SYNTHESIS AND CHARACTERIZATION OF CARBON DOTS DERIVED FROM BIOMASS SOURCES

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Abstract

Carbon dots (CDs), also known as C-dots, are microscopic carbon nanoparticles with dimensions less than ten nanometers. These particles possess numerous distinctive characteristics and have witnessed a growing use across diverse domains in recent times. In this report, CD was synthesized from various sources of organic waste peels (pomelo, dragon fruit, passion fruit, pineapple, mung bean, watermelon, and carrot), focusing on the hydrothermal method. The main target is to find appropriate biomass materials for the production of cost-effective, eco-friendly, and non-hazardous CDs. The morphology, crystal structure, optical and luminescent properties were investigated and evaluated for perspective applications in biomedical issues, optoelectronics, catalysis, anticounterfeiting and sensors.

Keyword: *Biomass, carbon dots, hydrothermal, photoluminescence, semiconductor nanomaterials.*

NGHIÊN CỨU TỔNG HỢP VÀ KHẢO SÁT CÁC TÍNH CHẤT CỦA CÁC CHẤM CARBON CÓ NGUỒN GỐC TỪ CÁC NGUỒN SINH HỌC

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Tóm tắt

Chấm carbon (CDs), còn được gọi là chấm-C, là các hạt nano carbon có kích thước nhỏ hơn 10 nm. Các hạt này có nhiều đặc điểm riêng biệt và đã được ứng dụng rộng rãi trong nhiều lĩnh vực khác nhau trong thời gian gần đây. Trong nghiên cứu này, CD được tổng hợp từ nhiều nguồn vỏ chất thải hữu cơ khác nhau (bưởi, thanh long, chanh dây, dứa, đậu xanh, dưa hấu và cà rốt) bằng phương pháp thủy nhiệt. Mục tiêu chính là tìm ra vật liệu sinh khối phù hợp để sản xuất CDs với giá thành thấp, thân thiện với môi trường và không độc hại. Hình thái, cấu trúc tinh thể, tính chất quang và quang phát quang được nghiên cứu và đánh giá để ứng dụng trong các vấn đề y sinh, quang điện tử, xúc tác, chống hàng giả và cảm biến.

Từ khóa: *Chấm carbon, phát quang, sinh khối, thủy nhiệt, vật liệu nano bán dẫn.*

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1. Introduction

Over the past decades, the substantial rise in food waste has increasingly become a critical concern. The huge fruit consumption makes peels an underutilized source of garbage. Fruit peels are frequently discarded rather than processed or recycled, causing pollution. To attain sustainable growth, it is imperative to identify solutions that convert waste into valuable resources, especially in tropical countries like Vietnam. The transformation of naturally derived compounds into specialized carbon nanostructures has garnered significant interest. This transformation can optimize the use of natural materials and minimize waste, while concurrently producing diversely usable materials with exceptional qualities (Dong et al., 2024).

Recently, carbon dot*s* have attracted significant attention due to their numerous potential applications in biosensors (Pourmadadi et al., 2023), bioimaging (Bartkowski et al., 2024; Jana & Dev, 2022), LED devices (Liu et al., 2020), anticounterfeiting (Jiang et al., 2016), solar cells (Gao et al., 2020), and supercapacitors (Xiao et al., 2021). They have presented outstanding characteristics such as stable luminescence intensity, broad excitation spectrum, multicolor fluorescence, and water solubility…The properties of quantum dots are closely dependent on many factors, including size, shape, purity, source materials, and the fabrication process. The synthesis of carbon dot*s* can be proceeded from pure chemicals or from biomass waste sources (Jelinek, 2017). The biomass sources consist of compounds such as carbohydrates and natural organic acids including glucose, fructose, sucrose, ascorbic acid, and citric acid. These chemicals serve as derivatives of carbon precursor (Cui et al., 2021). Therefore, using discarded fruit peels as precursor to fabricate carbon dot*s* can solve the environment pollution problem and achieve novel nanomaterial in parallel.

This paper reports the hydrothermal synthesis and characterization of carbon dots from biomass peels such as pomelo, dragon fruit, passion fruit, pineapple, mung bean, watermelon, and carrot. After grinding and extracting pure solutions from various peels, the organic precursor solutions are sealed and reacted in different hydrothermal reactors at a high temperature (170 °C) for 3 hours. The optical properties, morphology and structures of the carbon dot*s* were analyzed using Transmission electron microscopy (TEM), UV-Vis absorption spectroscopy, Fourier-transform infrared spectroscopy (FTIR), Raman spectroscopy, photoluminescence (PL) spectroscopy. The results reveal that, with the prior mentioned hydrothermal condition, carbon dots from pomelo, pineapple present good quality, great optical properties and suitable for different applications.

2. Experiments

Raw materials

Materials: different kinds of peels from pomelo, dragon fruit, passion fruit, pineapple, mung bean, watermelon, and carrot.

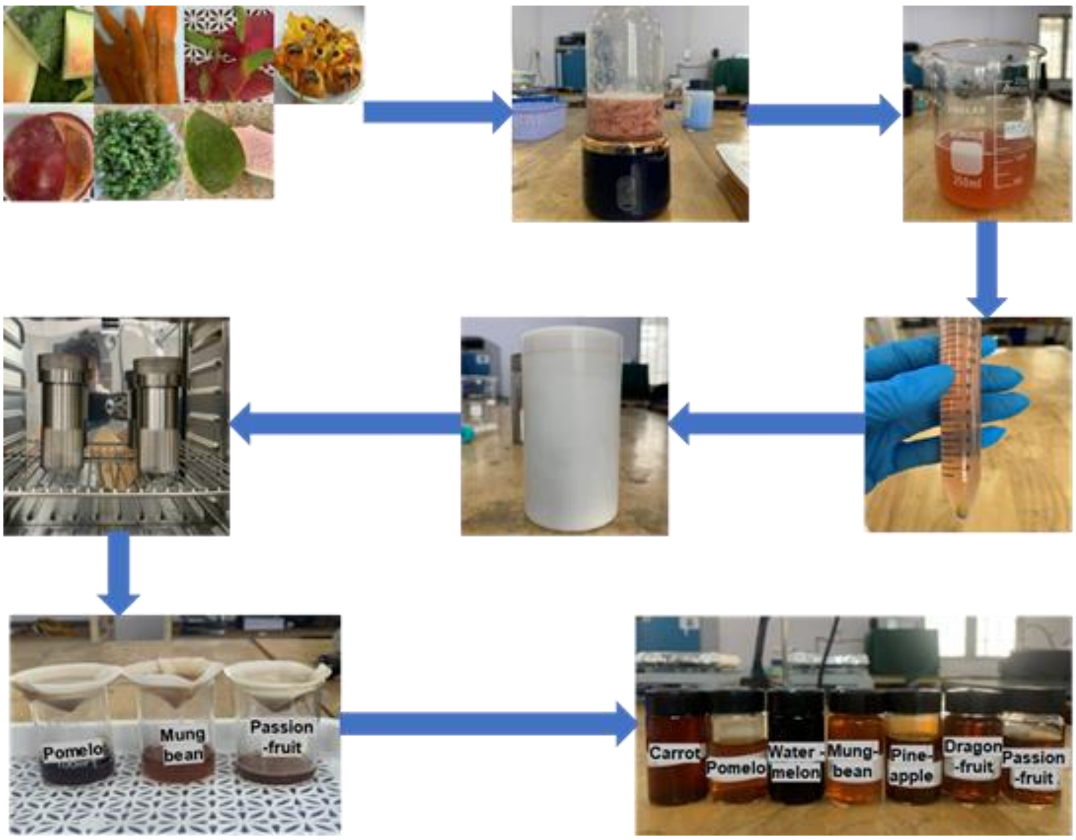

Figure 1. Carbon dot synthesis process

Experiments

The fruit peels were rinsed several times with pure water to remove dust and grime. Then, they were dried naturally in ambient air. Each kind of peels was minced into small fragments. They were mixed with distilled water, ground finely and subsequently each solution was run through two layers of filtering papers. This filtration process was repeated twice for each sample. After that, each filtered sample underwent centrifugation at a speed of 3000 rpm for 15 minutes to eliminate any leftover residue. After centrifugation, 60 ml of each solution samples was transferred to different Teflon flasks. The hydrothermal process was set at 170 °C in three hours. The resultant solutions were further subjected to the final filtration using filter paper to eliminate sizable carbon fragments. The obtained products were solutions that appeared in yellow-brown or light brown color. Synthesis process is briefly illustrated in Figure 1.

Formation of carbon dots

The hydrothermal synthesis of carbon dots from natural waste products involves several important phases, including carbonization, polymerization, condensation, and surface modification (Cui et al., 2021; Jelinek, 2017; Vasimalai et al., 2018). High temperatures are applied to the raw material during the carbonization stage, which causes organic bonds to break down and dehydrate, resulting in the creation of polymer structures and long chain molecules. Soluble polymers are then formed as a result of polymerization and condensation. Nucleation and the formation of nanoparticles take place when the concentration of aromatic clusters rises to a high enough temperature. These molecular groups subsequently merge to form nano-sized quantum dots (Cui et al., 2021; Jelinek, 2017; Vasimalai et al., 2018).

Organic functional groups like -COOH, -OH, or -NH_2 are attached to the surface of CDs to modify their optical and chemical properties (Mao et al., 2014). This process increases the solubility of the CDs in water and their capacity to interact with the environment. Different filtration methods are frequently employed as post-synthesis techniques to eliminate contaminants and by products.

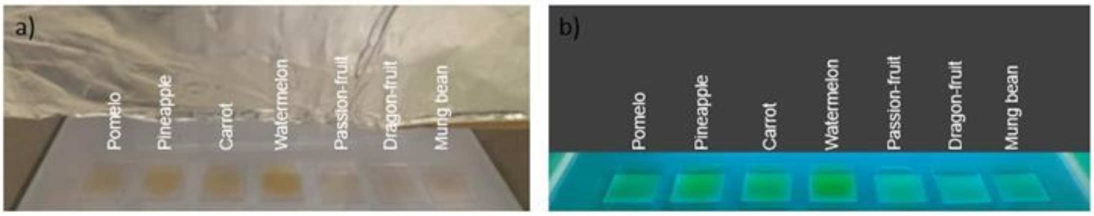

Figure 2. a) CDs before UV light exposure b) CDs after UV light exposure.

Figure 2a shows the yellow-brown and bright yellow color of the carbon dot dropped and dried naturally on glass substrates prior to UV light irradiation. When exposed to UV light in Figure 2b, the carbon dots derived from the seven kinds of fruit peels generally emitted greenish yellow light.

3. Results and discussion

Figure 3 displays the absorbance spectra of the CQD solutions. All samples exhibit an absorption peak at a wavelength of 283 nm, with the exception of sample derived from dragon fruit and mung beans. In dragon fruit spectrum, a broad peak at 265 nm indicates that this sample possesses a relatively smaller particle size in comparison to other samples. This peak blueshift could result from quantum confinement effect, in which smaller particle size would lead to higher optical bandgap. However, low absorption intensity could originate from the low carbon dot formation using synthesis condition mentioned above. The same conclusion could be applied to mung bean case. The carrot, pineapple and watermelon samples present a small absorption peak at 230 nm, whereas the pomelo sample shows an extra small peak at 345 nm. The absorption peaks at 230 nm and 283 nm are caused by the π−π* transition of the C=C bond in the carbon core, whereas the absorption peak at 345 nm is associated with the n−π* transition of the C=O bond (Yadav et al., 2023).The absorption region due to the n-π* transition of C=O and π-π* of C=C is characteristic of CDs synthesized from various biomass sources.

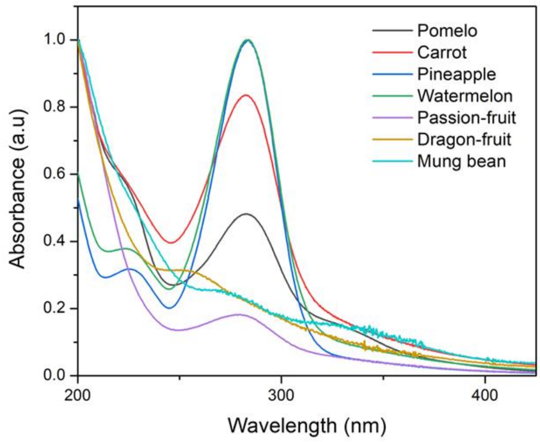

Figure 3. Normalized UV-Vis spectra of CDs solution from different types of fruit peels

The UV-Vis absorption spectroscopy results confirm the formation of carbon nanoparticles from pomelo peel, pineapple peel, carrot peel, watermelon peel, passion fruit peel, and dragon fruit peel, in accordance with previous studies (Wu et al., 2023). Based on UV-Vis spectra, peels from mung bean, dragon fruit and passion fruit seem not suitable for hydrothermal condition, which requires 170 °C for three hours.

For further investigation of optical properties of CDs from biomass sources, photoluminescent spectra were taken under stimulated wavelength of 350 nm.

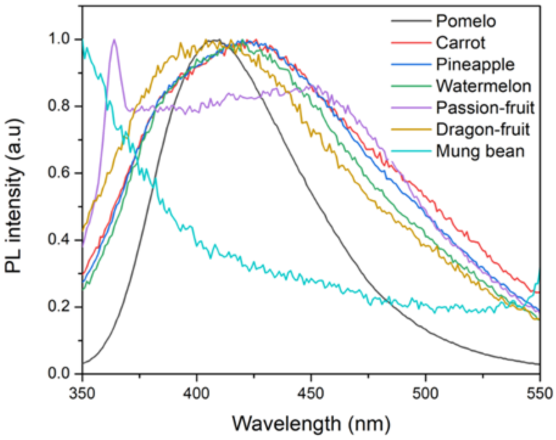

Figure 4. Normalized photoluminescent spectra of CDs synthesized from different types of waste products under stimulated wavelength of 350 nm

Figure 4 shows the photoluminescence spectra of CDs produced from natural waste products stimulated by 350 nm monochromatic photons. The CDs exhibit emission peak positions at the following wavelengths: pomelo peel (408 nm), carrot peel (421 nm), pineapple peel (421 nm), watermelon peel (421 nm), and dragon fruit peel (406 nm). In the case of passion fruit peels, the fluorescence emission is probably caused by the passion fruit's molecules, not by carbon dots. The absence of an emission peak in the mung bean sample can be attributed to the insufficient time and temperature during the carbonization process. This prevented the organic bonds from separating into organic chains and forming carbon dots, thus resulting in the absence of any discernible characteristics. Pomelo sample delivers clear, narrow photoluminescent peak compared to the rest. This is a strong indication for good quality of CDs synthesized from pomelo precursor (Roy et al., 2015).

Based on UV-Vis and photoluminescent spectra, morphology of TEM image of CDs from pomelo peels is taken and displayed in Figure 5. The size distribution is rather uniform and majority of the round-shaped CDs exhibited less than 5 nm in diameter. The hydrothermal condition in this work is quite suitable for synthesis of CDs from pomelo peels.

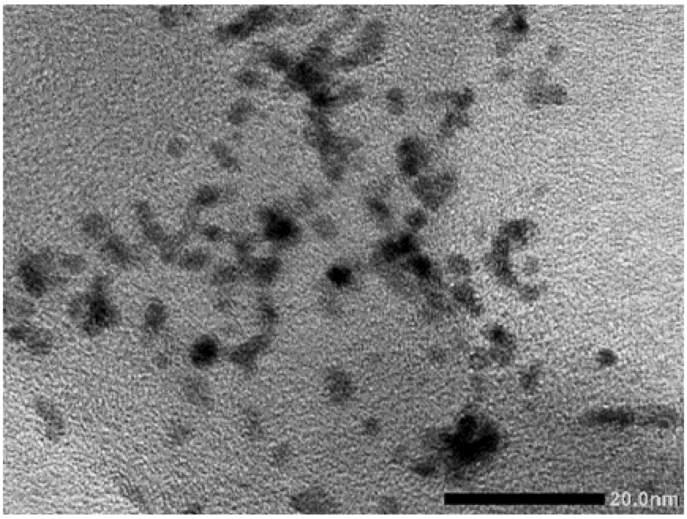

Figure 5. TEM image of carbon dots derived from pomelo peels

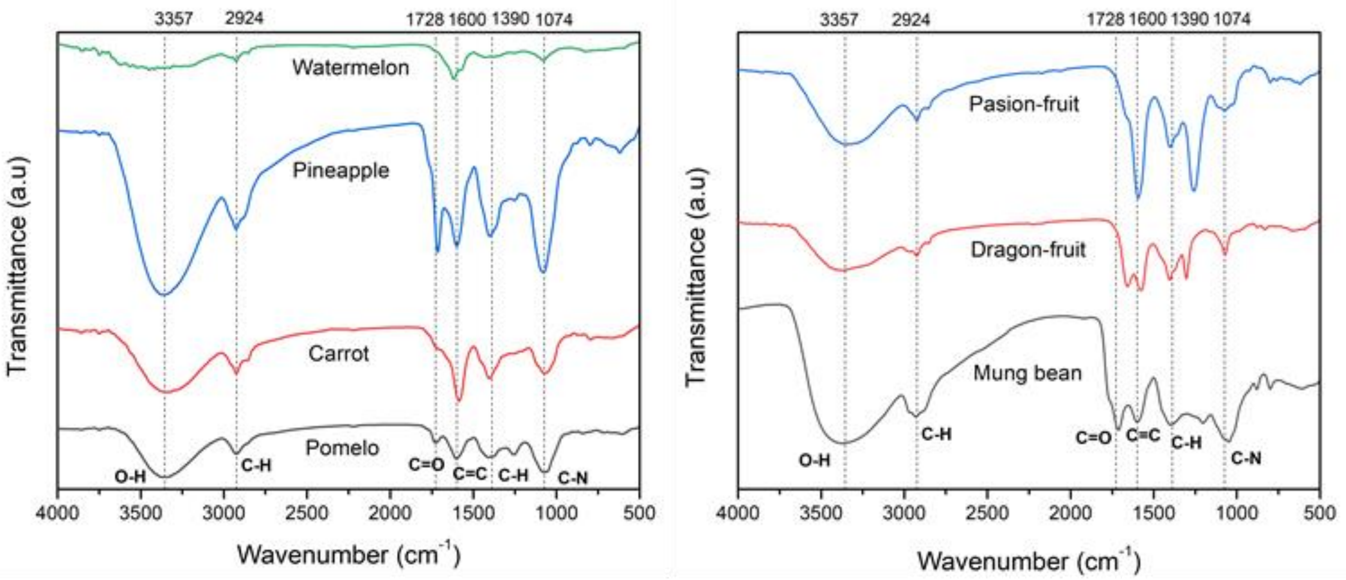

Figure 6. FTIR spectra of carbon dot synthesized samples.

The FTIR spectra of different biomass-derived CDs samples are depicted in Figure 6. It can be seen that their spectral structures are comparatively similar. Strong and wide absorption band observed in the 3000-3500 cm^{-1} region, usually ascribed to the hydroxyl group's stretching vibrations (O-H). Strong hydrogen bonding interactions and water solubility are frequently indicated by the presence of hydroxyl groups. The existence of hydrocarbon chains in the structure of CDs is indicated by the peak absorption at 2924 cm^{-1}, which is typical of the stretching vibrations of C-H bonds in saturated carbon chains. Furthermore, it is possible to link the absorption bands at around 1700 cm^{-1} to the stretching vibrations of the carbonyl group (C=O), which is frequently seen in amide or carboxyl compounds. The vibrations of the C=C bonds in aromatic rings or the vibrations of cutting the hydroxyl group are linked to the absorption range, which is roughly 1400 cm^{-1} to 1600 cm^{-1}. Furthermore, at the peaks of 1390 cm^{-1} and 1074 cm^{-1}, which correspond to the stretching peaks of C-H and C-N, respectively (Jelinek, 2017). From the obtained FTIR results, there are some important polar functional groups such as O-H and C=O in the structure of the carbon dots. The presence of these functional groups is a key factor explaining why carbon dots have good solubility in water. These polar functional groups not only help CDs dissolve easily in water but also affect their optical properties and their chemistry (Huo et al., 2021; Tang et al., 2019; Wu et al., 2023).For better understanding on the structure as well as the properties of quantum dots. We conducted a further analysis using Raman spectroscopy.

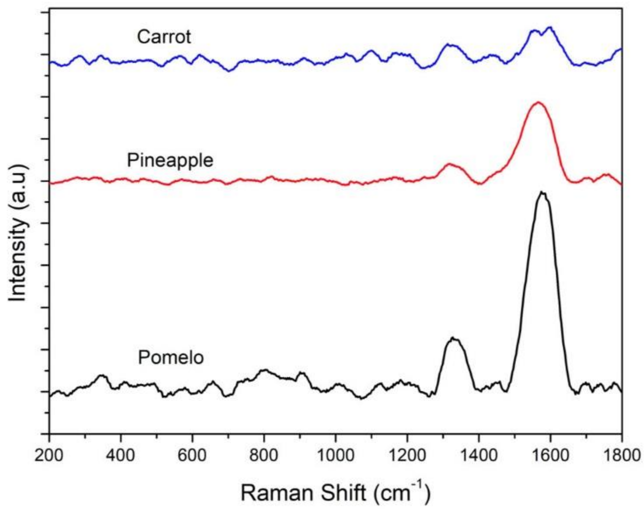

Figure 7. Raman spectrum of carbon dots synthesized from pomelo peel, pineapple peel and carrot peel

The results of the Raman spectrum analysis of CDs synthesized from three types of fruit peels can be observed in Figure 7. There appear two characteristic peaks, namely the D and G peaks. The D band typically locates at 1350 cm^{-1} and represents the vibrations of carbon atoms in disordered or imperfect structures, such as defects or disruptions in the lattice. In contrast, the G band exists around 1600 cm^{-1}. It relates to the stretching vibrations of carbon atoms in sp^2 graphitic structural networks and reflects the crystalline properties and conjugated structure of the material (Cui et al., 2021; Jelinek, 2017). The I_D/I_G intensity ratio in Table 1 indicates the degree of crystallinity of the core carbon atoms compared to the surface atoms.

From the results, it shows that the CDs synthesized from pomelo and pineapple peels have the rather low I_D/I_G ratios of 0.27 and 0.25, respectively. These values indicate the high degree of crystallinity of the core carbon atoms, a low level of defects in their structures (Cui et al., 2021).

Table 1. I_D/I_G ratio of CDs samples synthesized from pomelo peel, pineapple peel and carrot peel.

CDs samples	I_D/I_G
Pomelo peel	0.27
Pineapple peel	0.25
Carrot peel	0.62

4. Conclusion

Using a hydrothermal method at temperature of 170 °C for a duration of 3 hours, we have successfully synthesized carbon dots from various biomass waste peels, specifically pomelo, dragon fruit, pineapple, watermelon, and carrot. The carbon dots appear to present great water solubility due to hydroxyl and carboxyl functional groups on the surfaces. Samples derived from pomelo, pineapple and carrot peels also present quite good optical properties and high crystallinity. The obtained carbon dots are very promising for different perspective applications, such as in biosensors, LED, supercapacitors, anticounterfeiting.

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