

# Green synthesis of multi-doped carbon dots from Prickly Pear in the presence of nitrogen, phosphorus, and nitrogen-phosphorus solutions

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**Pablo Alfredo Sánchez-Pineda<sup>a</sup>, Itzel Y. López-Pacheco<sup>b</sup>,  
Angel M. Villalba-Rodríguez<sup>b</sup>, Reyna Berenice González-González<sup>c</sup>,  
Roberto Parra-Saldívar<sup>b</sup>, Hafiz M. N. Iqbal<sup>b</sup>**

**Abstract:** Carbon dots (CDs) have gained popularity in research due to their desirable characteristics in electrical, catalytic, and optical applications. The exploration of unique carbon sources with complex chemical compositions can open avenues for the straightforward production of multi-doped nanomaterials. In particular, prickly pear has distinctive properties and a mineral-rich composition, and high production yields under low water usage conditions. In this work, prickly pears were used to prepare fluorescent green multi-doped CDs through a carbonization technique in the presence of nitrogen, phosphorus, and nitrogen-phosphorus solutions at 180°C for 7 hours. Results from different characterization techniques such as Fourier Transform Infrared (FTIR), X-ray diffraction (XRD), UV-vis, and  $\zeta$  Potential demonstrated the functionalization of the surface, semi-crystalline structures, a broad absorbance at the UV range with a strong peak at 275 nm, stability in water, and negative surface charge of nitrogen-doped carbon dots (NCDs), phosphorus-doped carbon dots (PCDs), and nitrogen-phosphorus doped carbon dots (NPCDs). Overall, the prickly pear was demonstrated to be a suitable source for synthesizing multi-doped CDs while maintaining nano-synthesis towards sustainable and eco-friendly directions. .

**Keywords:** Nanomaterials; Nanotechnology; Green Synthesis; Carbon Dots.

<sup>a</sup> Tecnológico de Monterrey  
- School of Engineering and  
Sciences, Monterrey, 64849,  
Nuevo Leon, Mexico.

<sup>b</sup> Tecnológico de Monterrey  
- School of Engineering and  
Sciences, Monterrey, 64849,  
Nuevo Leon, Mexico.  
Institute of Advanced Materials  
for Sustainable Manufacturing,  
Tecnológico de Monterrey -  
Monterrey, 64849, Nuevo  
Leon, Mexico.

<sup>c</sup> Tecnológico de Monterrey  
- School of Engineering and  
Sciences, Monterrey, 64849,  
Nuevo Leon, Mexico.  
Institute of Advanced Materials  
for Sustainable Manufacturing,  
Tecnológico de Monterrey -  
Monterrey, 64849, Nuevo  
Leon, Mexico.  
reyna.g@tec.mx  
Corresponding author.

## 1. INTRODUCTION

The recent technological advance in material research has highlighted the versatility of carbon-based nanomaterials across a wide range of applications and forms. Among them, carbon dots (CDs) have raised significant interest; they are composed of a carbon core with  $sp^2/sp^3$  hybridization and different surface functional groups (Cui *et al.*, 2021). CDs exhibit advantageous features like high biocompatibility (Liao *et al.*, 2021), optical properties such as photoluminescence (Sabet and Mahdavi, 2019), and great chemical stability enabling a variety of potential applications (Liu *et al.*, 2019).

Sensing is one of the most studied applications of CDs, for example, CDs have been employed for the sensitive detection of ions (Batool *et al.*, 2022), cations (Xu *et al.*, 2015a), and organic molecules like glucose (Shan *et al.*, 2014). In addition, bioimaging applications have been widely reported because of their excellent optical properties

and biocompatibility (Wang *et al.*, 2022). Furthermore, catalytic applications have been achieved due to their easy surface functionalization, which has been usefully employed for the photocatalysis of contaminants (Yao *et al.*, 2022).

An important factor of CDs functionality is their surface functionalization, which plays a critical role for CDs either to achieve specific characteristics for certain applications or to present enhanced performances (González-González *et al.*, 2022b). A great diversity of dopants have been explored, including nitrogen, phosphorous, boron, sulfur, copper, and combinations of them (Cárdenas-Alcaide *et al.*, 2023; Kamali *et al.*, 2021; Najafu *et al.*, 2022; Sadhanala *et al.*, 2021; Tammina *et al.*, 2019; Y. Zhang *et al.*, 2018). In this regard, multi-doped CDs have demonstrated enhanced characteristics in terms of biocompatibility and photoluminescence (Miao *et al.*, 2020). The increased capabilities of multi-doped CDs have been attributed to superior electron transfer between dopant elements and CDs, resulting in radiative electron recombination and holes (Zhu *et al.*, 2023).

Eco-friendly and low-cost synthesis techniques are currently raising awareness; thus, green precursors have been presented as an excellent alternative to pure chemical precursors (González-González *et al.*, 2022c). Several waste-based, green, or natural precursors have been used for the sustainable synthesis of CDs; some examples include lemon, orange, pepper, and tires, among others (Beker *et al.*, 2020; González-González, *et al.*, 2022; Humaera *et al.*, 2021; Schneider *et al.*, 2019; Vasimalai *et al.*, 2018). Interestingly, the CDs' properties are highly dependent on the synthesis technique, process conditions, and selected precursors. Therefore, different characteristics and applications can be obtained by varying the precursor.

Although green synthesis is defined as the avoidance of toxic chemicals through the use of plant extracts or natural components, (Upadhyay *et al.*, 2019) additional aspects should be considered. For example, water utilization needs to be taken into account for future green nanomaterials since water scarcity and pollution are important issues given the recent rise in agriculture and industry (Truong *et al.*, 2024). Thus, it is critical to explore water-efficient sources for the synthesis of nanomaterials to improve them in terms of sustainability. In this context, prickly pear is a water-efficient crop because its cultivation requires less water than other crops, up to 80% less water consumption (Neupane *et al.*,

2021). Moreover, prickly pear can be an excellent source of nanomaterials due to its high content of minerals and glucose (Zenteno *et al.*, 2015). Glucose-derived CDs have been synthesized by other authors with excellent results (Ezati *et al.*, 2022); on the other hand, the high mineral content of prickly pear, can result in multi-doped CDs. In this work, we explored prickly pear as a green source for the production of CDs nanoparticles, and the possibility of achieving multi-doped CDs in the presence of nitrogen, phosphorous, and nitrogen-phosphorous medium through a simple carbonization technique.

## 2. MATERIALS AND METHODS

### 2.1. Chemicals/reagents

Prickly pears were purchased from the local market to use as a carbon source. L-asparagine and phosphoric acid were acquired from Sigma Aldrich for their usage as nitrogen and phosphorus sources, respectively. Ultra-pure (Milli-Q) water was used during the synthesis.

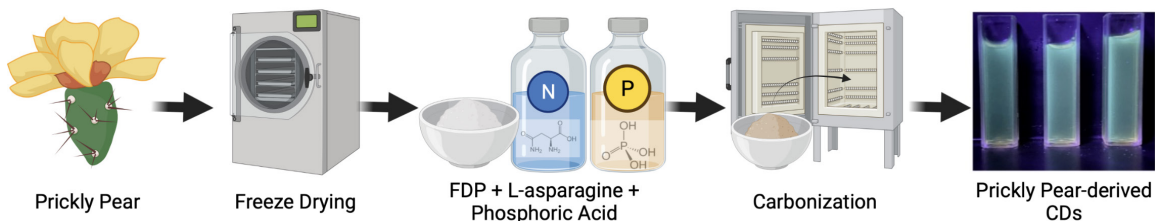
### 2.2. Preparation of CDs

Prickly pear required a pre-treatment before nano-synthesis; first, 1 kg of frozen prickly pear was blended and freeze-dried to obtain a fine powder to use as a carbon source. CDs were produced in the presence of nitrogen, phosphorous, and a combination of both elements during the synthesis process. Thus, 12 g of freeze-dried prickly pear (FDP), used as a carbon source, were placed in porcelain capsules, while the dopants were added in concentrations selected from the literature on co-doped CDs (Tadesse *et al.*, 2021). Nitrogen-doped carbon dots (NCDs) were prepared by adding 0.1g of L-asparagine to one of the capsules containing FDP with 5 mL of Ultra-pure water to dissolve the mixture. Similarly, phosphorus-doped CDs (PCDs) were synthesized by adding 10 mL of phosphoric acid (0.1M) into one of the capsules containing FDP. Finally, nitrogen-phosphorous-doped carbon dots (NPCDs) were prepared by adding both nitrogen and phosphorous sources at the same concentrations as the NCDs and PCDs.

Porcelain capsules were then placed into a Yamato DKN6026C oven at 180°C for 7 hours. After cooling down to room temperature, carbonized samples were scraped from the capsules using a stainless-steel spatula for further centrifugation

with 25 mL of Mili-Q water. CDs in suspension were then filtered using 0.22  $\mu\text{m}$  filters to finally freeze-dried them for analysis and characterization.

This procedure is shown as a schematic representation in Figure 1. The process was performed by duplicate.



**Figure 1.** General methodology for the production of multi-doped carbon dots through simple carbonization synthesis using prickly pear as the carbon source. *Note: FDP is used for Freeze-dried Prickly Pear and CDs for carbon dots.* Created with BioRender.com and extracted under premium membership.

### 2.3. Characterization

Ultraviolet-visible (UV-Vis) absorbance analysis was performed using a Lambda 365 instrument (PerkinElmer) with a 200–800 nm range, to understand the CDs' light exposure absorbance behavior. Fourier-transform infrared (FTIR) spectra were obtained using a Frontier TM FTIR Spectrometer (Perkin Elmer) to understand the chemical bonds in the CDs. X-ray diffraction (XRD) patterns were analyzed with a Rigaku Miniflex 600, using a Cu- $K\alpha$  lamp as a radiation source with a current and voltage of 15mA and 30Kv to evaluate the crystallinity.  $\zeta$  potential analysis was performed to evaluate surface charge using the phase-analysis light scattering (PALS) method with a Nanobrook 90 Plus Pals.

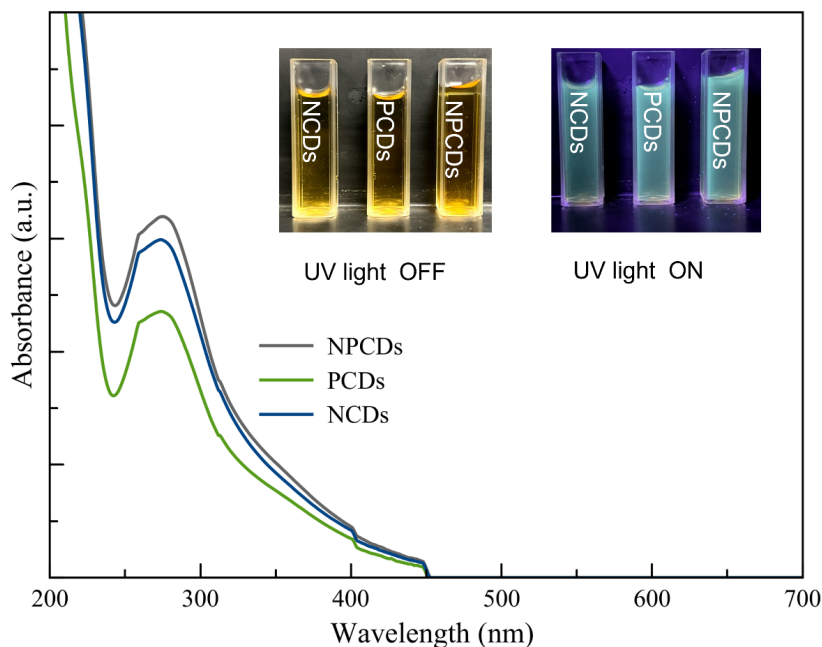
## 3. RESULTS AND DISCUSSION

UV-vis spectroscopy was utilized to analyze the absorption properties of the synthesized CDs. The CDs presented absorbance in both UV-B and UV-A ranges, which is in agreement with other reports (Arroyave *et al.*, 2021; Cárdenas-Alcaide *et al.*, 2023). As shown in Fig. 1, the absorbance of the CDs showcases a peak at around 275 nm, which can be associated with orbitals transition (Jiang *et al.*, 2020). The difference in dopants creates a variable in the absorbance of the CDs, where NPCDs showed a higher absorbance at 275 nm than PCDs, as expected from previous works. Nitrogen-doped CDs have demonstrated enhanced optical properties and good dispersibility (Zhou *et al.*, 2019); while, phosphorous-doped CDs usually present decreased optical properties, but improved stability and dispersibility (Kalaiyarasan *et al.*, 2020). Thus, it is suggested that the combination of both dopants

might create CDs with enhanced optical properties (Park *et al.*, 2020; Tammina *et al.*, 2019) as can be observed in Fig. 2.

The XRD pattern exhibited peaks at 27°, 22°, 29°, and 40° (Fig. 3). Interestingly, the XRD pattern can be compared with other studies that associate the peaks at 29° and 27° as part of a composite of carbon with manganese (Rather, 2019). Also, other peaks agreed with the potassium-nitrogen-phosphorus carbon hollow structure (Xu *et al.*, 2021). The presence of these elements can be explained by the synthesis process and the presence of several components of the prickly pear, which included manganese and potassium (Cota-Sánchez, 2016). NCDs presented clear amorphous hump and crystalline peaks, while NPCDs and PCDs presented well-defined crystalline peaks and less-defined amorphous humps. Overall, the XRD patterns of all synthesized CDs did not present a well-defined crystalline or amorphous structure, indicating a semi-crystalline structure. This type of semi-crystalline CDs has reported improved optical properties (Xu *et al.*, 2015b), which might be used for bio-imaging applications.

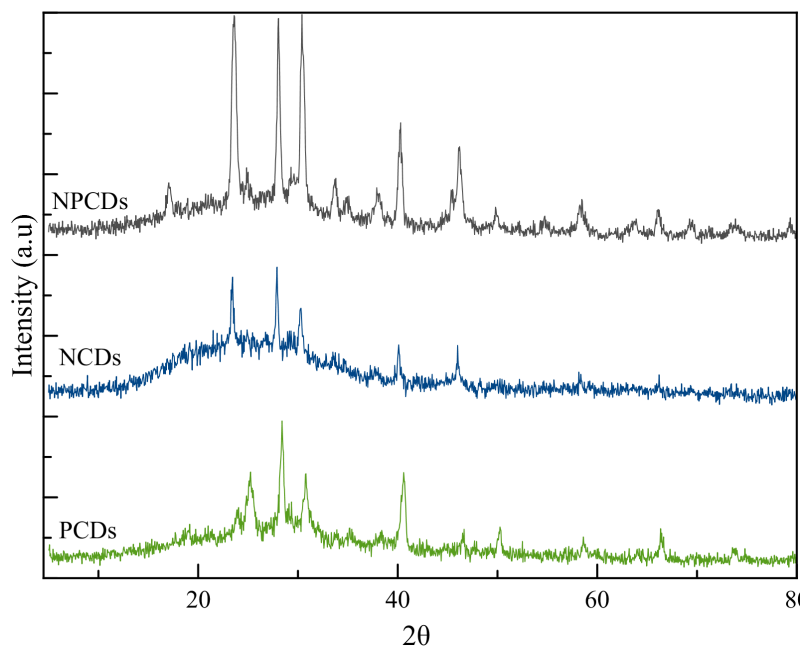
In addition, all three samples presented different intensities and shifts of their crystalline peaks, which can be associated with pH variations caused by the nitrogen and phosphorous sources added during the synthesis. Although the precise nucleation process of the simple carbonization synthesis performed in this study is not yet clear, other methods like hydrothermal and pyrolysis have reported that pH and mineral composition of samples are key factors during the CDs' nucleation (De and Karak, 2013; Hu *et al.*, 2015; Qu *et al.*, 2014). Thus, such variations may lead to the transformation of mineral contents, generating nucleation differences.



**Figure 2.** UV-visible absorption spectra of green synthesized prickly pear carbon dots in solutions rich in nitrogen (NCDs), phosphorus (PCDs), and nitrogen-phosphorus (NPCDs).

Furthermore, the formation of amorphous phases is in agreement with other studies, for example, glucose-derived CDs through hydrothermal technique reported the nucleation of CDs through a nucleation-polymerization process; the presence of crystalline CDs surrounded by an amorphous

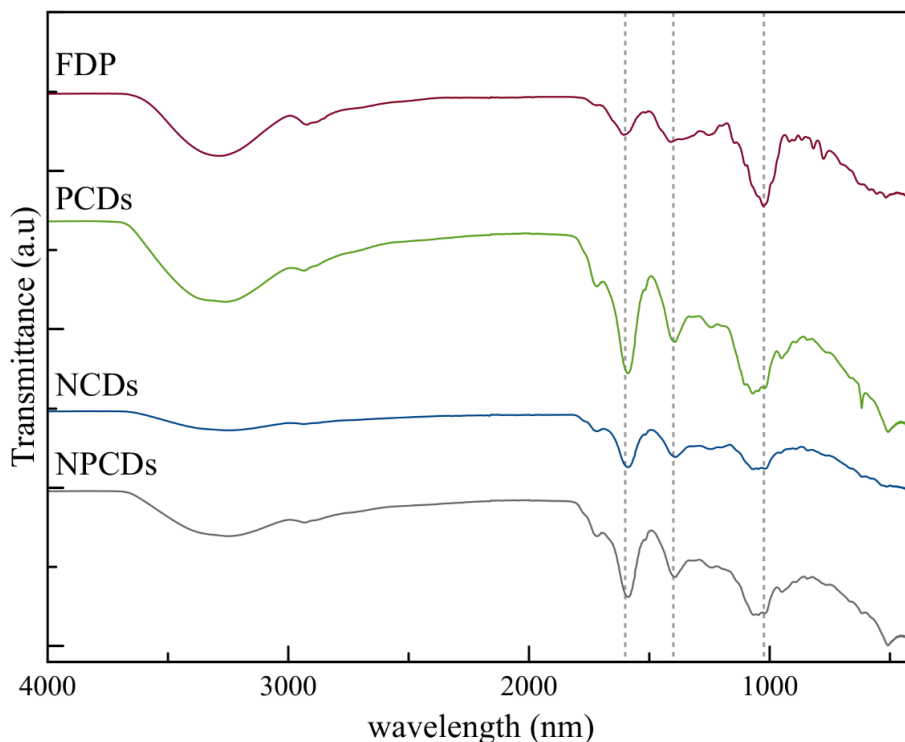
carbon matrix was confirmed by TEM (Papaioannou *et al.*, 2018). In addition, the differences found in the crystalline peaks and their shifts could be explained by the pH differences of the synthesis mediums and the mineral content of the prickly pear causing variations during the nucleation.



**Figure 3.** XRD spectra of green prickly pear-derived carbon dots doped with nitrogen (NCDs), phosphorus (PCDs), and nitrogen-phosphorus (NPCDs) multi-doped carbon dots showing peaks at 27°, 22°, 29°, 40°.

To identify the components of the CDs, FTIR spectra were obtained for all CDs samples. The surface chemistry of all CDs samples differs between them in terms of peak position and intensity, as can be observed in Fig. 4. Different band gaps confirmed the proper formation of CDs and the nitrogen attachment to the CDs' surface; N–H, C–H, C=O, C=C, C–N, C–O were confirmed by the presented band gaps at 3200  $\text{cm}^{-1}$ , 1650  $\text{cm}^{-1}$ , 1580  $\text{cm}^{-1}$ , 2925  $\text{cm}^{-1}$ , 1420  $\text{cm}^{-1}$ , and 1024  $\text{cm}^{-1}$ , respectively (Beker *et al.*,

2020). Furthermore, weak peaks at 880  $\text{cm}^{-1}$  and the broad gap at 1370  $\text{cm}^{-1}$  suggested the presence of P–O and P=O in PCDs and NPCDs (Shangguan *et al.*, 2017; Tammina *et al.*, 2019). Other weak peaks can be observed, it is suggested that those peaks are caused because of the natural complex composition of prickly pear which is minerals-rich (Zenteno. *et al.*, 2015). This mineral-rich composition of FDP sample—and its contribution to the CDs' composition—was confirmed with its FTIR spectra.



**Figure 4.** FTIR spectra of freeze-dried prickly pear (FDP) and their derived carbon dots doped with nitrogen (NCDs), phosphorus (PCDs), and nitrogen-phosphorus (NPCDs).

Table 1 shows the results of the  $\zeta$  potential indicating negative surface charge of all three CDs samples. The negative charge might be attributed to the presence of hydroxyls and carbonyl groups presented on the surface of the CDs, which is in agreement with the FTIR results and literature (Bao *et al.*, 2018; González-González, *et al.*, 2022; Mondal *et al.*, 2018).

The  $\zeta$  potential analysis was performed after two months of synthesis. NPCDs, PCDs, and NCDs samples were suspended in Milli-Q water showing high hydrophilic characteristics and no agglomerations after two months. The potential values and the observed hydrophilic characteristics are similar to those reported in other studies, in which

multi-doped CDs samples presented values below  $-10$  mV with negative surface charges, good stability in neutral solutions, and high hydrophilic characteristics (Chandra *et al.*, 2018; Qu *et al.*, 2020; Singh *et al.*, 2018; L. Zhang *et al.*, 2019).

Sample	$\zeta$ potential (mV)	
Nitrogen-doped carbon dots	$-3.48 \pm 0.67$	
Phosphorus-doped carbon dots	$-8.83 \pm 3.39$	
Nitrogen-phosphorus multi-doped carbon dots	$-0.45 \pm 0.24$	

**Table 1.**  $\zeta$  potential results of prickly pear-derived carbon dots



## 4. CONCLUSIONS

It is important to consider the current issues regarding water and resource scarcity for the proper selection of precursors for the synthesis of nanomaterials, thus, nano-synthesis research can be oriented towards greener directions. This study aims to evaluate the employment of prickly pear as a natural source of nanomaterials by taking advantage of its interesting chemical composition. Thus, multi-doped carbon dots were synthesized through a one-pot, simple, and low-cost synthesis procedure using prickly pear as the carbon source due to its natural high glucose content and its interesting mineral content. The synthesized carbon dots demonstrated the potential of prickly pear as an excellent carbon source for the synthesis of multi-doped CDs. They exhibited a high carboxyl functionalization on their surface, which was confirmed by the FTIR technique and  $\zeta$  potential results. Their characteristics seem suitable for catalytic applications; however, further studies must be developed to evaluate and understand the catalytic effect of the dopant elements. Furthermore, this simple synthesis of carbon dots opened the possibility of studying the synthesis of self- and multi-doped carbon dots avoiding the usage of dopant sources to explore the natural mineral content of prickly pear fruits to achieve self-surface functionalization. The semi-crystalline general structure of the carbon dots was confirmed by XRD; in addition, carbon dots showed good optical properties according to UV-Vis technique. Further research should be conducted in this direction to shed light on the functionality of sustainable multi-doped carbon dots.

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## Conflict of interests

The authors declare no conflict of interest. ♦

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