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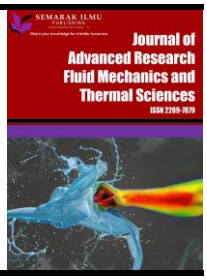

Hydrothermal Synthesis of Carbon Quantum Dots: An Updated Review

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ABSTRACT

Carbon Quantum Dots (CQDs) have exceptionally solid and tuneable fluorescence properties which empower their application in vast fields. The hydrothermal approach is regarded as direct and efficient, through polymerization and carbonization reactions, and has been widely applied to prepare various materials due to the high reactivity of the reactants, easy control of the solution, little harm to the environment and low energy consumption under hydrothermal condition. The attracting feature of this route is that neither any strong acid nor post-synthetic surface passivation is necessary. As of now, a lot of important progress in the hydrothermal synthesis of carbon quantum dots such as materials use as precursors and influence of hydrothermal synthesis parameters. Hydrothermal synthesis is one of the most commonly used methods for preparation of nanomaterials. It is basically a solution reaction-based approach. In this review, the main focus is to review the hydrothermal route for carbon quantum dots using different source of materials and their fundamental characterizations, followed by the influence of hydrothermal synthesis conditions to carbon quantum dots.

1. Introduction

Carbon quantum dots (CQDs)are also known as carbon nanoparticles (CNPs) are a new type of carbon nanomaterial, mainly with a diameter of fewer than 10 nanometres in combination with numerous functional groups/polymer chains [1-3]. Carbon quantum dots are categorized based on their carbon core configuration, surface classes, and properties [4]. The structure of CQDs is made up of a vast number of surface groups/polymer strings, such as carboxyl, hydroxyl, and amine; hence CQDs have exceptional water solubility and are simple to combine with other products without phase separation [5,6]. This fluorescent carbon nanoparticle has received a lot of attention as possible competitor and alternatives for semiconductor quantum dots (SQDs) because SQDs have some drawbacks, such as high toxicity due to heavy metals during their manufacturing. The metal content in these traditional quantum dots can be extremely harmful, even at low concentrations [7]. Thus, CQDs have been developed to substitute the conventional SQDs because of their various advantages: low toxicity [8], biocompatibility [9], low expense [10], and chemical inertness [11].

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Most of the prior and current researches focused on simple, cost-effective, large-scale methods for synthesizing CQDs with advanced functions and various compositions and structures [12,13]. The two main CQDs synthesizing protocols include top-down and bottom-up approaches [14,15]. In the top-down approach, CQDs are extracted from various macroscopic structures of carbon sources, such as graphite, activated carbon, and carbon nanotubes. The extraction methods, such as arc-discharge, laser ablation, and ultrasonic synthesis, are employed in the top-down method [16]. Meanwhile, bottom-up approaches are manufactured from molecular precursors, including citric acid, refined sugar, and glucose, through various methods of microwave exposure [17], thermal decomposition [18], hydrothermal treatment [19], template-based routes [20], and fluid treatment [21]. Among these methods, the hydrothermal method is considered a distinct alternative method in the manufacture of nanomaterials, as it is a low-temperature, low-cost, and environmentally friendly process [22-24]. This production method also has excellent control over the product size, structure, and morphology of the product. Hydrothermal method also used to make nanoparticles and other light-emitting materials [25,26].

Currently, one of the most fundamental and significant problems of CQDs development is the lack of a standardized and efficient synthesis procedure to generate high-quality CQDs with desired structures (e.g. correct size, shape, crystallinity, and amount of functional groups) [27]. The exact reaction process, nucleation mechanism, and growth step are still uncertain, impacted by the non-standard synthetic pathways and particle creation [28]. Therefore, to create an effective pathway for large-scale processing of high performing CQDs, the influences of precursors and reaction conditions (e.g. temperature, time, pH and so on) on the efficiency of CQDs should be thoroughly investigated [29,30]. This review is very important to summarize the fabrication of successful hydrothermal synthetic routes for CQDs and the influence of the synthesize parameters towards the CQDs.

2. Carbon Quantum Dots Precursors Synthesis by Hydrothermal Route

Various synthetic and natural carbonaceous substances have been used as starting materials for carbon point synthesis. Conventional bottom-up and top-down methods for synthesis led to complicated economic and environmental problems due to the need for large amounts of toxic solvents, hazardous organic molecules, expensive feedstock, and a lot of energy [31]. Therefore, considerable efforts have been made to develop green synthetic routes with less harmful starting materials. Typically, CQDs are synthesized from organic (chemical) molecules [32], natural sources [33] and biomass wastes [34]. The hydrothermal synthesis method is used by most researchers as it is a cheap, environmentally friendly and cost-effective method to synthesize CQDs from various sources, e.g. saccharides, amines, organic acids, and their derivatives. From previous studies, citric acid is one of the most commonly used organic molecules for the preparation of CQDs because it consists of carbonyl, carboxyl, and hydroxyl groups [35]. Carbon quantum dots functionalized with polyamines were hydrothermally synthesized by carbonization of citric acid with branched polyethyleneimine (BPEI) [36] and were measured with a quantum yield (QY) of 42.5%. In another study, CQDs were synthesized from citric acid and poly(ethyleneimine) (PEI) by heating the aqueous solution of citric acid and PEI at 110°C for 2 h, with a QY of 48.3% calculated [37]. These studies proved that it is possible to use small molecules as carbon precursors. In addition, CQDs synthesized from phenol derivatives showed photoluminescence quantum yields as high as 24.4% in water and 53.3% in ethanol [38]. Highly efficient orange fluorescent nitrogen-doped carbon dots (N-CDs) were prepared easily and simply from P-phenylenediamine as a precursor via a hydrothermal method [39]. The obtained QY was 11.5%. The tricolour emitting CQDs were synthesized by a one-pot

hydrothermal method and by using 5-amino-1,10-phenanthroline (Aphen) and citric acid (CA) as the precursors [40]. The QY of the synthesized CQD also reached a remarkable value of 52%.

Plant parts are natural resources and hence are considered environmentally friendly materials, as compared to organic molecules [41]. The utilization of plant parts as materials has several advantages. For example, they are cheap, easy to obtain, safe, abundant, and renewable. Plant parts, such as roots, stems, leaves, fruits, flowers, and seeds, have been used for the synthesis of green CQDs [42]. Moreover, the synthesis of CQDs from plant parts can transform various low-grade materials into functional materials with high biocompatibility. Plant parts containing various heteroatoms, such as nitrogen (N) and sulfur (S), are the most suitable starting materials for CQDs, as compared to other carbon sources that require additional heteroatoms [43]. Monodisperse N-CQDs with a quantum yield of about 13.6% were prepared hydrothermally for 3 hours at 180°C by employing rose-heart radish as a carbon source [44]. In other studies, hydrothermal carbonization of apple fruit at 150°C yielded C-points with surface functional groups, such as hydroxylamine, keto, and a carboxylic acid, with a QY of 4.27% [45]. Prunus avium fruit extract was investigated for non-metallic (N) doped CQDs to improve the inherent low emission efficiency, using aqueous ammonia as nitrogen dopant to obtain a QY of 13% [46].

Biomass waste management is a major challenge that needs to be addressed as a result of expensive population growth and ever-increasing demands for horticultural products [47]. Nowadays, attention has been shifted from traditional biomass to waste biomass for the production of value-added products carried out under mild experimental conditions. Waste biomass represents a sustainable and cheap source of raw materials, also considered an effective and potential alternative feedstock for carbon point production [48]. For example, water-soluble CQDs with a quantum yield of 6.9% are synthesized and developed from pomelo peels for the selective and sensitive determination of Hg^{2+} by a hydrothermal method [49]. Tea waste is also used as a starting material for CQDs. Moreover, highly florescent CQDs were synthesized from coconut shells by a single step of hydrothermal carbonization, and these dots were measured with a QY of 35.7% [50]. An overview of the hydrothermal synthesis of carbon quantum dots with different precursors has been summarized in Table 1.

Table 1
Different source use for hydrothermal synthesis of CQDs

Source	Precursor	Quantum Yield (%)	References
Organic molecules	Fructose and hydrochloric acid	6.8	[51]
	P-phenylenediamine	11.5	[52]
	Phenol derivatives and ethylenediamine	5.8,20. , 24.4	[53]
	(3-aminopropyl) triethoxysilane (APTS)	5.3	[53]
	Sodium sulfide+ Citric acid	21.1	[54]
Natural	Seville orange	13.3	[55]
	Cherry tomatoes	9.7	[56]
	Gardenia fruit	10.7	[57]
	Orange juice	31.7	[58]
	Miscanthus grass	11.6	[59]
	Mint leaf	7.64	[60]
	Oyster mushroom	12.51	[61]
	Tobacco leaves	27.9	[62]
	Pseudo-stem banana	48	[63]
	Water hyacinth	27	[64]
	Red lentils	13.2	[65]
Biomass waste	Wheat bran	33.23	[66]
	Food waste	28, 18, 10, 6	[67]
	Rice residue	23.48	[68]
	Lemon peel	14	[69]
	Prawn shell	9	[70]
	Peanut shells	9.91	[71]
	Waste tea	7.1	[72]
	Oil palm empty fruit bunch	24.6	[73]
	Sweet potato peel	8.9	[74]
	Snake gourd peel	28.6	[75]
	Grape skin	18.67	[76]
	Durian Peel	11	[77]
	Ananas comosus waste peels	10.65	[78]
	Date kernel	12.5	[79]

3. Characterization of Carbon Quantum Dots Synthesize by Hydrothermal Route

3.1 Morphological Analysis

Transmission Electron Microscopy (TEM) and Scanning Electron Microscopy (SEM) can be used to analyze the shape, size distribution, and particle size of carbon dots, as well as to determine whether the particles produced are agglomerated or dispersed. Normally, SEM is used to analyze particle sizes of between 1 to 20 nm [80]. However, if the measurement exceeds the resolution of SEM, then TEM that has a higher resolution is recommended. Nowadays, high-resolution transmission electron microscopy (HRTEM) is widely used to analyze the structure and crystalline nature of materials [81]. In other words, HRTEM can be used to characterize the internal structure of CQDs. Currently, most of the CQDs produced by hydrothermal approaches are uniformly distributed and typically have a spherical structure in aqueous fluids with a particle size or diameter of 10 nm [82]. As such, HRTEM detection of CQDs allows us to gain more information about their structure, such as the spacing of the lattice fringes. Despite the fact that the size of the CQDs is still less than 10 nm, the lattice spacing has not been clearly detected in some previous studies [83].

3.2 Zeta potential analysis

The zeta potential, also known as the electro kinetic potential, was measured by using a zetasizer in order to determine the surface charge of the synthesized luminescent CQDs [84]. This method can reveal a double layer of CQD with numerous hydrophilic functional groups (namely hydroxyl, carboxyl and carbonyl groups). The negative potential is caused by a dense electron cloud colliding with the CQDs [85]. The value of zeta-potential measured depends on the short- or long-term stabilities of CQDs particles. Particles in nanodimensions with zeta potential values of between -10 and +10mV are considered neutral [86]. The CQDs hydrothermally synthesized with a high zeta potential (either positive or negative) are considered electrostatically stable [87], while the particles with a low zeta potential coagulate or aggregate within a short period of time. A low potential results in CQDs having low physical stability. If the number of CQDs is large, this indicates that the repulsive forces have exceeded the attractive forces, resulting in a relatively stable system.

3.3 Phase study using X-ray Diffraction (XRD)

The internal structure and crystallinity of materials are commonly studied through X-ray diffraction analysis [88]. The X-ray diffraction study is carried out to reveal the unit cell properties of crystalline carbon nuclei, including the presence of a distinct crystal lattice in the structure of the particles. Furthermore, X-ray diffraction analysis also reveals the crystalline structure of materials at the atomic level [89,90]. Each solid crystalline material has a unique X-ray diffraction pattern that can be used to determine the structure of a tested substance. Similarly, the study of X-ray diffraction is useful for determining the chemical combination level of an element. The information contained in a substance is essentially and mainly determined by its crystalline structure and not by other factors. The XRD analysis of CQDs synthesized by hydrothermal approach often shows that they are amorphous due to the arrangement of the carbon atoms. The CQDs often have a large diffraction peak in their XRD spectra between two Tetha values of 20° and 25°, respectively. While this technique is excellent for determining crystallite features, it can also be used for the investigation of amorphous CQDs.

3.4 The Fourier transform infrared (FTIR)

This approach is used to analyse the surface of CQDs, in addition, to determining the functional groups that determine the formation of new energy states and thus the nature of photoluminescence [91]. This approach is applicable to samples in various states, such as gaseous, liquid and solid states. However, the use of water as a solvent is not practical, mostly due to the large absorption peaks of water in the infrared region. The FTIR spectra of hydrothermal synthesized CQDs showed the presence of carboxyl, hydroxyl, epoxy and ester functional groups on the surface [92]. Additionally, FTIR spectroscopy allows a complete characterization of the functional groups on the sample surface, leading to a better knowledge of the mechanisms underlying PL in a specific wavelength range [93]. Biomass derived CQDs generally contains - OH, C-H, C=O, C=C and other chemical bonds, while C-N and C-S bonds may also be present. These chemical bonds can be identified from the peaks at 3427, 1720, 1620 and 1400 cm^{-1} in the infrared spectrum of CQDs, respectively.

3.5 Absorbance

Carbon quantum dots have a high absorption coefficient in the ultraviolet (UV) range (mostly from 280 to 360 nm) [94]. Electronic transitions are responsible for the absorption of light in the ultraviolet and visible regions of the optical spectrum (also known as UV/Vis absorption). CQDs created by a variety of methods have been observed to absorb UV light, with the location of the absorption peak varying depending on the parameters controlling particle formation. Fluorescent optical discs exhibit significant optical absorption in the ultraviolet region, with a long tail extending into the near-infrared region [95]. The n* transition, involving aromatic sp^2 carbons (aromatic C=C bonds), corresponds to short-wavelength bands of below 300 nm (band I). In contrast, the n* transition, involving aromatic carbon nuclei, is due to intrinsic absorption between 300 and 400 nm (band II) [96]. The absorption bands above 400 nm (bands III to V) are the results of a change in the surface state of a lone pair of electrons. Meanwhile, the red shift of the UV-Vis absorption spectrum from about 420 nm (band III) to the near-infrared region (band V) is directly related to the addition of graphitic nitrogen to the sp^2 carbon lattice, as the centers of graphitic nitrogen inject excess electrons into the unoccupied *-orbitals, significantly lowering the HOMO LUMO gap and covalent bond energies [96,97]. O-containing functional groups (for examples hydroxyl, carboxyl and epoxy) constrict the energy levels on the surface of CQDs, resulting in red-shifted absorption [97].

3.6 Photoluminescence (PL)

Studies related to optical properties of CQDs become very important to understand and reveal the origin of luminescence and further applications of CQDs. Many studies on optical properties of carbon dots, such as (down-conversion) photoluminescence, up-conversion photoluminescence, time-resolved photoluminescence, phosphorescence and multi-color luminescent, have been done to reveal the origin of luminescence of CQDs [98,99]. Photoluminescence (PL) is a physical process in which spontaneous emission of light from a material will occur under optical excitation [100]. PL investigations of a material will provide a lot of information related to material parameters. The PL emission spectrum can be used to identify surface, interface, and impurity levels in a material. The intensity of the PL signal provides information on the quality of surfaces and interfaces. By using pulsed excitation sources, the transient PL intensity gives knowledge about lifetime of non-equilibrium interface and bulk states.

4. Influence of Hydrothermal Synthesis Parameters on The Carbon Quantum Dots

4.1 Temperature

Based on the reaction mechanism and experimental conditions, the hydrothermal method can be divided into two main categories, namely the high-temperature (400°C to 800°C) and low-temperature (200°C to 300°C) hydrothermal processes [101]. The low-temperature hydrothermal process is environmentally friendly, in addition can synthesize numerous carbon materials with different morphology, size, shape, and surface functionality, and utilizes multiple chemical transformations, therefore making it suitable for the synthesis of CQDs [102]. Since hydrothermal carbonization is an endothermic process, the temperature is a key factor in the synthesis of CQDs. It was reported that for the synthesis of CQDs from biomass components (e.g. cellulose, hemicellulose, chitin, lignin), a temperature of above 180°C is required for the hydrolysis and carbonization of the biomass components [103]. However, at temperatures of above 300°C, the carbon source is over oxidized, and the surface structure of the CQDs is mostly destroyed, leading to the optical

performance degradation of CQDs. At a higher temperature, more non-radiative channels would be activated and more excited electrons would return to the ground state via a non-radiative mechanism, consequently leading to a decreased in fluorescence intensity [104,105]. This is because the energy transfers between the core and surface states of the CQDs are insufficient thus, lowering the reaction temperature was the reason for the observed shift from the blue to the red range in the emission of their CQDs.

4.2 Time

The exact residence time could not be determined because the reaction rates are largely unknown, but the residence times that ranged from 1 to 24 hours had been reported by the prior published works on this similar topic [106,107]. A longer residence time had been observed to result in higher reaction severity and lower organic loss in the sugar solution. For example, when HTC experiments were performed at a constant concentration of 0.5 mol/L and at 160°C, the diameters grew from 0.2 to 0.5, 0.8, 1.1, and 1.5 µm specifically when the residence times were increased from 2 to 4, 6, 8 and 10 h [107]. In the first two hours of synthesis, no solid residues were detected, while the glucose residues dried and disintegrated into small soluble organic molecules. After 4 hours, the solution turned dark orange, indicating polymerization and aromatization (polymerization step). After 5 hours, the first solid settled. After 8 hours, a brown colloidal dispersion was formed. After 12 hours, the black-brown substance formed spherical particles with a diameter of 0.5 µm. The output of hydrogen and oxygen (H=O) molecules increased for up to 10 minutes, but then dropped due to the decomposition of main hydrothermal products (H=O groups). However, when the reaction time was too long, the produced carbon spheres fused, and particles with irregular shapes were formed [108]. In another study, CQDs were synthesized hydrothermally from biomass cellulose with variable reaction times of between 2 to 12 hours [109]. The results showed that the photoluminescence decreased with increasing time, while the CQDs synthesized after 6 hours no longer had PL.

4.3 Concentration

One of the properties of CQDs is having concentration-dependent fluorescence due to multiple emitting centers. The close spacing between emitting centers on the particle surface of a highly concentrated CQD solution may be causing non-radiative recombination of charge carriers. In contrast, lower non-radiative relaxation and higher light yield are shown by low concentration of CQDs [110]. The inductive and conserving effects of decreasing concentration can lead to a blue-shifted emission wavelength [111]. As a result, the tunable fluorescence of CQDs can be modified by controlling the distance between particles. Since particle contact is reduced at low CQD concentrations, the blue shift is associated with an increased in fluorescence intensity, partly due to the radiation process. At higher CQD concentrations, the particle interaction increases, subsequently resulting in a red shift and a decreased in fluorescence intensity, due to self-absorption [112]. The original concentration of carbon dots (12.8 mg/mL) was diluted into different concentrations(0.1,1,and 10 mg/mL) suggested that the different concentrations of CQDs are related to the number of CQDs available in the samples and the relative distance between CQDs nanoparticles. The peak wavelength shift was approximately linear to the CQDs concentration, but the photoluminescence intensity was only approximately linear to the carbon dots concentration, only up to a concentration of 1.6 mg/mL. This means that the photoluminescence intensity tends to be saturated at high carbon dots concentration. This saturation is probably due to the process of re-absorption that occurs between carbon points at high concentrations [113].

4.4 pH

The pH dependency analysis is very important for the researchers to study the emission behaviour of CQDs, as the stability of PL is an important aspect for the applications of fluorescent nanomaterials [114]. The PL stability of CQDs under different conditions was further investigated and the findings suggested that the molecular state is affected under both strongly acidic and basic atmospheres, while the PL intensity of the carbon core edge state may increase, due to the protonation or deprotonation of the functional groups [115]. In a study, the Ph values of cellulose –based CQDs were measured by using NaOH and HCl to adjust the pH values (in the range of 1 to 13) of the CQD aqueous solution [116]. The results showed that the PL intensity of CQDs in an aqueous solution was strong and even stable in a wide range of pH values from 3 to 11, except for the strongly acidic (pH=1) and alkaline (pH=13) environments. This is probably because the electronic transition of some functional groups would be disturbed in the presence of too many H^+ or OH^- ions. The pH has a significant effect on the nature of the energy levels and, consequently, on the nature of electrical transitions in CQDs [117]. Besides, the PL intensity of CQDs produced from green tea decreased as the pH of the solution increased from 4.0 to 10.0 [118,119]. These studies indicated that the different PL intensities of the CQDs at different pH values might be related to the surface chemistry of the CQDs. CQDs are emitting longer wavelengths of fluorescence because new CQDs species are constantly being created in the medium. At an acidic pH, the carboxyl group (COOH) can remove electrons from the band of the carbon conduction core, further preventing electron-hole recombination and thus reducing fluorescence. On the other hand, the carboxyl group loses hydrogen as the pH increases, decreasing its ability to remove electrons. Thus, electron-hole recombination becomes possible and leads to an enhancement of fluorescence [120].

5. Summary and Future Direction

Hydrothermal treatment of CQDs can be done using different precursors such as organic molecule, natural source and biomass waste at varied synthesis conditions. At present, most hydrothermally synthesized CQDs are in a state of uniform dispersion and usually spherical in structure. The crystal morphology of crystals under hydrothermal conditions is closely related to the growth conditions. The same crystals may show different morphologies under different hydrothermal conditions. Due to their fascinating luminescent features, CQDs have been attracting an increasing number of researchers to explore the PL mechanisms of their bright emission, preparation of distinct multicolor products independent of excitation, and use in creative applications exploiting their prominent luminescent properties. As the particle size distribution of typical CQDs (<10 nm) is comparable to the quantum size range, some research groups believe that, similar to the traditional SQDs, the size-dependent quantum confinement effect, contributes to the luminescence emission of CQDs. Many challenges remain, including the lack of thorough interpretation of CQDs structures and luminescence origins, the difficulty in controlling CQDs reactions, time-consuming post-treatment procedures, broad emission peaks, and relatively low QYs in the long wavelength emission that limit further applications. However, it is possible that in the near future, studies on mechanisms, optimization of syntheses, and development of applications will benefit from one another and bring about inspiring progress.

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