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Critical Analysis of Microwave Heating Mechanisms in Carbon Quantum Dot Synthesis

Srijita Basumallick*

Department of Chemistry, Asutosh College under Calcutta University, 92, SP Mukherjee Road, Kolkata-700026, India.
*Corresponding Author (Srijita Basumallick) Email: srijita.basumallick@asutoshcollege.in**

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ABSTRACT

The unique characteristics of carbon quantum dots (CQDs), including their high water solubility, outstanding photostability, programmable photoluminescence, and non-toxicity, have made them a very promising family of nanomaterials. Under the bottom-up technique, microwave-assisted synthesis has drawn a lot of interest among different synthesis methodologies since it is quick, more greener and energy-efficient. This method produces CQDs with improved quantum yield, tunable emission wavelength and a greater yield and purity by allowing for homogeneous heating, shorter reaction periods, fewer side reactions. The size, shape, surface functionalization, and emission properties of the CQDs are all significantly influenced by the precursor selection, especially with regard to amino acids and carbohydrates. The mechanism of microwave heating are highlighted such as dipole and interfacial polarization as well as resistive or conduction heating as well as mechanism of microwave losses are discussed such as dielectric, conduction, and magnetic losses. The study also examines the benefits and drawbacks of alternative techniques, including hydrothermal, pyrolysis, combustion, and template approaches, in comparison to microwave synthesis. The impact of precursor chemistry and reaction parameters on the level of carbonization and surface characteristics is also covered. Despite obstacles including uneven heating and scalability problems, microwave-assisted synthesis provides an environmentally friendly, effective, and adaptable path to CQDs that can be used for a variety of bioimaging, sensing, and optoelectronic applications.

Keywords: Carbon Quantum Dots; Microwave-assisted Synthesis; Green Chemistry; Photoluminescence; Bottom-up Approach; Carbohydrates; Amino Acids; Quantum Yield; Surface Functionalization; Reaction Mechanism; Bioimaging; Nanomaterials.

1. Introduction

CQDS have recently attracted lots of attention [1]. This is because, compared to any other quantum dots, CQDS are non-toxic, have good water solubility, have high photostability, and show photoresponse. It also allows facile surface functionalization and tunable excitation emission [2]. There are several other carbon nanomaterials like graphene, carbon nanotubes, etc., but they suffer from poor water solubility [3] and weak visible fluorescence. Talking about the synthesis strategy of CQDS, there are mainly two types of synthesis processes followed to date. Top-down approach where larger carbon resources such as carbon nanotubes, fullerene, graphite, graphene, carbon soot, activated carbon, etc., are broken down into small CQDS. For this laser ablation method, electrochemical methods and arc discharge methods are used. The major bottleneck for this method is that pure nanomaterials are difficult to obtain, as larger carbon precursors are difficult to purify. Also, the morphology and size distribution of these types of CQDS are not well monitored. On the other hand bottom-up approach where smaller carbon sources like amino acids, carbohydrates, and organic small molecules can be combined to form CQDS with the help of hydrothermal or solvothermal methods, combustion, pyrolysis, and microwave irradiation. Compared to the top-down approach, here particle size can be tuned by tuning temperature, pressure, and reaction time together with the nature of the solvent used. Usually chemical precursors used here are polymer like starch, amino acids and glucose, sucrose, citric acid, lactic acid, ascorbic acid, glycerol, etc. [4-7]. Seeds [8] and leaves are also used as natural sources to prepare carbon dots by this method. Among all these approaches microwave assisted carbon quantum dot synthesis is much easier because it has rapid reaction time it takes only seconds or minutes to complete the whole reaction which is much faster than conventional hydrothermal or solve a thermal method. It minimises energy loss as the vessel remains cool and only the solvent or the reactant heats up itself. Uniform and controlled heating minimises its side reactions resulting in higher yield of CQDS with less impurities. Also the size and the surface are controlled by changing exposure time, power of microwave as well as precursors used. This can change fluorescence property of carbon quantum dot used. Of course microwave synthesis can be carried out without solvent and thus it is best for doing green chemistry. Microwave synthesis is easily scalable as hazardous solvents and vessels heating are not associated with these types of reactions. Interestingly microwave assisted synthesis of carbon quantum dot has increased defects and surface functional groups that can enhance quantum yield and photo-luminescence of the particles. But microwave induces non-uniform heating or microwave field create hotspots which leads to formation of inconsistent particle size and surface properties. Microwave also has limited penetration depth especially in dense and highly absorbing materials. This restricts scalability without sophisticated equipments. Microwave reactors with controlled temperature pressure and power are expensive and complex to operate. It is seen that reproducibility of microwave assisted products are compromised. Also undesired functional groups are formed. Improper shielding of microwave system can cause thermal or radiation hazards especially in high power systems. There are several review articles elaborately discussed about microwave assisted synthesis of carbon quantum dot which are worth reading [9-11].

1.1. Study Objectives

The specific objectives of this study are as follows:

(1) To elucidate the fundamental mechanism of microwave-assisted CQD synthesis. (2) To compare microwave synthesis with other conventional bottom-up techniques such as hydrothermal, combustion, pyrolysis, and template methods. (3) To analyze the effects of precursor type and reaction parameters on the morphology and photoluminescence of CQDs. (4) To describe the mechanisms of microwave heating and associated energy losses. (5) To highlight challenges such as non-uniform heating, scalability, and reproducibility. (6) To identify potential applications of CQDs in bioimaging, sensing, and optoelectronics.

2. Discussion

As discussed in the introduction among two processes 1. Top down and 2. Bottom up approach of carbon quantum dot preparation. Bottom up approach is better and cleaner. There are several methods used to prepare carbon quantum dot under bottom up methods.

2.1. Hydrothermal method

Particularly speaking, hydrothermal synthesis is usually done in a Teflon-lined stainless steel autoclave heated in a hydrothermal chamber at high temperature and pressure for a few hours. Here, water is used as a solvent. There are several early works on hydrothermal methods of preparation of CQDS [12]. The hydrothermal method for carbon quantum dot synthesis was first reported by Zhang et al. [13]. They observed high quantum (around 7%) and blue fluorescent CQDS when ascorbic acid was used as a precursor. They have used several solvents, and they found that these CQDS were very stable at room temperature for a long time (about six months), and the fluorescence was stable at a wide range of pH and in high ionic strength conditions. Usually protein, polymers, and polyols like glucose are used as precursors in the hydrothermal method. Advantages of the hydrothermal method include a single-step synthesis, low cost, and an eco-friendly or green chemistry approach with non-toxic byproducts. The quantum dots prepared under hydrothermal treatment are highly homogeneous, water-soluble, monodispersed, photostable, and salt-tolerant with high quantum yield without any surface passivation.

Also, in this method, particle size can be controlled by changing pressure, temperature, and time of reaction. A similar terminology, the solvothermal method, is also used where, instead of water, ammonia, alcohol, and other organic and inorganic solvents are used.

2.2. Combustion method

This was first reported by Lue in 2007 [14]. The combustion method involves oxidative acid treatment of partially combusted carbon materials. This helps in the aggregation of smaller carbon units into CQDS, improves solubility, and controls fluorescent properties.

Typically Lue et al. [14] used candle ashes, which were refluxed into nitric acid in an oxidative treatment step. Followed by dissolution in a neutral medium and purification through centrifugation and dialysis. Through the combustion method, the quantum field obtained was low, but without doping, it shows very good fluorescence.

2.3. Pyrolysis method

The next process used to prepare CQDS in the bottom-up approach is the pyrolysis method, which involves thermal decomposition of precursors at elevated temperatures (typically greater than 430°C) and under high pressure in the absence of oxygen. The presence of alkali and strong acids acts as a catalyst, promoting cleavage of precursors into nanoscale colloidal particles. An advantage of the paralysis method is that it is capable of producing CQDS with high quantum yield. Also, the process is practical, repeatable, and simple.

A major bottleneck is that it is difficult to separate small precursors from raw materials. Several precursors are used. Lue et al. [15] used resol resin as carbon source and surfactant-modified silica spheres in 2009, and Pan et al. [16] used EDTA in 2010, Martindale et al. used citric acid in 2015, and Rong et al. [17] prepared N-doped CQDS using citric acid and guanidinium chloride in 2017.

2.4. Template method

Another very important process for making CQDS is a template method. The template method has two main steps. 1. First preparation of a template where CQDS are synthesized. Followed by CQDS preparation within this template (mainly silicon spheres) via calcination. 2. The second step is the itching step, where template material is removed through itching and the carbon quantum dot is isolated. This has been shown in the Figure 1 below.

The major advantages of the template method are that it enables surface passivation of CQDS, it prevents particle agglomeration, and particle size can efficiently be controlled. But the disadvantages are the purity of the carbon quantum dot. Also, in template method the fluorescence property and quantum yield are low.

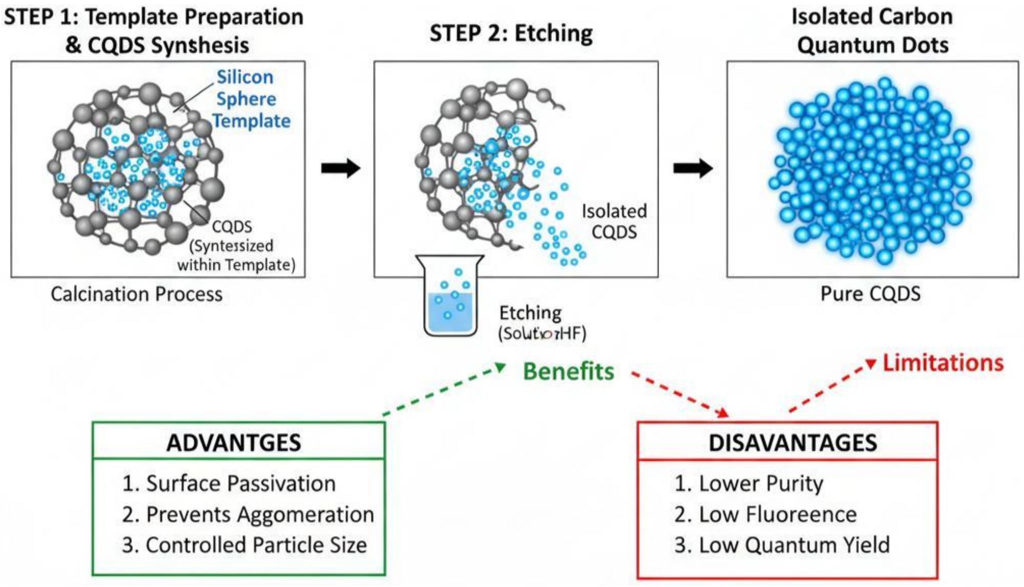

Figure 1. Schematic illustration of the two-step template method for synthesizing Carbon Quantum Dots (CQDs). Step 1: Template Preparation & CQDs Synthesis involves the calcination process where CQDs are synthesized within a Silicon Sphere Template. Step 2: Etching uses a solution (e.g., HF) to dissolve the template, releasing the Isolated CQDs and resulting in Pure CQDS.

2.5. Microwave method

The next very important process for making CQDS is microwave-assisted formation of CQDS. It is a fast, cost-effective, and energy-efficient method for synthesizing CQDS. Zhu et al. [18] were the first to synthesize fluorescent CQDS using microwave irradiation at 500 W. He used reactants such as polyethylene glycol (PEG 200) and saccharide. He took 2 to 3 minutes to complete the reaction.

2.5.1. Advantages associated with microwave method: There are several advantages associated with microwave-assisted reactions, like direct and uniform heating, reduced reaction temperature, and minimum overheating risk. In direct heating method using microwave irradiation, the polar molecules directly heat up without heating the vessel. As a result, the reactants are not exposed to prolonged heat, which is usual in common reactions during the cooling phase. Thus there is less byproduct. Microwave-assisted reactions are not associated with temperature gradient and thus minimize by-products as well. Microwave can be stopped instantaneously; thus, overheating risk can be neglected. Microwave heating takes a shorter reaction time as it does not need equilibration of heat through convection or conduction.

2.5.2. Disadvantages associated with microwave method: The major bottleneck associated with the microwave irradiation process is non-uniform heating and non-uniform size distribution.

2.5.3. Types of interactions between microwave and reactant: Microwaves interact with materials via 1. Reflection, 2. Refraction, and 3. Absorption. Reflection means microwave bounce off the surface of target materials. Refraction means microwaves bend as they pass through media with different refractive indices. Finally, absorption means where energy is directly absorbed and converted into heat. The last one is very important for carbon quantum dot formation.

2.5.3.1. Microwave irradiation loss: While irradiating reactants with microwaves, there is a significant amount of energy loss or microwave radiation loss. So it is important to know the mechanisms of microwave irradiation losses during a reaction. There are several processes through which microwave energy can be lost during the synthesis of CQDS. 1. The first dielectric loss. This is caused by dipole build due to interfacial polarization. Here, energy gets lost in the form of heat as this dipole tends to rotate under microwave irradiation. 2. The second one is conduction loss. Here, free electrons in conducting materials cause resistive heating when moved by a microwave electric field. 3. Third is magnetic loss, which includes hysteresis and ED current losses in magnetic or semi-magnetic materials. Let’s now elaborate on the mechanisms of microwave energy loss one by one.

2.5.3.2. Dielectric loss: Interface polarization, also called Maxwell-Wagner-Sillars polarization, occurs in heterogeneous materials, especially those found in composite or porous structures or compounds having grain boundaries. In a material with regions of different dielectric properties, the electric field does not interact homogeneously across the material. So when microwaves are applied, ions or electrons move more easily in one phase and less easily in the next phase, having lower conductivity. As a result, moving charges under irradiation accumulate at the boundaries between these types of heterogeneous phases. As mentioned the reason is very easy, charges cannot pass through the low conductivity region, and the charge starts accumulating on one side of the phase boundary. This results in the formation of localized dipoles. The accumulated charge at the interface creates a net dipole moment across the boundary; as a result, overall dielectric polarization of the material is altered. This leads to enhancement of microwave adsorption and localized heating, especially in heterogeneous materials.

2.5.3.3. Conduction loss: Next is resistive or conduction heating, where tiny charged particles like electrons and ions move through a material due to the influence of an electric field, such as microwaves. During their movement, electrons and ions face resistance inside the material. This creates resistive heating, and the material gets warmer. Finally, microwave radiation energy is dissipated as heat energy through resistive heating.

2.5.3.4. Magnetic loss: In magnetic loss, the magnetic material, when exposed to an alternating magnetic field like a microwave, has magnetic domains, or tiny regions where magnetic moments are aligned try to realign with the rapidly changing field. There is a lag generated, and this lag is called hysteresis. Each cycle of this lag or delay consumes energy, and that energy is released as heat. This is significant only in materials with strong magnetic properties and at lower frequencies. This is called hysteresis loss. This type of energy loss is associated with eddy current losses. Microwave-induced circulating electric current in conductive or semiconductive magnetic materials causes eddy current loss. When circulating electrons from eddy currents face resistance causes resistive heating. This type of loss increases with higher electric conductivity, thicker material, and high frequency.

2.5.4. Microwave heating mechanism: After the energy loss mechanism, now we shall talk about microwave heating mechanisms. There are several mechanisms through which microwave heating converts electromagnetic energy into thermal energy. 1. The first one is dipole polarization, where molecules with permanent dipoles are rotated with the alternating field, causing friction and heating. 2. The second one is interfacial polarization, where charge accumulates at the interface, leading to heating. 3. Third is resistive or conduction heating, where movement of charge carriers causes heat through internal friction. 4. The fourth one is hysteresis and eddy current occurring in magnetic material, leading to localized heating through magnetic energy dissipation.

2.5.5. Mechanism of carbon quantum dot formation: Though not much evidence is seen to illustrate the mechanism of carbon dot formation, the major steps followed are polymerization, carbonization, and carbon dot formation. Carbon dots thus formed can be of three types. 1. Hydrophilic with COOH, OH, or NH_2 groups on the surface. 2. Hydrophobic and 3. Amphiphilic CQDS. Hydrophilic CQDS are easy to prepare because they are made in water. Wang et al. [19] formed water-soluble CQDS from carbohydrates with -OH and -COOH as surface functionalization. But phosphate and other cation and anion concentrations helped increase the fluorescence quantum yield of such particles. Synthesis of carbon quantum dots and their origin of fluorescence property has been illustrated in several review articles [20,21].

2.5.5.1. Mechanism of carbon quantum dot formation with starch as precursor: Usually starch or glucose is taken as a carbon source, and phosphoric acid is also used. Steps followed in this process for the formation of CQDS are degradation via dehydration to form furfural intermediates from glucose and saccharides. Furfural intermediates polymerize into aromatic polymers, which condense into carbonaceous materials. Phosphoric acid acts as a chemical activator and facilitates the formation of oxygen-containing polar functional groups like the carboxylic acid group. Also, phosphoric acid, being low volatile, remains in solution at high microwave irradiation temperatures above 300 °C. Finally, it maintains an acidic environment, which is essential for carbon quantum dot synthesis. So overall this process follows hydrolysis of starch, dehydration of the monomer that is glucose, polymerization and aromatization of the furfural molecule, nucleation and growth of the carbon-rich structure, and finally carbonization into CQDS. Very importantly in this process, the surface functional groups play an important role in fluorescence. CQDS emit blue fluorescence at around 435 nm. Fluorescence arises from surface states associated with oxygen-containing functional groups. It is very important to note that shorter microwave irradiation leads to aggregation and decreased fluorescence quantum yield. This also broadens the absorption band and changes the fluorescence color from blue to green or orange. These aggregated CQDS tend to be hydrophobic, and they can easily be extracted into an organic layer. Carbon dots that remain in the aqueous phase are hydrophilic. It is obvious that while these CQDS become hydrophobic, the surface functional groups condense or disappear. In contrary hydrophilicity increases with more surface functional groups. Prolonged microwave radiation that is greater than 10 minutes leads to evaporation of water, resulting in concentrated phosphoric acid. This concentrated phosphoric acid improve surface functional groups enhance hydrophilicity and solubility.

2.5.5.2. Carbon quantum dot formation with amino acid as precursor: Kolanowska et al. [22] have worked on synthesis of carbon quantum dot from amino acids. Though basic steps are similar to synthesis of carbon quantum dot from other precursor and it follows assembly of amino acids via hydrogen bonding. Initially, amino acids self-assemble through hydrogen bonds during the hydrothermal process then undergo dehydration & polymerization, formation of a carbon skeleton, growth and nucleation and surface functionalization. But when amino acid is used as precursor distinct features are observed. Degree of carbonization, and surface functionalization of thus formed CQDS are greatly influenced by amino acids used because of their unique chemical structures and functional groups. Carbon quantum dot derived from phenylalanine have a narrow size distribution (~1–5 nm), primarily having spherical shape. Whereas other amino acids show range of size as well as variety of shapes. Functional groups incorporated are precursor-specific like sulfur-containing groups are introduced by cysteine, whereas amino groups are introduced by lysine. CQDS made from different amino acids exhibit emission at different wavelengths as well as have variable quantum yields. Precursors rich in aromatic structures (like phenylalanine) tend to produce more graphitized (more crystalline) CQDS, whereas aliphatic amino acids lead to more amorphous structures.

3. Conclusion

In conclusion microwave assisted CQDS formation is more greener and energy efficient method. But detailed mechanism of CQDS under microwave needs to be explored. The equipment needs to be more sophisticated to rule out hot spot formation and non-uniform CQDS formation. Microwave-assisted synthesis is highlighted as a promising bottom-up strategy for CQD preparation due to its rapidity, energy efficiency, and alignment with green chemistry principles. This technique offers advantages over conventional methods by providing uniform heating, shorter reaction times, and reduced side reactions, which results in higher yields and better control over particle size and surface functionalities. While it allows for flexibility in precursor selection (like amino acids and carbohydrates) to tailor properties for specific applications, challenges such as non-uniform heating (hot spots) and limited penetration depth persist, leading to issues with polydispersity and inconsistent quantum yields. Addressing the current bottlenecks, particularly the lack of detailed mechanistic understanding and scalability issues requiring advanced reactor designs, is crucial for realizing the industrial and diverse technological potential of microwave-synthesized CQDs. Future research should focus on advanced reactor technology, computational modeling, and exploring the incorporation of dopants to further tune optical properties and enhance quantum yield for applications in bioimaging, sensing, and optoelectronic devices.

4. Future Suggestions

1) Development of continuous-flow and large-scale microwave reactors needed to be explored.

2) The major bottleneck associated with every microwave synthesis that is non-uniform heating or hotspot spot formation. Optimization of uniform heating strategies to eliminate hot spots.

3) Research on incorporation of dopants and heteroatoms to improve quantum yield needs to be done.

4) Use of computational modeling to study carbonization kinetics.

5) Integration of green, solvent-free precursors for sustainable synthesis.

6) Exploration of CQD applications in targeted bioimaging, catalysis, and sensing.

Declarations

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Competing Interests Statement

The author declares that she has no competing interests related to this work.

Consent for publication

The author declares that she consented to the publication of this study.

Authors' contributions

Author’s independent contribution.

Availability of data and materials

Supplementary information is available from the author upon reasonable request.

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Not applicable for this study.

Informed Consent

Not applicable for this study.

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