e.g., amine, hydroxyl, carboxyl) on the surface of N-CQDs [41]. In the present case, according to the FTIR results, many amino- and hydroxyl-containing functional groups appeared on the surface of the N-CQDs; these functional groups may have chelated Fe^{3+} , resulting in the interaction of lone pairs of electrons on the nitrogen atoms to form complexes via coordination effects. The quenching occurs via an electron transfer between N-CQDs and the metal ion's partially filled or empty 3d orbitals. For Fe^{3+} , the half-filled 3d orbitals allow for efficient electron transfers, disrupting the radiative recombination of excited states and thus leading to fluorescence quenching [14]. Specifically, when Fe^{3+} is added to the N-CQDs, the fluorescence intensity decreases from an initial value (F_0/F) of 1.0 to 0.23 when 1000 μM Fe^{3+} is added, indicating a considerable quenching effect of 77%. Cd^{2+} , Ca^{2+} , and K^{+} slightly increase the fluorescent intensity; this is due to the ions having moderate charge densities and forming weak electrostatic interactions with negatively charged or polar functional groups (e.g., carboxyl and hydroxyl groups) on the N-CQDs [42]. These interactions may stabilize the excited electronic states of the N-CQDs, reducing non-radiative decay pathways and increasing the radiative recombination, which enhances fluorescence [43]. No effect was observed after adding Na^{+} , Mg^{2+} , Zn^{2+} , and Al^{3+} ; this is due to their inability to bind strongly to the functional groups of N-CQDs or the fact that their interaction does not facilitate electron transfer. To further study the interference of Fe^{3+} with other metal ions, Fe^{3+} detection in the presence of other ions was also measured. As shown in Figure 6d after the addition of Fe^{3+} , the other metal ions start to exhibit a slight quenching effect on the fluorescence of the N-CQDs. The obtained results indicate that the prepared N-CQDs have a high sensitivity and selectivity towards the detection of Fe^{3+} and can be used as a chemosensor in an aqueous environment with competing ions.

Figure 7a shows the different metal ions in solution under normal light and UV light. They all show a blue emission, with Fe^{3+} appearing to weakly fluoresce followed by Cu^{2+} when compared to the others. The pH level is one of the critical parameters that affect the sensing performance of the N-CQDs. As shown in Figure 7b, the fluorescence emission intensity decreases slightly between pH 4 and pH 10, while larger variations are observed outside this range. The stability can be attributed to the behavior of the

surface functional groups under different pH conditions, which influence the electronic and chemical interactions on the quantum dot surface [44]. The ability of the metal free N-CQDs to maintain stable fluorescence at slightly acidic to neutral pH conditions suggests their applicability in diverse aqueous environments. For instance, they can be effectively used in monitoring water quality in acid mine drainage, where high levels of Fe^{3+} ions are commonly encountered. Additionally, the pH-dependent fluorescence behavior observed in the N-CQDs suggests their potential as a fluorescence-based pH sensor, providing a dual functionality for detecting both specific ions, like Fe^{3+} , and variations in pH.

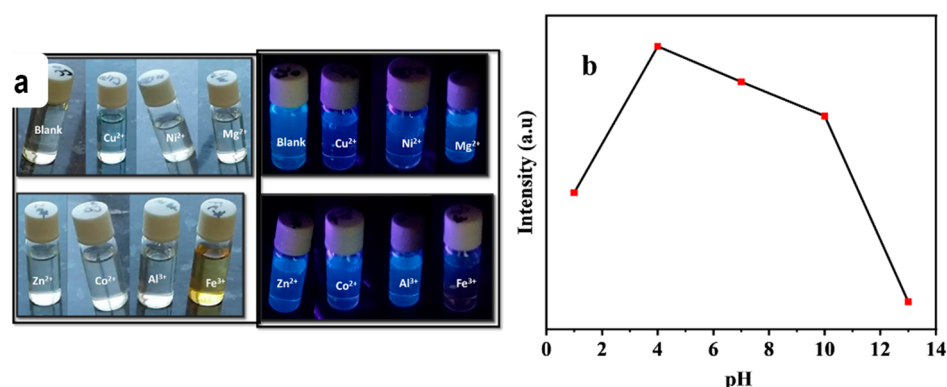


Figure 7. (a) Picture of different metal ions in solution under normal light and UV lamp (365 nm). (b) Effect of pH on the fluorescence emission of N-CQDs.

4. Conclusions

In this study, highly fluorescent N-CQDs with sensitivity towards Fe^{3+} in aqueous solutions and a bright blue emission were synthesized by a one-step microwave synthesis from a mixture of cellulose nanocrystals and urea in 10 min. N-CQDs with an average diameter of 5 ± 2 nm were obtained. The prepared N-CQDs showed excitation-dependent fluorescence, good selectivity toward Fe^{3+} , and a pH-dependent fluorescence. The LOD was determined to be 75 nM, which is comparable to previously reported Fe^{3+} fluorescence detection methods, while exhibiting the highest sensitivity when compared to a majority of reported data. Characterization using TEM, FT-IR, XPS, XRD, and Raman spectroscopy confirmed the spherical morphology, successful nitrogen incorporation, and predominantly amorphous structure of the N-CQDs with some graphitic features. These results demonstrate that N-CQDs possess a combination of structural, optical, and functional properties suitable for environmental monitoring and analytical sensing applications. Despite these promising findings, the reusability of the N-CQDs and their long-term stability in aqueous solutions remain unexplored. Future studies will investigate these aspects to assess their practicality for repeated use and durability in real-world conditions. Further research will also focus on optimizing reaction parameters to enhance sensitivity and selectivity, as well as exploring the impact of heteroatom doping and quantitative analysis comparisons. This work underscores the potential of transforming sustainable agricultural materials into high-performance nanostructures, contributing to green nanotechnology and offering innovative solutions for environmental monitoring and sensing applications.

Author Contributions: Conceptualization, E.C.L.-D.; writing—original draft preparation, L.P.M.; writing—review and editing, E.C.L.-D., L.P.M., C.M.M., T.E.M. and N.M. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded the National Research Foundation of South Africa Africa (Thuthuka grant number 129532) and the University of the Witwatersrand. This work is based on the research supported in part by the WITS Chancellor’s Female Academic Leaders Fellowship.

Data Availability Statement: The original contributions presented in the study are included in the article; further inquiries can be directed to the corresponding author.

Acknowledgments: The authors would like to acknowledge the financial support received from the National Research Foundation of South Africa and the University of the Witwatersrand.

Conflicts of Interest: The authors declare no conflicts of interest.

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