

Synthesis of Carbon Quantum Dots and Catalytic Applications Towards Renewable Energy

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Abstract

The global enthusiasm for carbon-based nanomaterials, particularly carbon quantum dots (CQDs), has grown significantly due to their unique physicochemical characteristics and a broad spectrum of potential applications. In this review, the synthesis, classification, and potential applications of carbon quantum dots (CQDs) have been comprehensively deliberated. The synthesis approaches can be largely categorized into two different groups: the top-down approach and the bottom-up approach, while hydrothermal synthesis, microwave-assisted carbonization, and electrochemical methods are some of the most effectual and scalable tactics. CQDs are characterized by unique features: biocompatibility, less toxicity, tunable photoluminescence, and excellent chemical stability, which makes them fit for bioimaging, photocatalysis, electrocatalysis, energy generation and storage applications. The applications of CQD are conversed, with the possibility of being functional in several fields, together with optoelectronics and environmental sensing. Although scaling up and ensuring consistency are significant challenges, ongoing research holds the promise of unlocking new opportunities in advanced materials and technologies within the fields of nanotechnology and materials science.

Keywords: Nanomaterials, Carbon dots, Sustainable, Renewable Energy, Catalyst

1. INTRODUCTION

Nanomaterials based on carbon have fascinated researchers globally with utmost curiosity since the discovery of fullerenes [1]. Based on their dimensionality, researchers classified carbon nanomaterials, namely 3-dimensional, 2-dimensional, 1-dimensional, and 0-dimensional [2]. Carbon dots (CDs) have evolved as an 0D allotrope of carbon with a size ≤ 10 nm in the family of carbon nanomaterials [3]. The Valcarcel group conducted the systematic and logical categorization of CDs based on crystalline structure, quantum confinement, and nature [4]. There are three major types of CDs, including carbon quantum dots (CQDs), graphene quantum dots (GQDs), and carbonised polymer dots (PDs) [5]. CQDs are crystalline spherical nanodots with quantum confinement, while CNDs are amorphous quasi-spherical nanodots lacking quantum confinement. GQDs are nanodots with a π -conjugated graphene sheet possessing quantum confinement and crystalline structures [6]. The concept of quantum dots originated during the early 20th century with the development of principles in quantum mechanics by Max Planck and Niels Bohr that laid the foundation of nanotechnology and quantum physics [7]. Both of them have been working on theoretical backgrounds on the behaviour of electrons within solid materials and atoms. With the invention of transistors, the physics of semiconductors made tremendous progress in the mid-20th century. The characteristics of electron mobility in semiconductors began to be comprehended. In the same period, researchers started to explore the assets of semiconductor crystals. The concept of semiconductor nanocrystal was understood earlier, and its optical study has been performed well by researchers. Optical studies performed on the semiconductor nanocrystals or quantum dots were shown to be size-dependent [8]. The approach that has been used for the synthesis of CQDs is categorized into two approaches namely "top-down" and "bottom-up" [9]. The "top-down" procedure adopts the breaking down of carbonaceous materials to turn out CDs of small sizes. The "bottom-up" method uses tiny molecules as carbon sources, and CQDs are created via the condensation or chemical fusion of these molecules [10]. These nanoparticles have drawn much interest recently because of their intriguing physicochemical characteristics and wide range of potential uses. Their exceptional qualities make them

a viable option for use in biomedical, photovoltaics, batteries, supercapacitors, LEDs, fluorescent films, electrocatalysis, photocatalysis, ion detection or sensing, etc., as depicted in Figure 1.

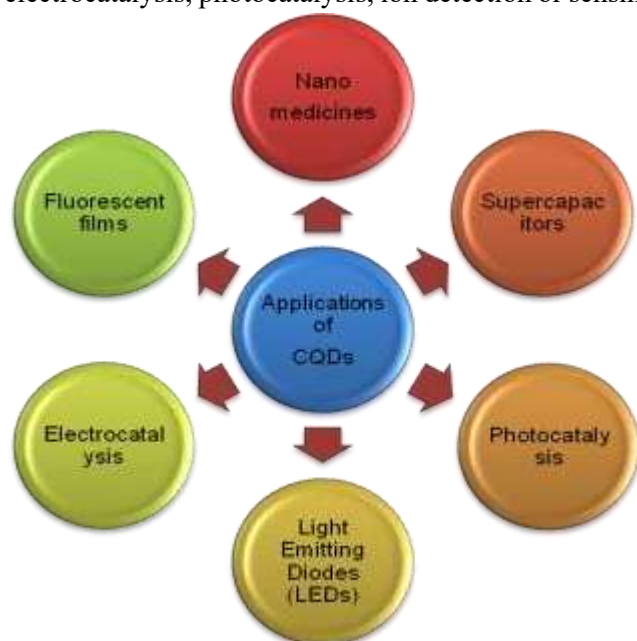


Figure 1. The representation of the various applications of carbon quantum dots (CQDs).

In other ways, CQDs are ascribed as carbon dots (CDs). While the size and photoelectrochemical characteristics of GQDs, CNDs, and PDs are comparable, their internal structures and surface chemical groups are different. CQDs from carbonaceous sources have shown outstanding performances with low toxicity, excellent biocompatibility, and simplistic synthesis methods that render them environmentally benign for several applications. This review deeply focuses on providing a comprehensive account of the synthesis, and energy applications of CQDs. This article shall provide an in-depth understanding of the subject along with the future perspectives that lies ahead.

2. SYNTHESIS OF QDS

The ideal range for the QDs length is between 4 and 20 nm. Their synthesis is essential for QDs to have a consistent size and shape. The direct approach and geometrical limits on electrons in two directions on quantum wells are used to create them. These dots are synthesized by electron etching or colloidal synthesis from a two-dimensional heterostructure [11].

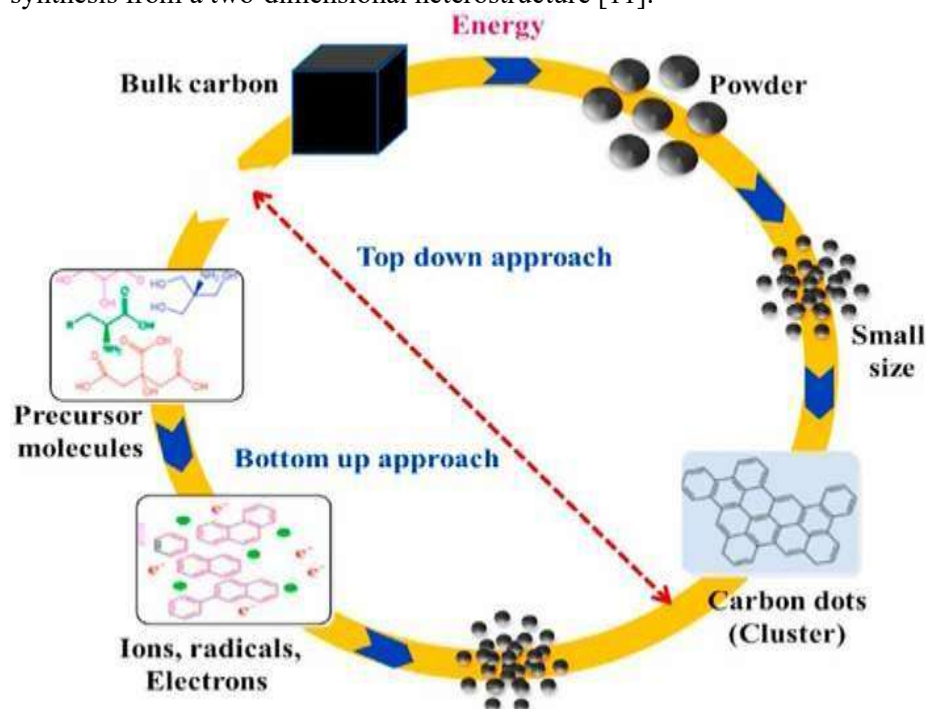


Figure 2: Synthesis routes for CQDs, exhibiting two primary strategies and a few crucial steps involved during the processes [12].

So far, several synthetic routes for the synthesis of CDs using diverse precursors have been reported. This includes both "top-down" and "bottom-up" methods, as shown in Figure 2. The top-down procedure converts macro or microscopic carbon materials to nanoscale-sized CDs by chemical or physical destruction. In contrast, the bottom-up methods start by building CDs at the nano-dimensions from molecular precursors via polymerization and carbonization. CQDs having unique properties and different synthesis techniques as shown in figure 3.

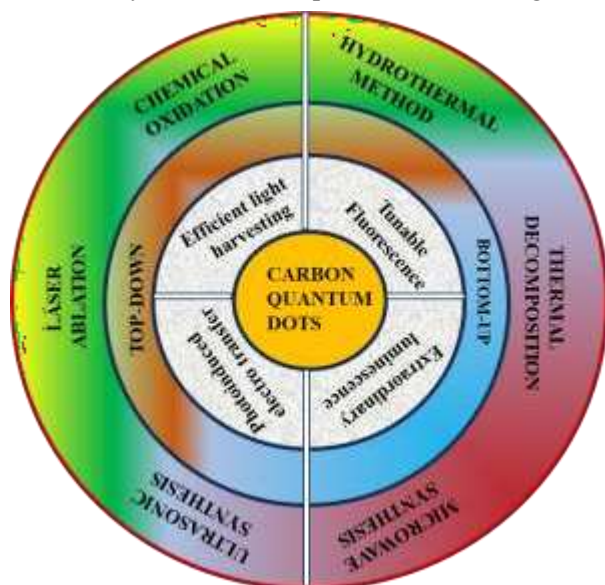


Figure 3: CQDs correlating exceptional properties and different synthesis techniques.

2.1 Top-down approaches

This approach involves the use of procedures such as laser ablation, electrochemical reactions, and arc discharge, to break down the bigger carbon resources such as carbon nanotubes, fullerene, graphite, graphene, carbon soot, activated carbon, etc. into smaller parts in a top-down approach [13]. For top-down processes, carbon structures with sp^2 hybridization that don't have effective energy gaps or band gaps are frequently utilized as starting materials [14]. The inability to obtain pure nanomaterials from the large carbon precursor, the high cost of purification, and the inability to precisely control the morphology and size distribution of CQDs are some of the drawbacks of the top-down approach, despite its great usefulness and suitability for microsystem industries ¹.

2.1.1 Ultrasound synthesis

This is regarded as a new mechanism of CQD synthesis, which uses the high energy of an ultrasonic wave to break down carbon compounds into nanoparticles (NPs) in the presence of an oxidant, alkali, or acid. The simple synthesis of CQDs with small sizes is made possible by the application of high intensity ultrasonic waves, which circumvent the difficult post-treatment procedure [15]. Li and his colleagues successfully produced PL CQDs with a diameter of less than 5 nm and a QY of 7% in 2011 after ultrasonically treating glucose in an acidic or basic environment for the first time [16].

2.1.2 Arc discharge method

The flow of electric current through a gaseous medium, resulting in an arc. Arora et al. used an arc discharge approach that involved the reorganization of carbon atoms degraded from bulk carbon precursors. This reorganization takes place inside the anodic electrode, fuelled by a gas plasma generated in a sealed reactor [17]. The temperature within the reactor can rise to a maximum of 4,000 K due to the effect of an electric current, which allows for the creation of plasma with high energy levels. CQDs are synthesized by assembling carbon vapor in the cathode. Xu et al. established the first work on the synthesis of CQDs via arc discharge. Inadvertently, Xu et al. generated three distinct carbon nanoparticles with varied relative molecular mass and fluorescence characteristics when manufacturing SWNTs using arc discharge [18]. When stimulated at a wavelength of 365 nm, the synthesized CQDs generate fluorescence in the orange, yellow, and blue-green areas. Subsequent experiments showed that a hydrophilic carboxyl group was linked to the surface of CQDs. Because diverse sizes of carbon particles are created during the

discharge process, the CQDs produced using this approach generally exhibit a broad particle size distribution despite having acceptable water solubility.

2.1.3 Laser ablation

This approach involves applying a high-energy laser pulse at the top layer of a target material, exposing it to a thermodynamic condition of high pressure and temperature. As a result, the material is rapidly heated and subsequently evaporated into plasma. Subsequently, vapor crystallizes to form nanoparticles [19]. Laser ablation is widely recognized as a highly effective method for the production of carbon quantum dots (CQDs). However, its complex functioning and associated costs have limited its application [20].

2.1.4 Electrochemical oxidation

The electrochemical oxidation process involves disintegrating larger carbon structures of coal into minor units through the use of an electrolyte in an electrochemical cell to synthesize carbon dots (CDs). He et al. proposed an extensive synthesis of multi-colored GQDs from low-cost coke using a coke lump as a working electrode and a platinum plate as a counter electrode, with a methanol-water mixture as the electrolyte. This method produced orange-GQDs, yellow-GQDs, and green-GQDs with dimensions between 3.02 and 4.61 nm by adjusting the water concentration in the electrolyte. The photoluminescence emissions in GQDs were up to 500-560 nm. Sodium borohydride further reduces green GQDs in which surface -C=O groups transform into -OH groups and produce blue fluorescent GQDs (B-GQDs) with fewer defects [21]. It was found that the most typical mechanism for electrochemical oxidation corresponds to the splitting of graphitic domains by active radicals, as well as to the intercalation of anions within a coal matrix. However, despite all the advantages in low cost, efficiency, and scale-up potential, the scope of research into electrochemical exfoliation synthesis of CDs remains significantly limited in the area and requires new approaches in the field.

2.1.5 Chemical oxidation and etching

Numerous techniques have been developed to fabricate carbon dots (CDs) from coal however, the most popular ones are chemical oxidation and etching. $\text{HNO}_3/\text{H}_2\text{SO}_4$ and other strong acids are typically used in the traditional oxidative method for making CDs, although this process is linked to environmental damage [22]. By introducing O, N, and S-contained functionalities through chemical oxidation techniques, the small crystalline carbon domains in coal matrices can be readily replaced, rendering them hydrophilic and flawed. Green oxidants like ozone and hydrogen peroxide (H_2O_2) have emerged as safer alternatives. For example, Dong et al. produced monodispersed spherical CDs of 3-5 nm by refluxing coal with HNO_3 . [23]. Hu et al. employed a carbonization and acidic etching strategy, showing that carbonization temperature affected CD size [24]. Similar oxidation techniques were used to synthesize graphene quantum dots (GQDs) in other studies, which highlighted their optical characteristics and produced sizes of 2-4 nm.

2.2 Bottom-up approach

2.2.1 Microwave pyrolysis

This method has received extensive appreciation and implementation in bottom-up methods, mainly because of its outstanding performance in promoting rapid production and further commercialization. For instance, an experiment that employs a simple microwave pyrolysis approach to synthesize CQDs consists of mixing PEG with sugar (like glucose or fructose) in a liquid solution, leading to a transparent solution. Then, the obtained solution was heated using a microwave oven [25].

2.2.2 Combustion/thermal routes

The bottom-up method to CQD synthesis has seen a significant increase in interest recently. This interest can be attributed to several factors, including the simplicity of the technique, scalability, the ability to precisely control the environmentally friendly aspects of the operation, and cost-effectiveness. Wang et al. initially proposed the combustion/thermal oxidation method for synthesizing CQDs, which garnered interest from several researchers in the field [26]. Li et al. synthesized bright carbon quantum dots (CQDs) by pyrolyzing citric acid and subsequently functionalizing them with carboxyl groups at elevated temperatures using acetic acid moieties [27]. The surface of the GQDs exhibited prominent carboxyl groups, and the synthesized CQDs consistently measured 8.5 nm in particle size. The presence of oxygen-containing functional groups enhances the electrocatalytic process in aqueous environments by increasing the capacity for water molecule adsorption.

2.2.3 Solvothermal/Hydrothermal synthesis

The hydrothermal and solvothermal synthesis approach involves breaking the interlayer forces of coal particles within the reaction medium and cutting large sp^2 carbon domains into smaller pieces. For instance, Saikia and team published a simple hydrothermal approach for synthesizing CDs from low-grade

sub-bituminous coal [28]. This procedure involved the mixing of coal in ultra-pure water. There was no additional agitation when this mixture was placed inside an autoclave, followed by being placed in a furnace for 120 min at 200°C. Following filtration and dialysis, CDs could be obtained after concentrating the filtrate by rotary evaporation. The CDs exhibited a spherical morphology with an average diameter size ranging from 3-6.5 nm. These CDs were found to be uniform and without agglomeration distributed. The hydrothermal method is shown to be easy and useful in the synthesis of carbon nanomaterials at a rational cost.

Table 1: Synthesis methods advantages and disadvantages of CDQs [28].

Sl. no.	Techniques	Advantages	Disadvantages	Applications
1.	Top-up	Use raw carbon sources	Less control over shape and size	GQDs
2.	Bottom-Down	specific control over size, shape, and properties	It may require exclusive equipment	Quantum dots for optoelectronics, biomedicine, and catalysis
3.	Arc Discharge	Simple setup	High energy consumption	Single-walled carbon nanotubes (SWNTs) and CQDs with variable fluorescence
4.	Laser Ablation	Limited size distribution, excellent water solubility	Complex and expensive	CQDs with tunable fluorescence characteristic
5.	Acidic Oxidation	Introduces hydrophilic groups, enhances water solubility and fluorescence	Uses strong acids, potential environmental concerns	Heteroatom-doped CQDs for electrocatalysis
6.	Combustion/Thermal Routes	Simple, inexpensive, scalable	Partial control over size distribution	Functionalized CQDs for electrocatalysis
7.	Microwave Pyrolysis	Fast synthesis, environmentally friendly	Limited research on specific properties	CQDs with O ₂ containing functional groups for metal ion coordination
8.	Hydrothermal/Solvothermal	Simple, effective, produces uniform CQDs	It requires high pressure and temperature	Coal-based CDs for various applications
9.	Electrochemistry	Room temperature and pressure, easy control over properties	Limited research on application as electrocatalysts	Blue-emitting CQDs for Hg ²⁺ detection

10.	Electrochemical Oxidation	Large-scale synthesis, tunable emission colors	Limited research on coal-based CDs	Multi-colored graphene quantum dots (GQDs)
11.	Colloidal Synthesis	Accurate control over shape, size, and optical properties	Requires specific solvents and an inert atmosphere	Quantum dots for various applications
12.	Microemulsion	Precise size and property control	Complex procedures	Quantum dots for optoelectronic devices
13.	Sol-Gel	Versatile, controlled synthesis	Requires purification steps	CQDs for optoelectronics, biomedicine, and catalysis
14.	Molecular Beam Epitaxy (MBE)	Atomic-level precision, superior crystalline structures	High cost, involves ultra-high vacuum	Quantum dots for quantum computing and photonics
15.	Chemical Oxidation and Etching	Effective	Traditional methods can be polluting	CDs from coal for various applications

3. APPLICATIONS OF CQDS

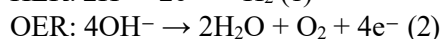
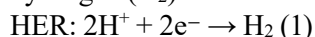
CQDs are gaining importance owing to their unique characteristics, including high fluorescence, tunable surface chemistry, excellent dispersibility, and low toxicity [29]. These features make CQDs valuable in fields like bioimaging, environmental sensing, optoelectronics, energy storage, and catalysis. In energy applications, CQDs are effective in solar cells, supercapacitors, and batteries, benefiting from their high surface area and electron transfer capabilities. Scalable synthesis methods enable their widespread use across sectors, including flexible electronics, smart packaging, and anti-counterfeiting. Overall, CQDs' versatility and functional adaptability position them as powerful materials for novel applications in a variety of scientific and technical disciplines.

3.1 Solar cells, fluorescent films, and LEDs

The sun is a sustainable and renewable source of energy, and harnessing its energy efficiently is essential for sustainable energy solutions. Solar cells, or photovoltaic (PV) cells, are a prominent technology for producing electricity from solar energy through the absorption of sunlight and subsequent electron-hole pair generation and separation in solid-state devices [30]. Recent advances in solar cell technology have resulted in third-generation solar cells that emphasize low-cost, eco-friendly materials with high energy conversion efficiency. The characteristics and structure of these new-generation cells' constituent parts, especially their absorption capacity for visual light and producing electron-hole pairs via photoexcitation, have a significant impact on how well they function. Kovalchuk et al. synthesized a nanocomposite of polymer that was luminescent by combining coal-derived GQDs with polyvinyl alcohol (PVA). In this composite, PVA serves as a matrix of polymers, while GQDs add fluorescent properties, resulting in high dispersibility and brightness without additional surface modification [31]. This emission stability and broad visible light coverage underscore CQDs' potential as a low-cost, non-toxic alternative to traditional phosphors in lighting and optoelectronic devices.

3.2 Electrocatalysis

Water splitting is the most desirable method despite its high costs due to its reliance on abundant resources and minimal environmental impact [32]. The electrochemical mechanisms for oxygen (O₂) and hydrogen (H₂) evolution during water electrolysis are as follows:

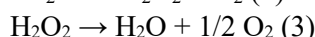
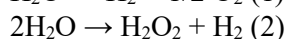
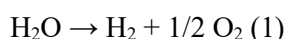


Enhancing the efficiency of electrocatalysts is crucial for optimizing these reactions, particularly for OER, which involves complex electron transfer processes and the creation of O-H and O-O bonds.

In addition to their small size, stability, and increased conductivity, CQDs are becoming viable alternatives to expensive metal catalysts like ruthenium and platinum for essential energy reactions like the oxygen evolution reaction (OER) and hydrogen evolution reaction (HER) using electrochemical setup [33]. These reactions are crucial in applications like water electrolysis, metal-air batteries, and fuel cells. Heteroatom doping and hybridization with carbon nanoparticles significantly enhance CQDs' electrocatalytic activity. For instance, N-doped CQDs (N-CQDs) with oxygen-rich functional groups show improved ORR performance, while CQD-based nanocomposites such as CoP/CQDs, Co₃O₄/N-CQDs, and MnO₂/CQDs demonstrate excellent activity for OER due to better charge exchange and numerous active sites [34]. Furthermore, CDs can improve electrocatalytic performance by combining with other functional carbon nanostructures. They used solvothermal treatment after electrochemically exfoliating graphite powders to create a hybrid of graphene and CQDs [35]. Carbon-based materials, such as CQDs, function better when doped because they give them more characteristics that increase their electrocatalytic efficiency. However, choosing alloy nanoparticles for doping carefully is essential because making the wrong decisions could ruin the intended results. Despite the potential of pure CQDs, doping can be taken into consideration if supplementary elements provide synthetic or financial difficulties. One particularly sustainable and eco-friendly way of producing CQDs is the hydrothermal synthesis process, which uses naturally occurring precursors.

3.3 Photocatalytic Hydrogen Generation

Tian et al. used CQDs of sizes ranging between 2 and 10 nm to modify g-C₃N₄ sheets. Compared to graphitic carbon nitride alone, the CQD/g-C₃N₄ composite demonstrated an improved photocatalytic hydrogen production rate of 116.1 mmol h⁻¹, which is three times greater. The increased production of photocatalytic hydrogen by CQD/g-C₃N₄ composites was attributed to the CQDs' dual roles as light harvesters and electron reservoirs. CQDs/g-C₃N₄ were produced as well by Pan et al., investigated that the band gap for CQDs/g-C₃N₄ dropped from 2.77 eV for g-C₃N₄ alone to 2.68 eV. When exposed to visible light, the photoexcited holes and electrons will produce g-C₃N₄ and produce an electric field in the interior portion. This was further supported by first-principle DFT calculations, which showed the composite's local electronic structure and the strong hybridization between g-C₃N₄ and CQDs that produces an internal electric field. Rhodamine B will degrade due to the rapid transfer of photogenerated electrons from g-C₃N₄ to CQDs and the retention of photogenerated holes on g-C₃N₄, which creates spatial separation. This led to the simultaneous degradation of rhodamine B and hydrogen generation rate of 1291 mmol g⁻¹ h⁻¹ in the CQDs/g-C₃N₄ composite. Compared to the identical CQD/g-C₃N₄ photocatalyst in water, 1.94 times as much hydrogen was generated. In summary, photocatalytic hydrogen production is enhanced when holes are effectively used to break down rhodamine B, preventing the rapid recombination of photogenerated holes and electrons. CQDs displayed up-conversion fluorescence via a two-photon process when exposed to visible and near-ultraviolet light. The electrons in TiO₂ will be energized by some of the visible light that is transformed into ultraviolet light, producing photoexcited charge carriers. In addition to the breakdown of H₂O₂ by CQDs, heterojunctions like TiO₂-C₃N₄ lengthen the life span of photogenerated charge transporters. The four-electron process is explained as follows:



The tertiary composites C₃N₄ produces H₂O₂ on the surface of photocatalysts and serves as a hole acceptor. Equation (3), illustrates how the CQDs on the surface of TiO₂/C₃N₄ heterojunctions break down H₂O₂ into H₂O with the evolution of oxygen. Equation (1) illustrates how the photogenerated electrons of TiO₂ will cause the thus-produced H₂O to liberate hydrogen. Additionally, the formation of a porous structure by C₃N₄ increases the specific surface area, which supplies the active sites for the synthesis of hydrogen. Comparing CQD-TiO₂-C₃N₄ tertiary composites to other binary and individual components, the combined impacts of all these improve the photocatalytic hydrogen production activity, as shown in Figure 4 [38].

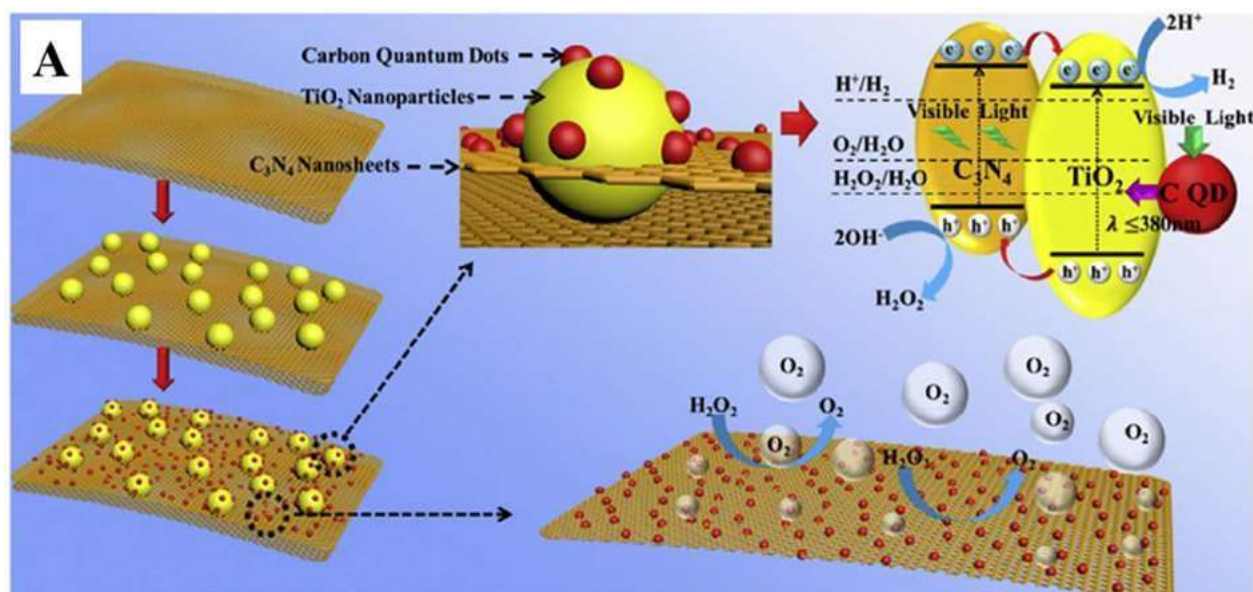


Figure 4: Diagram showing the CQDTiO₂-C₃N₄ tertiary composite's photocatalytic hydrogen generation capability. [38]

4. FUTURE PERSPECTIVES ON UTILIZING CQDS

The challenges to be faced in the near future regarding energy storage are enormous due to the increase in industrial activity, population growth, transition to electric mobility, among other factors. For this reason, it is imperative to both develop new technologies and scale them to the real world. As discussed in this review article, carbon materials play a key role in the development of these new technologies. Nowadays, the trend is to use carbon materials of natural origin, due to their wide availability and low cost, also this enhances the suitability of the process.

The future of activated carbon research is poised for substantial expansion, driven by advancements in sustainable and cost-effective production methods. This growth will hinge on the creation of activated carbons with precisely tailored properties, a crucial aspect underscored by notable performance variations observed in activated carbons derived from different raw materials under identical processing conditions. This highlights the important impact of precursor composition and structure on reactivity in pyrolysis and activation reactions. Achieving a high surface area and a well-defined pore size distribution is essential for efficient electrolyte transport. Additionally, the ability to incorporate controlled oxygen functionalities is crucial for enhancing electrical conductivity and pseudocapacitance. The attributes are crucial for maximizing the potential of plant-derived activated carbons and facilitating the advancement of next-generation energy storage devices with enhanced capabilities.

5. CONCLUSIONS

Carbon quantum dots (CQDs) represent a rapidly advancing field within nanotechnology and materials research, offering numerous opportunities for theoretical exploration and practical applications. Advancements in physical and synthesis techniques have enabled precise control over the size, structure, and surface chemistry of CQDs. Consequently, scalable and effective synthesis methods, including hydrothermal methods, microwave-assisted techniques, and electrochemical routes, have been developed, facilitating their application across various disciplines. Due to their unique optical properties, including size-dependent photoluminescence, carbon quantum dots (CQDs) are in high demand for display technologies and optoelectronic applications. Their superior biocompatibility and adjustable surface chemistry enable various biological applications, including drug administration, photothermal therapy, and bioimaging. CQDs have demonstrated potential as versatile sensors for the detection of ions, biomolecules, and environmental pollutants, thereby enhancing their applicability in environmental monitoring and diagnostics. As research on CQDs progresses, it is anticipated that synthesis methods will improve in precision, allowing for tailored characteristics to meet specific application requirements. Additionally, advancements in our comprehension of the structure of CQDs and surface functionalization processes will enable the development of new materials with enhanced functions and performance. The transition of carbon quantum dots (CQDs) from experimental studies to practical applications represents a significant scientific endeavor with considerable potential benefits for society.

and industry. Future advancements in advanced materials and technologies will be influenced by current research initiatives, which have the potential to create new avenues in materials science and nanotechnology.

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