RESEARCH ARTICLE

Nanofabrication and characterization of green-emitting N-doped carbon dots derived from pulp-free lemon juice extract

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Abstract: In this work, highly fluorescent green-emitting N-doped carbon dots (N-CDs) were derived from pulp-free lemon juice extract, as a green precursor, through a one-pot carbonization at 180 oC for 3 to 5 h. The newly fabricated N-CDs were thoroughly characterized using different imaging and analytical techniques, including Scanning electron microscopy (SEM), Fourier-transform infrared spectroscopy (FTIR), Fluorescence spectroscopy, X-ray diffraction analysis (XRD), and Ultraviolet-visible (UV-Vis) spectroscopy. The preliminary evaluation showed that N-CDs synthesized at 180 °C for 3 and 5 hours emit bright green light under UV or blue light irradiation with a quantum yield of 16.33% and 21.80%, respectively. The fluorescence spectroscopic profiles revealed that as-developed N-CDs exhibit excitation-independent photoluminescence (PL) emission at 365 nm. FTIR profile reveals the functional group entities with evident peaks in 3190 cm^{-1}, 1660 cm^{-1}, 1580 cm^{-1}, 1405 cm^{-1}, 1365 cm^{-1}, 1190 cm^{-1}, and 1060 cm^{-1} regions, among others that correspond to the presence of N-H, C-H, C=O and C=N, C=C, C-H, COOH, C-O-C, and C-O. SEM unveils uniform and well-crystalline morphology of N-CDs..

Keywords: carbon dots; fabrication; green precursor; lemon juice; photoluminescence.

List of abbreviations

Abbreviation	Definition
CDs	Carbon dots
QDs	Quantum dots
PL	Photoluminescence
N-CDs	Nitrogen doped carbon dots
FTIR	Fourier-transform infrared
SEM	Scanning electron microscopy
PALS	Phase-analysis light scattering
UV-Vis	ultraviolet-visible
XRD	X-Ray Diffraction
QY	Quantum yield

1. INTRODUCTION

With ever-increasing scientific knowledge and mechanical awareness, carbon-based fluorescent materials, so-called carbon dots (CDs), as a mixture of quantum dots (QDs) and multifunctional molecules, which make this new interface of materials fascinating in recent years. For said reason, CDs offer combined advantages from photo-physics and quantum chemistry (Wang and Lu 2022), making them robust candidates for multipurpose applications. For instance, some key characteristics and highly requisite attributes of CDs include excellent optical features (i.e., photoluminescence - PL), photo-catalytic potential, high aqueous solubility, size effect, photo-induced electron transfer properties, up-conversion photoluminescence, photochemical stability, conductivity, low/no toxicity with high biocompatibility, sensing attributes, robust bio-carriers or delivery vehicles, environmentally friendly and green reaction conditions, etc. (Sun and Lei 2017; Cruz-Cruz *et al.*, 2022; He *et al.*, 2022).

For a sustainable and efficient application, it is equally essential to understand CDs' structure, chemistry, and surface available functional entities. From the structural viewpoint, a single CD's central core comprises sp2 carbon, whereas the surface is tunable with multifunctional groups. Doping with oxygen, nitrogen, and/or other entities, the CD's surface can be functionalized. For instance, the combination of N-doped sp2-based carbon core, s-triazine constructs, and aliphatic functional groups are connected by C-N bonds (Mintz *et al.*, 2021). The central core diversity is also reported as graphitic-like, which is related to the amorphous or crystalline-like appearance with rich amino, carboxyl, and hydroxyl groups on its surface (Ansi *et al.*, 2019; Gallareta-Olivares *et al.*, 2023). An amorphous core of pure sp3 carbon or a ratio of sp3:sp2 carbon has also been reported (Tepliakov *et al.*, 2019).

Furthermore, surface passivation and doping enhance the PL properties and broaden the application scope of CDs (González-González *et al.*, 2022). Doping is a valuable method for controlling the physicochemical characteristics of CDs. It has attracted much research interest in recent years due to its additional benefits compared to pristine CDs. The overlapping atomic orbitals of the heteroatoms and carbon atoms, as well as the push/pull electron action of heteroatoms, lead to modifications in the electronic structure, nanostructure, and chemical composition of heteroatom-doped CDs (González-González *et al.*, 2022). However, the distinction between carbon precursors and the fabrication process recognizes CDs' morphological variation and structural diversity.

So far, various multi-process approaches (e.g., microwave, laser ablation, solvothermal ultrasonication, electrochemical oxidation, hydrothermal, and other synthetic processes) have been proposed and developed to fabricate CDs with robust and requisite features. It is evident from the literature that each of the techniques mentioned above has its own merits and demerits. However, the one-pot fabrication of CDs via the carbonization of green precursors, e.g., lemon juice, that follows the green agenda is of supreme interest, as it exploits facile, low-cost, and large-scale preparation routes (He *et al.*, 2022). Moreover, one-pot synthesis yields highly fluorescent CDs as new materials of interest to replace conventional QDs. In addition, green synthesis approaches also limit the utilization of heavy metals, which can lead to toxicity concerns and environmental problems (Zuo *et al.*, 2016).

By considering the above critiques and green synthesis requisites, herein, we report the one-pot fabrication of N-CDs from lemon juice as a green precursor through a carbonization mechanism. The present work also aimed to develop a cost-effective and energy-saving process by performing carbonization at relatively low-temperatures and minimal reaction periods. Moreover, the exploitation of lemon juice as a green precursor supports the green chemistry agenda, thus keeping the CDs synthesis sustainable compared to the chemical-based synthesis process. N-CDs were thoroughly characterized using different imaging and analytical techniques, including Scanning electron microscopy (SEM), Fourier-transform infrared spectroscopy (FTIR), Fluorescence spectroscopy, X-ray diffraction analysis (XRD), and Ultraviolet-visible (UV-Vis) spectroscopy.

2. MATERIALS AND METHODS

2.1. Chemicals/reagents

Lemon *(citrus aurantifolia)* was purchased from the local market. Urea and sodium fluorescein were obtained from Sigma-Aldrich, USA. Other reagents and chemicals were of analytical grade and used without further purification unless otherwise specified. Deionized water (DI) was used to prepare triplicate samples for fabrication and analytical purposes.

2.2. Preparation of N-CDs

The one-step carbonization treatment was used to prepare all N-CDs as follows. First, fresh lemons were hand squeezed to obtain their crude juices. These juices were centrifuged at 4,000 rpm for 15 min and the obtained supernatant was passed through a filter paper (with a pore diameter of 25 µm) under a vacuum. Afterwards, 1.6 g of urea was dissolved in 15 mL of the as-prepared pulp-free lemon juice. The solution was lightly mixed and then transferred into porcelain capsules to create a uniform solution. The capsules were kept at a constant temperature of 180 °C for 3 and 5 hours in an oven. After cooling at room temperature, the resulting black powder was dispersed in acetone. This solution was centrifuged at 4,000 rpm for 20 min. The N-CDs in the precipitates were purified by washing with acetone and methanol/acetone solution (10/90 v/v). Subsequently, the purified N-CDs were stored at -80 °C for 12 h and freeze-dried for 48 h. Finally, the N-CDs (1 mg mL-1) were redispersed in DI water and stored in a dry and dark place for further usage.

2.3. Characterization

The Fourier-transform infrared (FTIR) spectra of the N-CDs were recorded on a FrontierTM FTIR Spectrometer (PerkinElmer, Waltham, MA, USA) in a transmission range of 400 cm-1 to 4000 cm-1 with 64 scans. The surface morphology was observed by scanning electron microscopy (SEM) EVOMA25 (Carl Zeiss, Oberkochen, BW, GE) with a voltage of 20kV. To analyze the surface charge, the ζ potential values were measured by Phase-analysis light scattering (PALS) with a Nanobrook 90 Plus PALS (Brookhaven instruments, Holtsville, NY, USA). Four measurements per sample were performed, and using the Smoluchowski equation, the electrophoretic mobility was converted into the ζ potential. The ultraviolet-visible (UV-Vis) absorbance was measured using a UV/Vis Lambda 365 instrument (PerkinElmer, Waltham, MA, USA) in a 200-800 nm wavelength range. The fluorescence measurements were carried out with a QE Pro-FL Fluorescence Spectrometer (OceanInsight, Orlando, FL, USA) with an excitation light of 365 nm. X-ray diffraction (XRD) patterns were investigated using a Rigaku Miniflex 600 (Rigaku Corporation, Tokyo, JP) using Cu-Kα as a radiation source and a current and voltage of 15mA and 30kV.

2.4. Quantum yield measurements

Linearizing integrated fluorescence intensities performed the measurement of the quantum yield (QY) at an excitation wavelength of 365 nm of N-CDs and the fluorescence standard versus their corresponding absorbance. QY was then calculated by the following equation (1):

$$QY = QY_r \left(\frac{m}{m_r}\right)\left(\frac{\eta^2}{\eta_r^2}\right) \quad (1)$$

Where, *QY* is the quantum yield of the N-CDs, *m* is the slope of the curve, and *n* is the refractive index of the solvent. The subscript *"r"* refers to the fluorescence standard reference sodium fluorescein in 0.1 M NaOH (QY_r = 0.79). The refractive index of the 0.1 M NaOH solution and deionized water is 1.33. The absorbance was kept below 0.1.

3. RESULTS AND DISCUSSION

The functional groups present on the surface of the N-CDs were determined by FTIR spectroscopy. The surface chemistry of both samples was identical. As shown in Figure 1a, the peaks at 3440-3340, 3190 and 2765 cm-1, can be attributed to stretching vibrations of O−H, N−H (corresponding to the presence of −NH2 groups), and C−H, respectively (Monte-Filho *et al.*, 2019; Wang *et al.*, 2022a). The absorption peak at 1365 cm-1 is usually ascribed to the vibration of the COOH group. This peak may be related to the presence of acids in the synthesis when the temperature ranges between 150 and 200 °C (Hoan *et al.*, 2019). The presence of these hydrophilic groups (−OH, −COOH, and −NH2) on the surface of the N-CDs indicate good dispersibility in water (Khairol Anuar *et al.*, 2021). The absorption peaks at 1660-1580, 1445, and 1405 cm-1 can be attributed to the stretching vibrations of C=C/C=O, N−H and C−N, respectively (Mondal *et al.*, 2016; Monte-Filho *et al.*, 2019; Wang *et al.*, 2022a). In addition, peaks at 1190-1150 and 1060 cm^{-1} correspond to the stretching of the C−O−C, and C−O bonds, respectively (Mondal *et al.*, 2016; Hoan *et al.*, 2019).

As can be seen in Figure 1b, the XRD pattern of both samples of N-CDs is displayed from 0 to 80º. The diffraction pattern shows a sharp peak centered at 27º, corresponding to the typical (002) diffraction of crystalline graphitic carbon. Therefore, the existence of CDs is successfully demonstrated (Tammina *et al.*, 2019; Wang *et al.*, 2022b).

The obtained ζ potential results (Table 1) show negative values due to a dense electron cloud concentrating on the N-CDs. Highly dispersed CDs typically have values above 30 mV or below -30 mV, indicating electrostatic stability. Since the obtained ζ potential results from N-CDs are not high enough to exceed -30 mV, its stability can be considered neutral (Yahaya Pudza *et al.*, 2020).

Sample	ζ potential (mV)		
3 h, 180 °C	-8.53	±	15.64
5 h, 180 °C	-7.04	±	14.10

Table 1. ζ potential from the N-CDs.

UV-Vis absorption and fluorescence emission spectra of N-CDs were measured accordingly to analyse the optical properties. As shown in Figure 1c, the optical absorption spectrum showed two main absorption centers with 310 and 400 nm peaks in both samples. The first absorption peak corresponds to the n−p* transition of the C=O (nonbonding oxygen states) band (He *et al.*, 2018). The second peak indicates the n−p* transition of the C=C (sp^2 domain) groups (Khairol Anuar *et al.*, 2021). In the fluorescence spectra of N-CDs (Figure 1d), the samples synthesized for 3 and 5 hours displayed identical emission peaks at 570 nm when excited at 365 nm. This emission wavelength indicates strong fluorescence from both samples in the light green region (Kasprzyk *et al.*, 2018).

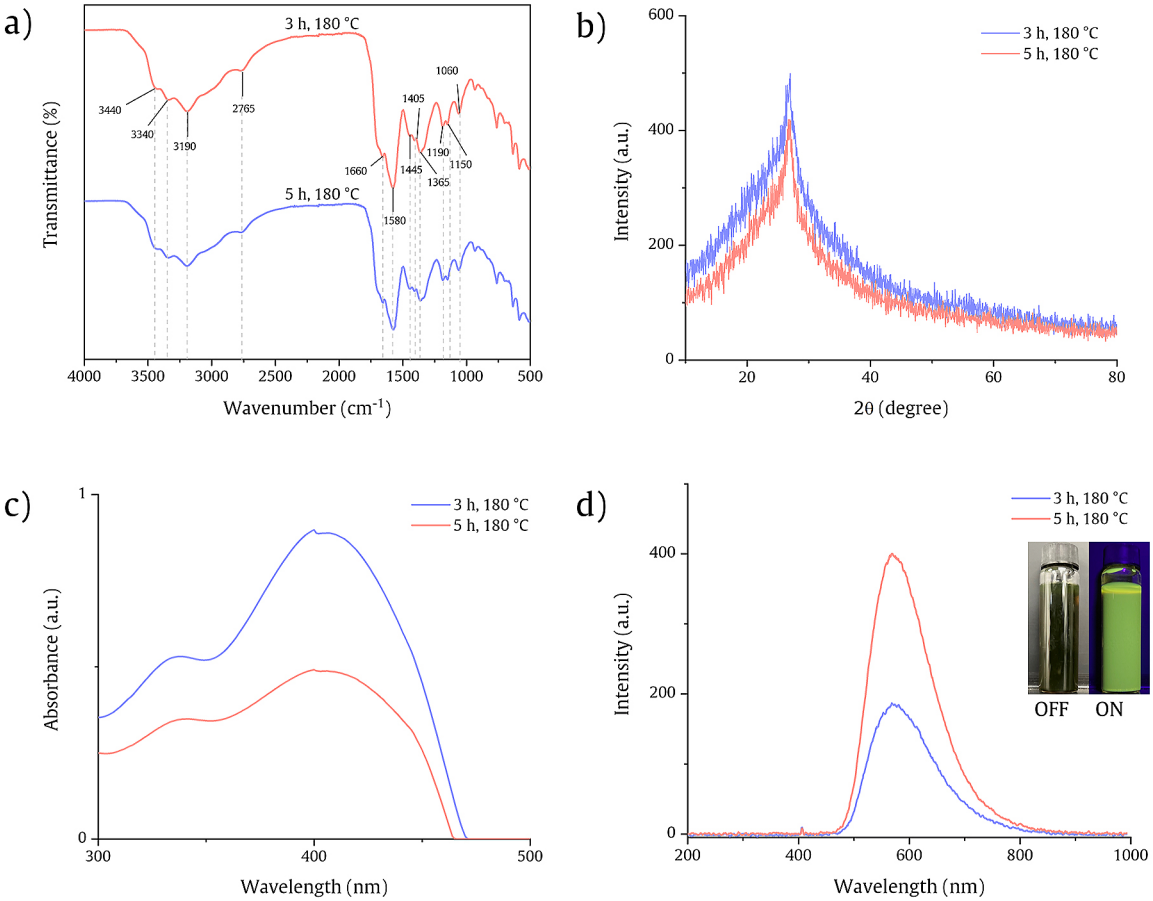


Figure 1. a) FTIR spectrum, b) XRD pattern, c) UV-Vis absorption spectra, and d) fluorescence emission spectra with photographs ("OFF" under natural light and "ON" under UV light at 365 nm) of the N-CDs.

The SEM images (Figure 2) display finely dispersed particles in a size 2-100 nm range. This morphology characteristic contributes to the high dispersion of the N-CDs particles in a solution (Nuryadin *et al.*, 2016). However, big chunks formed on the surface of the dispersed N-CDs due to agglomeration of the N-CDs. These big particles contain smaller particles with a minimum size of 2-3 nm on their surface.

 https://doi.org/10.37819/nanofab.008.299

The most common technique for QY determination is the relative method, which involves the comparison of the luminescent intensity of a sample with a standard compound. The sample and standard must be absorbed and emitted in the same regions (Hoan *et al.*, 2019). The sodium fluorescein, with a known QY of 0.79, was selected as a standard reference for the QY determination because its absorption spectra are located in the same region as the N-CDs. In addition, the solvent chosen for the N-CDs was deionized water (n = 1.33). The literature has reported that the luminescence of the CDs is the highest in water due to its stronger polarization than other solvents (Hoan *et al.*, 2019). Five different concentrations of the sodium fluorescein diluted in 0.1 M NaOH and N-CDs diluted in deionized water were prepared, so their absorption was below 0.1 to avoid the inner filter effects (Brouwer, 2011). The absorption and luminescent spectra with an absorbance and excitation wavelength of 365 nm were measured. As can be seen in Figure 3, the results of the standard (Figure 3a) and the N-CDs produced at different reaction times (Figures 3b and 3c) display a linear dependence of integral luminescence intensity on the absorbance. The QY of the N-CDs synthesized at 180 ºC for 3 and 5 hours was 16.33% and 21.80%, respectively. Interestingly, N-CDs exhibited a higher QY than the previous CDs from various natural carbon precursors reported in the literature. These results reveal that the lemon juice used in this study enhanced the QYs to a certain extent.

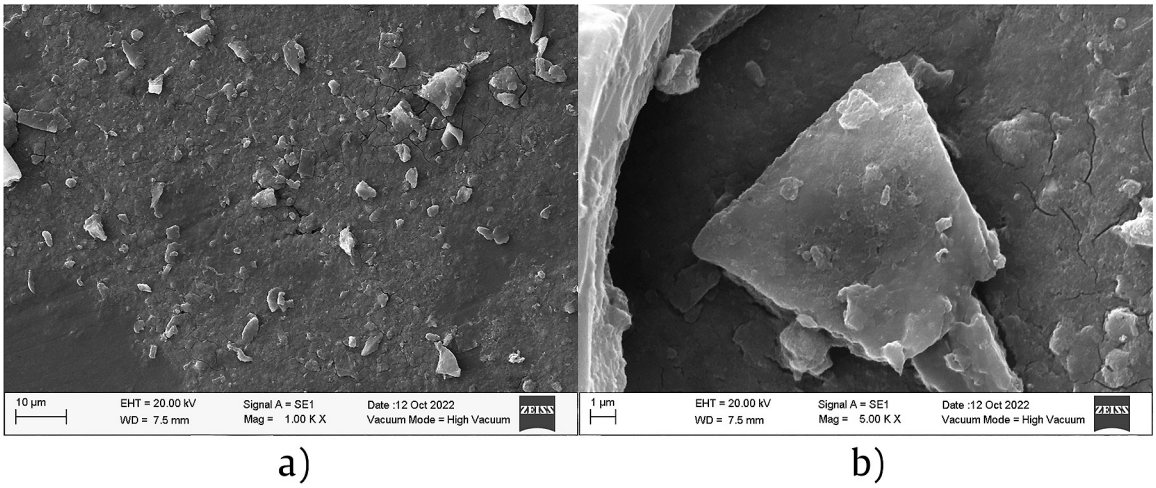
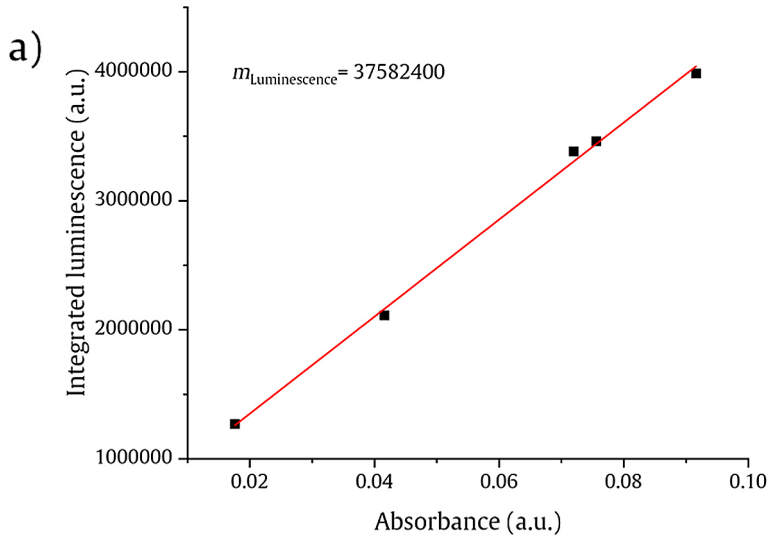


Figure 2. SEM images of the N-CDs synthesized at 180 ºC for 3 and 5 h, with zoom-in at a) 10 μm and b) 1 μm.



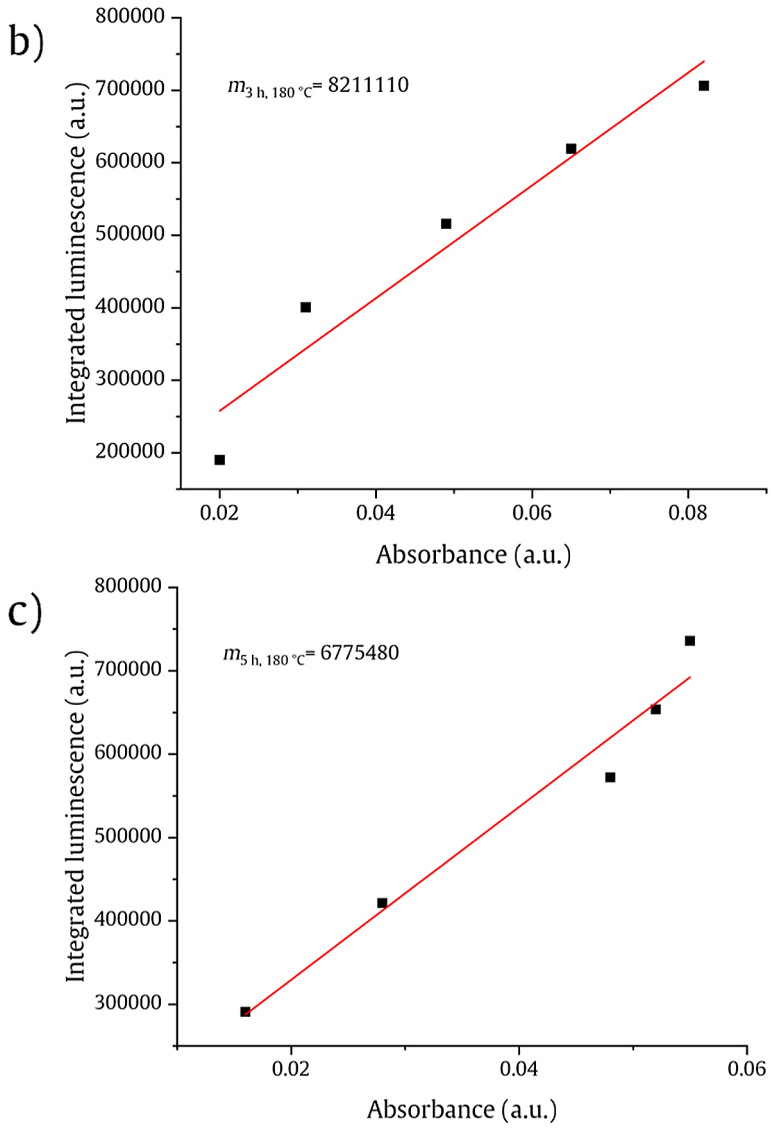


Figure 3. The dependence of luminescent intensity on the absorption of a) Sodium fluorescein, b) N-CDs synthesized at 180 °C for 3 h, and c) N-CDs synthesized at 180 °C for 5 h.

4. CONCLUSION

In this study, a one-pot method to synthesize the nitrogen-doped carbon dots (N-CDs) with strong fluorescence intensity with a QY of 16.33% and 21.33% was achieved *via* a simple, cost-effective, and eco-friendly approach without any passivation or chemical modification. The surface morphological evaluation by SEM unveils the uniform and well-crystalline appearance of in-house fabricated N-CDs. However, further research and development of strategies to fully understand the correlation of material structure and properties with the photoluminescence mechanism are still highly desirable in this field. It is hopeful that through this study, more progress can be made to maximize the potential of CDs.

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Competing interests

The authors declare no conflicting interests. ♦

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