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Green Synthesis of Fluorescent Carbon Dots from Sweet Basil (Ocimum basilicum) Leaves via Hydrothermal Method

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Abstract

Carbon-based materials play significant roles in the development of material science. Carbon dots (CDs), a new rising star in the carbon family. Carbon dots (C-dots) have gained more attention among researchers due to their outstanding fluorescent property which can be tuned based on several factors like high photostability, excellent water solubility, tunable fluorescence and optical properties, high quantum yield (QY), low toxicity, good biocompatibility, and environmental friendliness. One among them is based on a carbon source, so scientists have widely investigated several synthetic and natural materials to produce C-dots for diverse applications. Among these approaches, hydrothermal/carbonization treatment is frequently applied for the preparation CDs because of the outstanding advantages, such as high yield, simple manipulation, easy control, uniform products, lower air pollution, low energy consumption and so on. In this protocol we intend to use the sweet basil, scientifically known as *Ocimum basilicum* leaves as a precursor to produce C-dots using water as a solvent by adopting the hydrothermal methodology. The green synthesis methods by means of green precursors of synthesis involves the usage of inexpensive or recycled materials, while the chemical synthesis methods involve toxic chemical reagents or organic solvents as precursors. The synthesized C-dots have been confirmed by their fluorescent image and UV-Visible spectrometer.

Materials

List of equipment's required

- 1. Magnetic stirrer
- 2. Heating oven
- 3. Sonicator
- 4. Centrifuge
- 5. UV light source
- 6. UV-Visble spectroscopy
- 7. Pestle & mortar
- 8. Sterile syringe filters
- 9. Quartz cuvette
- 10. Polytetrafluoroethylene (PTFE) lined stainless steel autoclave reactor

Step 1: Preparation of basil leaves

1 Freshly collected Sample should be sorted first to ensure only healthy leaves are chosen for the C-dots synthesis.



Figure 1. *Ocimum basilicum* (Sweet Basil), depicting the plant at the developmental stage appropriate for the collection of fresh leaves.

- 2 Then it is washed thoroughly and rinsed with distilled water to remove the debris, dust or any other unwanted substance attached on to the leaf surface.
- 3 Excess water is decanted.
- 4 The leaves are allowed to dry at $60 \circ C \pm 5 \circ C$ for a duration of 1d 0h 1m $24:00:00 \pm 2:00:00$ in a heating oven with a temperature increment of $10 \circ C \pm 2 \circ C$ / $10 \circ C \pm 2 \circ C$ / $10 \circ C \pm 2 \circ C$ / $10 \circ C \pm 2 \circ C$ /



Figure 2. Dried Ocimum basilicum (Sweet Basil) Leaves Post Oven-Heating.

Note

To ensure homogeneous drying process it is recommended to separate the leaves and placed on a tissue paper.

5

Dried leaves are crushed to fine powder using pestle and mortar.

Note

This process plays an important role in the optical property of C-dots.

6 The resulting powder can be stored at $4 \circ C \pm 1 \circ C$ for less than a week.

Note

More than week time is NOT recommended for the synthesis.

A



Step 3: Synthesis process

9 As this methodology uses hydrothermal process, a non-corrosive A Polytetrafluoroethylene (PTFE) lined stainless steel autoclave reactor must be used.



Figure 3. Detailed Structure of a Non-corrosive Polytetrafluoroethylene (PTFE) Lined Stainless Steel Autoclave Reactor.

Note

PTFE chamber must be cleaned before the synthesis process.

- 10 The characteristic of the reactor it should withstand a temperature of $250 \circ C \pm 30 \circ C$ and an applicable pressure is $3.0 \pm 1.0 \text{ MPa}$.
- 11 Depends on the synthesis volume the reactor should be filled only 60-70 % capacity.
- 12 Then the precursor solution is added to the reactor completely.
- 13 Finally, the reactor should be tightened to its maximum limit.

Safety information

To avoid any accidents or breakage of the reactor, it is necessary the whole process is carried out under the supervision of experienced personal.

А

14 The whole setup is placed inside the oven which is pre-heated to $\begin{bmatrix} 180 & 0 \\ 2 & 5 \end{bmatrix}$ for 10h a period of () 10:00:00 \pm 00:30:00 without any external disturbances.



Figure 4. Oven with the complete setup, pre-heated to 180 °C ± 5 °C, shown here during a strictly controlled experiment conducted over a period of 10 hours ± 30 minutes.

15 After the process, the reactor is allowed to be cooled down naturally inside the oven.

Step 4: C-dots purification

- 16 After completing the step 3, the resulting black turbid solution is transferred to the centrifuge tubes and C-dots are collected.
- 17 The solution centrifugation is purified by with **4000** rpm for 30m O0:30:00 ± 00:05:00 .
- 18 Discard the pellet which is burnt residue of leaf powder, and the brownish solution is collected without disturbing the pellet at the bottom.

Δ

- 19 Later, to obtain pure C-dots the solution is centrifuged at 13000 rpm for 10m $\textcircled{1}00:10:00 \pm 00:02:00$ at $\textcircled{1}8 \circ C \pm 1 \circ C$.
- 20 The solution collected from (step 4-19) is passed through 0.22 μm pore size nylon sterile syringe filters.
- 21 The final solution is preserved at $4 \circ C \pm 1 \circ C$ for further use.

Note

Solution is stable for 8-10 months without contamination.

Step 5: C-dots formation confirmation

22 Qualitative testing 1 - The formation can be checked primarily by exposing the solution under the UV light to visualize the blue fluorescence.

Note

Dilution factor is proportional to the fluorescence intensity but not applicable for all C-dots.

23 The fluorescence of C-dots is shown in Figure 5 at 1:3 dilution.



Figure 5. Fluorescence under UV light: the left cuvette shows a non-fluorescent water control, while the right cuvette displays bright fluorescence of carbon dots (C-dots) at a 1:3 dilution.

- 24 Qualitative testing 2 The C-dot solution can be diluted for example $\boxed{_ 500 \ \mu L}$ with $\boxed{_ 3 \ m L}$ of water in a quartz cuvette and measured in UV-Visible spectroscopy without any background noise.
- 25 The UV-visible spectroscopy data is shown is Figure 6.



Figure 6. UV-visible spectra of synthesized C-dots.

Application of C-dots:

26 The synthesized can be used for wide range of applications and are broadly classified into:

1. Biomedical Applications

- a. Bioimaging
- b. Drug delivery
- c. Photodynamic therapy

2. Optoelectronic Applications

- a. Light-emitting diodes (LEDs)
- b. Photodetectors
- c. Photovoltaic devices

3. Sensing Applications

- a. Biosensors
- b. Chemosensors
- c. Environmental monitoring

4. Security and Anti-Counterfeiting

- a. Security inks
- b. Security labels
- c. Anti-counterfeiting coatings

5. Agricultural and Environmental Applications

- a. Plant growth promotion
- b. Crop yield enhancement
- c. Soil remediation

Protocol references

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