

AGRITECH

National Research Centre for Agricultural Technologies

BOtanical REsources for ALternative battEries - “BO.RE.AL.E.”

**AMBITO: NUOVE MOLECOLE, PRODOTTI E PROCESSI AD ALTRO
VALORE AGGIUNTO PER LA VALORIZZAZIONE DI RIFIUTI, SCARTI,
SOTTOPRODOTTI E COPRODOTTI AGRICOLI O PER
L'AGRICOLTURA**

ASCLA SOCIETA' COOPERATIVA IMPRESA SOCIALE (Leader)

RADICE CUBICA S.R.L. (Partner)

UNIVERSITÀ DEL SALENTO (Partner)

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2. Executive Summary

Carbon dots (CDs) are emerging as a highly versatile class of nanomaterials due to their small size (<10 nm), water dispersibility, low toxicity, and photoluminescence. This report presents the results of a study aimed at synthesizing carbon dots from plant-based waste materials using green, scalable methods. Two synthetic approaches were investigated: acetic acid-assisted thermal carbonization and microwave-assisted carbonization. Among the tested biomasses—tea leaves, *Laurus nobilis* leaves, outer artichoke leaves, and hemp stalks—*Laurus nobilis* proved to be the most effective precursor, offering a yield four times higher than the others and demonstrating high reproducibility. Although artichoke and hemp also generated CDs with comparable structural and optical properties, their lower yields represent a limitation for potential scale-up. Overall, the study confirms that microwave-assisted synthesis is an efficient and environmentally friendly method for carbon dot production, and highlights the relevance of precursor selection for sustainable nanomaterial synthesis.

3. Introduction

Carbon dots (CDs) are quasi-spherical carbon-based nanomaterials, typically below 10 nm in size, characterized by high aqueous solubility, good photostability, tunable fluorescence, and surface functionalizability. These features render them particularly promising for applications in bioimaging, drug delivery, optoelectronics, catalysis, and environmental monitoring.

Despite the promising features of CDs, one of the main barriers to their widespread application remains the development of sustainable, cost-effective, and reproducible synthesis protocols. In particular, the choice of renewable biomass precursors—abundant and inexpensive—plays a critical role in promoting eco-friendly nanomaterial production in line with green chemistry principles. This study investigates various waste biomasses from agro-industrial processes as carbon sources. The goal was to evaluate their suitability in producing CDs through two low-energy synthesis techniques: acetic acid-assisted thermal carbonization and microwave-assisted carbonization. The selected plant matrices included tea leaves (as a model), *Laurus nobilis* (bay leaves), outer leaves of the artichoke flower, and stalks of hemp (Canapa). These materials were

selected due to their structural richness in lignocellulosic content, which is generally favorable for carbonization processes.

4. Methodology

Acetic Acid-Assisted Thermal Synthesis

This synthesis was used only for tea leaves. Dried leaves (600 mg) were soaked in 20 mL of 0.1 M acetic acid at room temperature for 48 hours. This step facilitated partial hydrolysis of structural polysaccharides. The pre-treated biomass was then heated at 100 °C for 24 hours to induce mild thermal carbonization. After cooling, the reaction mixture was centrifuged at 10,000 rpm for 30 minutes. The supernatant was collected and redispersed in Milli-Q water to obtain a stable colloidal suspension of CDs.

Microwave-Assisted Carbonization

This method was applied to *Laurus nobilis*, artichoke outer leaves, and hemp stalks. Each dried biomass (3 g) was irradiated at full power for 3 minutes in a microwave-compatible vessel. Rapid localized heating promoted efficient carbonization. Post-synthesis, samples were centrifuged at 10,000 rpm for 30 minutes and resuspended in Milli-Q water.

Post-Treatment and Characterization

All samples were subjected to the same characterization workflow:

- Dynamic Light Scattering (DLS) and Transmission Electron Microscopy (TEM) for size and morphology.
- Fourier Transform Infrared Spectroscopy (FTIR) for surface chemistry.
- UV-Vis and fluorescence spectroscopy for optical properties.
- X-ray Diffraction (XRD) for structural features.
- Zeta potential analysis for colloidal stability.
- Thermal conductivity measurements to assess physical behavior.

5. Results and Discussion

Results

All biomass sources successfully yielded fluorescent CDs dispersed in aqueous media. Both DLS and TEM confirmed uniform, quasi-spherical particles with average sizes between 3–5 nm. FTIR analysis revealed consistent functional groups across samples, including hydroxyl, amine, carbonyl, and ether groups—indicative of hydrophilic surfaces and stable dispersion.

- UV-Vis spectra showed absorption centered at ~300 nm.
- Fluorescence was observed in the visible range and remained stable after 96 hours.
- XRD patterns showed broad peaks at ~22°, confirming amorphous structure.
- Zeta potential for *Laurus*-derived CDs was -15 ± 2 mV, indicating negative surface charge and good colloidal stability.

Despite similar quality, yields varied significantly:

- *Laurus nobilis*: ~200 mg CDs from 3 g biomass.
- *Artichoke leaves*: ~50 mg CDs.
- *Hemp stalks*: ~50 mg CDs.

Discussion

The results reveal that while all three biomasses (*Laurus*, artichoke, and hemp) are chemically suitable due to their lignocellulosic content, *Laurus nobilis* consistently produced the highest yield and most reproducible results. Its favorable chemical composition appears to enhance carbonization efficiency under microwave irradiation. The low yields from artichoke and hemp may be related to structural resistance to rapid thermal decomposition or differing moisture retention properties. The microwave-assisted route was especially advantageous due to minimal reaction time, absence of solvents or reagent and potential scalability. Interestingly, while yield impacts process sustainability, it does not influence the intrinsic properties of the CDs. Morphology, dispersion, optical features, and thermal behavior were consistent across all biomasses, emphasizing the decoupling between process yield and nanomaterial performance.

6. Conclusions

This study demonstrates the feasibility of synthesizing carbon dots from plant-based waste using green synthetic strategies. Among the tested precursors, *Laurus nobilis* stood out for its superior yield, stability, and reproducibility. Although artichoke and hemp also yielded structurally suitable CDs, their lower conversion rates suggest the need for process optimization or pre-

treatment. Microwave-assisted carbonization emerged as the most promising method, combining efficiency with environmental sustainability. The resulting CDs exhibited excellent optical and structural features suitable for downstream applications, including bioimaging and catalysis. Future efforts will explore additional agricultural waste materials from the WP1 supply chain and assess their integration into WP3 applications, focusing on sustainability, scalability, and end-use functionality.

7. Annex

Characterization Summary of CDs

Biomass Source	Synthesis Method	Average Particle Size (nm)	Yield (mg / 3 g biomass)	Zeta Potential (mV)	Notable Features
Tea leaves	Acetic acid + thermal	3–5	n.a.	–	Mild synthesis, model precursor
Laurus nobilis	Microwave-assisted	3–5	~200	-15 ± 2	High yield, good reproducibility
Artichoke outer leaves	Microwave-assisted	3–5	~50	–	Low yield, lignocellulosic matrix
Hemp stalks	Microwave-assisted	3–5	~50	–	Low yield, good dispersion