

## Synthesis and Characterization of Carbon Quantum Dot for Device Fabrication

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**Abstract:** Quantum dot have been extensively investigated in the last decade by virtue of their unique physicochemical, optical and electronic properties, have displayed tremendous momentum in numerous fields such as bio sensing, bio imaging, drug delivery, optoelectronics, photovoltaics and photo catalysis. In this review we describe the quantum dot, types of quantum dot, properties of carbon quantum dot like Adsorption, Fluorescence, Phosphorescence, Up-conversion photoluminescence (UCPL), PL mechanism. We described various characterization techniques like Energy-dispersive X-ray spectroscopy, Transmission electron microscopy (TEM), UV –Visible Spectroscopy, X-RAY Diffraction (XRD). We have also described various synthesis method for CQD like Chemical ablation, Electrochemical carbonization, Laser ablation, Microwave irradiation and specially detailed discussion on the Hydrothermal/solvothermal treatment method for synthesis of CQD is provided here. We have proposed work of carbon quantum dot in device fabrication.

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### Introduction:

Carbon quantum dots are intriguing recently discovered members of the carbon nanomaterials family alongside carbon nanotubes, fullerenes, and graphene.<sup>[1,2]</sup> The general description of CQDs is that of nanometer size particles consisting of an sp<sup>2</sup> hybridized graphitic core functionalized with polar carboxyl or hydroxyl groups on the surface.

#### 1.1 Carbon quantum dot-

Carbon-based quantum dots consisting of graphene quantum dots (QGDs) and carbon quantum dots (CQDs, C-dots or CDs) are a new class of carbon nanomaterials with sizes below 10 nm. They were first obtained during the purification of single-walled carbon nanotubes through preparative electrophoresis in 2004, and then *via* laser ablation of graphite powder and cement in 2006. Carbon-based quantum dots with fascinating properties have gradually become a rising star as a new Nanocarbon member due to their benign, abundant and inexpensive nature. Carbon is commonly a black material, and was generally considered to have low solubility in water and weak fluorescence. Wide attention has been focused on carbon-based quantum dots because of their good solubility and strong luminescence, for which they are referred to as carbon Nano lights.

Major property of QDs is they can emit light. The light emitted by these dots depends on wavelength of light used for excitation. The emission is size dependent that is the smaller the size of dots the better their photoluminescence efficiency.<sup>[3]</sup> Conventional QDs are semiconductor nanocrystals that inherently

fluoresce at specific wavelength in the visible range. conventional quantum dots are based on metallic elements, which has raised concern over toxicity, stability and high cost. Quantum dots with size below 10 nm exhibit strong fluorescence with tunable emission depending on the size, surface structure and excitation wavelength etc.

Compared to traditional semiconductor quantum dots, photo luminescent CQDs are superior in terms of aqueous solubility, functionalizability, resistance to photo bleaching, toxicity, biocompatibility and exhibit broader photo luminescence profiles.<sup>[4,5]</sup>

#### 1.2 Types of quantum dot (QD)-

There are many types of quantum dot. Two of them are described below-

##### 1.2.1 Carbon quantum dot (CQD)-

CQDs were first discovered by Xu et al. in 2004 accidentally during the purification of single-walled carbon nanotubes.<sup>[6]</sup> This discovery triggered extensive studies to exploit the fluorescence properties of CQDs. Much progress has been achieved in the synthesis, properties and applications of CQDs.

As a new class of fluorescent carbon nanomaterials, CQDs possess the attractive properties of high stability, good conductivity, low toxicity, environmental friendliness, simple synthetic routes as well as comparable optical properties to quantum dots.<sup>[7]</sup> Carbon quantum dots have been extensively investigated especially due to their strong and tunable fluorescence emission properties, which enable their applications in biomedicine, optronics, catalysis, and sensing.

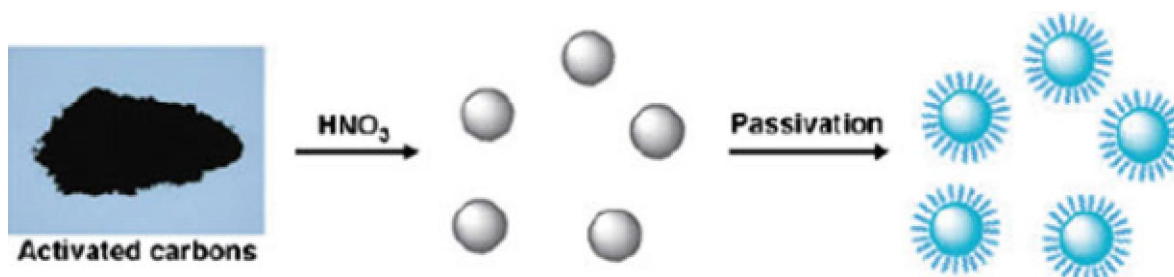


Fig. 1.2.1 Synthesis of Carbon-Dots from activated carbon. Reproduced from Ciao et al. with permission of the Royal Society of Chemistry

### 1.2.2 Semiconductor quantum dot-

Three-dimensionally confined semiconductor quantum dots have emerged to be a versatile material system with unique physical properties for a wide range of device applications. With the advances in nanotechnology and material growth techniques for both epitaxial and colloidal quantum dots, recently the research has been shifted largely towards quantum dot based devices for practical applications. Semiconductor Quantum Dots presents an overview of the background and recent developments in the rapidly growing field of ultra-small semiconductor micro crystallites, in which the carrier confinement is sufficiently strong to allow only quantized states of the electrons and holes.

### 1.3 Properties

The structures and components of CQDs determine their diverse properties. Many carboxyl moieties on the CQD surface impart excellent solubility in water and biocompatibility.

#### 1.3.1 Adsorption-

CQDs typically show optical absorption in the UV region with a tail extending to the visible range

(see Fig.1.3.1A) for representative spectra). There may be some absorption shoulders attributed to the p-p\* transition of the C-C bonds, the n-p\* transition of C=O bonds and/or others.

#### 1.3.2 Fluorescence-

One of the most fascinating features of CQDs, both from fundamental and application-oriented perspectives, is their PL (see Fig.1.3.1 for representative spectra). In most cases of early study, one unique feature of the PL of CQDs was the clear  $\lambda_{ex}$  dependence of the emission wavelength and intensity. Whether this occurs because of optical selection of differently sized nanoparticles (quantum effect) and/or different emissive traps on the CQDs surface or another mechanism is currently unresolved. Moreover, the requirement for surface passivation is only partially understood, but appears to be linked to the synthetic method. However, more and more cases have emerged with an  $\lambda_{ex}$  independent emission position<sup>[8]</sup> which may be attributed to their uniform size and surface chemistry.

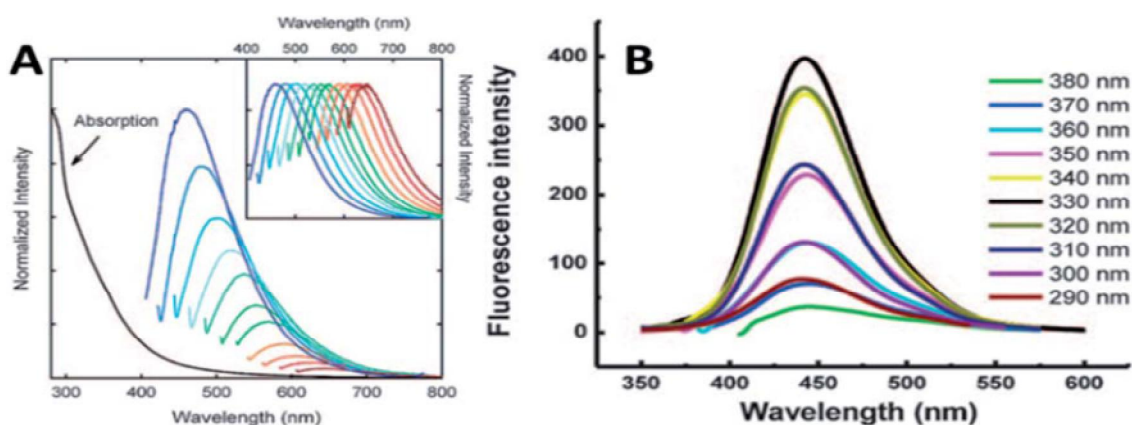


Figure 1.3.1. Absorbance and PL spectra with increasingly longer excitation wavelengths (in 20 nm increments starting from 400 nm) of 5 nm PPEI-EI CQDs in an aqueous solution formed using laser ablation methods (inset shows the normalized PL spectra). Adapted with permission.2 Copyright 2006, American Chemical Society. (B) PL of 1.9 nm CQDs at different excitation wavelengths of 290–380 nm. Adapted with permission.19 Copyright 2008, Royal Society of Chemistry

### 1.3.3 Phosphorescence-

The phosphorescence properties of CQDs were discovered recently. A pure organic room temperature phosphorescent (RTP) material was obtained based on water soluble CQDs and its phosphorescent lifetime was lengthened to the sub-second order ( $\approx 380$  ms).<sup>[9]</sup> By dispersing the CQDs into a polyvinyl alcohol (PVA) matrix, clear phosphorescence could be

observed at room temperature when excited with UV light. Preliminary investigations suggested that the phosphorescence originated from the triplet excited states of aromatic carbonyls on the surface of the CQDs. The matrix PVA molecules can effectively protect the triplet excited state energy from rotational or vibrational loss by rigidifying these groups with hydrogen bonding (Fig.1.3.2).

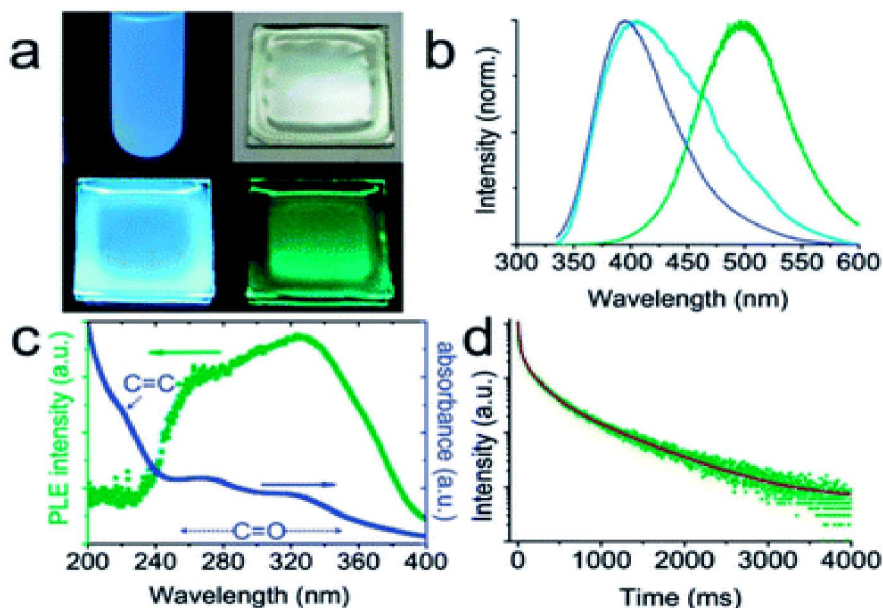


Figure 1.3.2 Digital photographs and the corresponding spectra of CQDs: dispersed in water under UV light (a: upper left; b: blue line) dispersed in a PVA matrix under daylight (a: upper right), UV light (a: lower left b: cyan line) and right after UV light has been turned off (a: lower right; b: olive line). The UV excitation for the photographs is 365 nm, while that for the spectra is 325 nm.

(c) Phosphorescence excitation spectrum (olive dots) and absorption spectrum (blue dots) of CQDs dispersed in water.

(d) Time-resolved phosphorescence spectrum. Adapted with permission. Copyright 2013, Royal Society of Chemistry.

### 1.3.4 Up-conversion photoluminescence (UCPL)-

The UCPL properties of CQDs can be attributed to the multi-photon activation process, in which the simultaneous absorption of two or more photons leads to the emission of light at a shorter wavelength than the excitation wavelength (anti-Stokes type emission). The UCPL of CQDs opens up new opportunities for cell imaging with two-photon luminescence microscopy, as well as highly efficient catalyst design, for applications in bioscience and energy technology. Upon excitation in the NIR region, the PL spectra showed a fixed emission peak at 540 nm that did not shift with a variation of the excitation wavelength which was different from that previously reported<sup>[10]</sup> The fixed emission position showed that the emission

occurs from the lowest single state irrespective of the mode of excitation.

### 1.3.5 PL mechanism-

Although there have been many efforts focused on the physicochemical properties of CQDs, the origin of the observed optoelectronic behavior is a topic of discussion to date.

The origin of the PL of CQDs has been assigned to several reasons in the literature: optical selection of differently sized nanoparticles (quantum effect), defects and surface states, surface groups, surface passivation, fluorophores with different degrees of  $\pi$ -conjugation, and the recombination of electron-hole pairs localized within small  $sp^2$  carbon clusters embedded within a  $sp^3$  matrix.

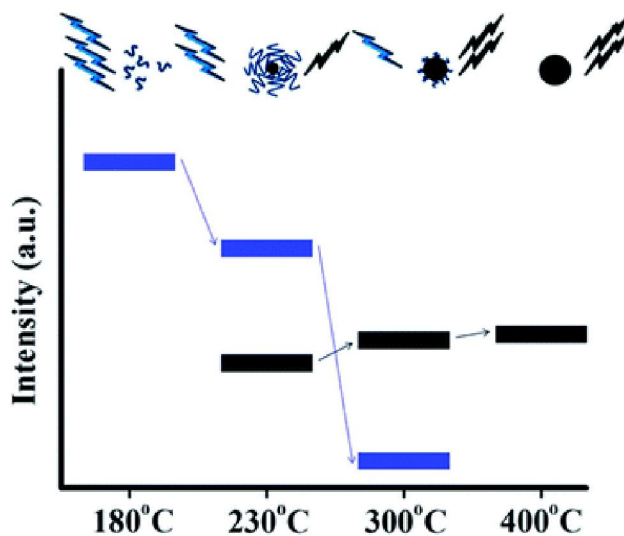


Figure 1.3.3 Schematic representation of the emission characteristics of three photoactive species produced from the thermal treatment of a mixture of CA and EA. During pyrolysis, the organic fluorophores (blue groups) are consumed for the build-up of the carbonaceous core (black sphere) so that the PL component, which corresponds to the carbonaceous core (black bars), increases at the expense of the component that arises from the organic fluorophores (blue bars). Adapted with permission. Copyright 2011, American Chemical Society.

This are the different reviews we have studied about properties, synthesis, characterization techniques of CQD. Now we have decided to synthesis CQD by Hydrothermal/solvothermal method because this method is a low cost, environmentally friendly, and nontoxic route to produce CQD.

### Synthesis and characterization techniques of CQD

#### 3.1 Synthesis Methods-

There are different methods for synthesis of CQD. Few of them are described below.

##### 3.1.1 Chemical ablation-

Strong oxidizing acids carbonize small organic molecules to carbonaceous materials, which can be further cut into small sheets by controlled oxidation. This method may suffer from harsh conditions and drastic processes. Peng and Trivas-Sejdic reported a simple route to prepare luminescent CQDs in an aqueous solution by dehydrating carbohydrates with concentrated  $H_2SO_4$ , followed by breaking the carbonaceous materials into individual CQDs with  $HNO_3$ , and finally passivating with amine-terminated compounds (4,7,10-trioxa-1,13-tridecanediamine).<sup>[27]</sup>

##### 3.1.2 Electrochemical carbonization-

Electrochemical soaking is a powerful method to prepare CQDs using various bulk carbon materials as precursors. However, there are only a few reports about electrochemically carbonizing small molecules to CQDs. Preparation of CQDs *via* the electrochemical

carbonization of low-molecular-weight alcohols is proposed by Zhang and co-workers.<sup>[28]</sup>

##### 3.1.3 Laser ablation-

**Laser ablation synthesis in solution (LASiS)** is a commonly used method for obtaining colloidal solution of nanoparticles in a variety of solvents. In the LASiS method, nanoparticles are produced during the condensation of a plasma plume formed by the laser ablation of a bulk metal plate dipped in a liquid solution. LASiS is usually considered a top-down physical approach. In the past years, laser ablation synthesis in solution (LASiS) emerged as a reliable alternative to traditional chemical reduction methods for obtaining noble metal nanoparticles (NMNp).

##### 3.1.4 Microwave irradiation-

Microwave irradiation of organic compounds is a rapid and low-cost method to synthesize CQDs. Using sucrose as the carbon source and diethylene glycol (DEG) as the reaction media, green luminescent CQDs were obtained within one minute under microwave irradiation<sup>[29]</sup> These DEG-stabilized CQDs (DEG-CQDs) could be well-dispersed in water with a transparent appearance. With an increase in the excitation wavelength, the intensity of the PL first increased to a maximum (360 nm excitation) and then decreased.

##### 3.1.5 Hydrothermal/solvothermal method for synthesis of CQD-

There are different types of synthesis method for CQD as we have discussed above. We are planning to do synthesis of CQD using this method because Hydrothermal/solvothermal method is a low cost, environmentally friendly, and nontoxic route to produce novel carbon-based materials from various precursors. Typically, a solution of organic precursor is sealed and reacted in a Hydrothermal/solvothermal reactor at high temperature. CQDs were prepared *via* HTC from many precursors such as glucose, citric acid.

Bhunia *et al.* synthesized two kinds of the CQDs, hydrophobic and hydrophilic with diameters less than 10 nm from the carbonization of carbohydrates.<sup>[30]</sup>

Hydrothermal/solvothermal synthesis includes the various techniques of crystallizing substances from high-temperature aqueous solutions at high vapor pressures; also termed "Hydrothermal/solvothermal method". The term "Hydrothermal/solvothermal" is of geologic origin. Geochemists and mineralogist have studied Hydrothermal/solvothermal phase equilibria since the beginning of the twentieth century. George W. Morey at the Carnegie Institution and later, Percy W. Bridgman at Harvard University did much of the work to lay the foundations necessary to containment of reactive media in the temperature and pressure range where most of the Hydrothermal/solvothermal work is conducted.

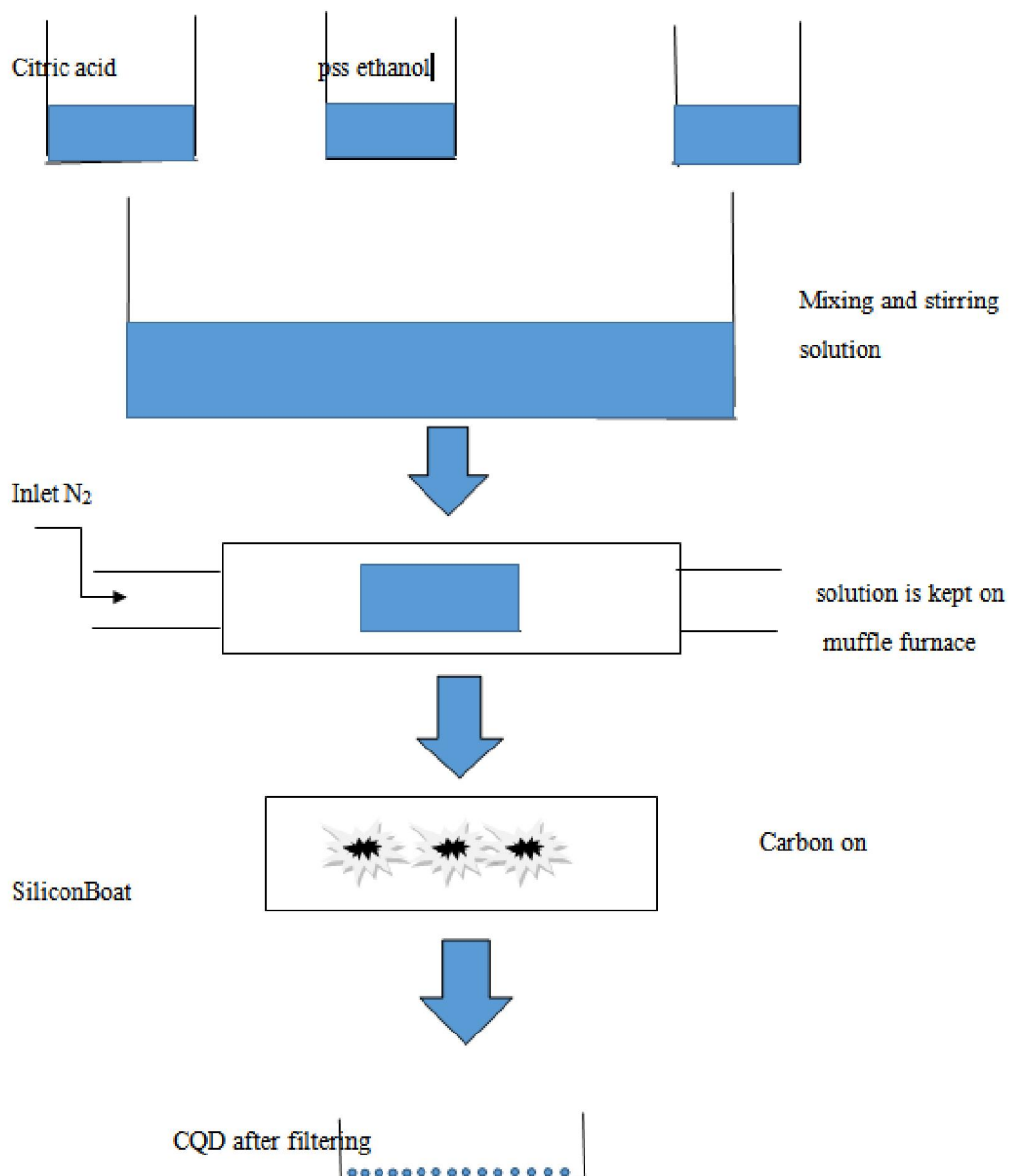


Figure 3.1.5.1 schematic diagram of hydrothermal/solvothermal process.



Hydrothermal/solvothermal synthesis can be defined as a method of synthesis of single crystals that depends on the solubility of minerals in hot water under high pressure. The crystal growth is performed in an apparatus consisting of a steel pressure vessel called an autoclave, in which a nutrient is supplied along with water. A temperature gradient is maintained between the opposite ends of the growth chamber. At the hotter end the nutrient solute dissolves, while at the cooler end it is deposited on a seed crystal, growing the desired crystal.

Advantages of the Hydrothermal/solvothermal method over other types of crystal growth include the ability to create crystalline phases which are not stable at the melting point. Also, materials which have a high vapor pressure near their melting points can be grown by the Hydrothermal/solvothermal method. The method is also particularly suitable for the growth of large good-quality crystals while maintaining control over their composition. Disadvantages of the method include the need of expensive autoclaves, and the impossibility of observing the crystal as it grows.

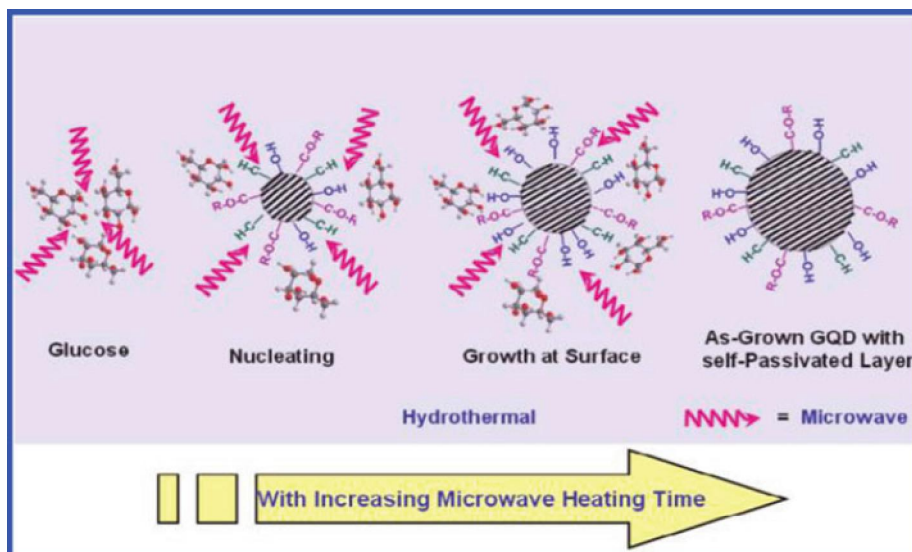


Figure 3.1.5.2 Hydrothermal/solvothermal synthesis of Carbon-Dots using glucose as a carbon source. A model for nucleation and growth of Carbon-Dots induced by microwave heating of glucose solution. Reprinted with permission from Tang et al. Copyright (2006) American Chemical Society

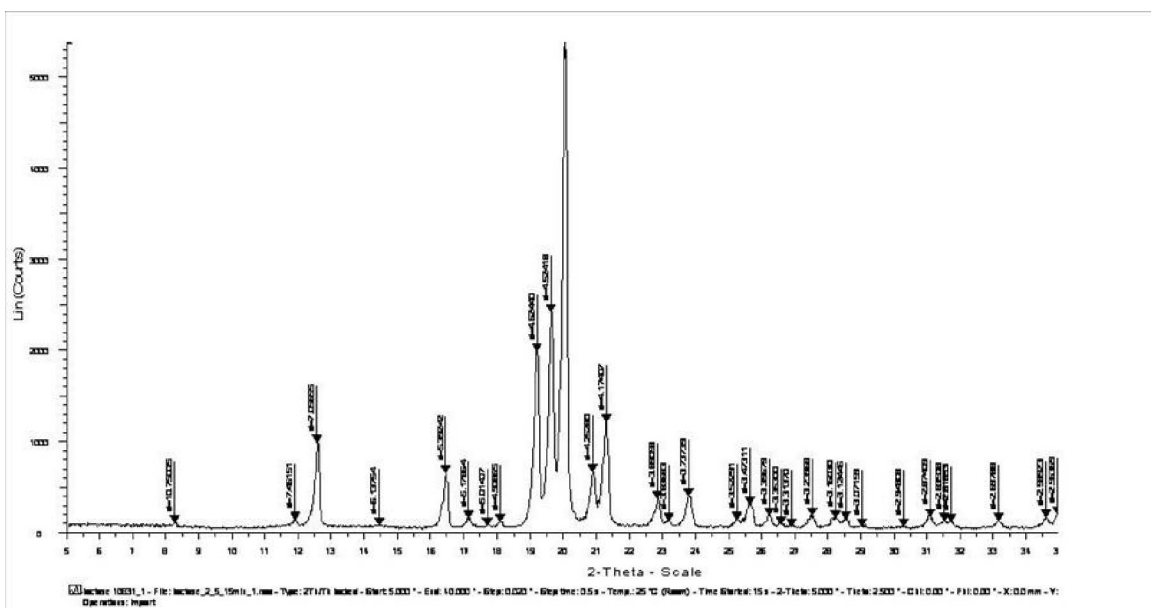


Fig 3.2.4 graph representing XRD peaks

## Applications

### 4.1. Bio imaging-

As fluorescent nanomaterials with biocompatibility and low bio toxicity, CQDs show great potential for fluorescent bio imaging, and multimodal bio imaging of cells and tissues, which

have been reviewed elsewhere. The pioneering work on CQDs for bioimaging *in vitro* and *in vivo*<sup>[31]</sup> was reported by Sun's group. Confocal microscopy images of *E. coli* ATCC 25922 labeled with the PEGylated CQDs were obtained at different excitation wavelengths.

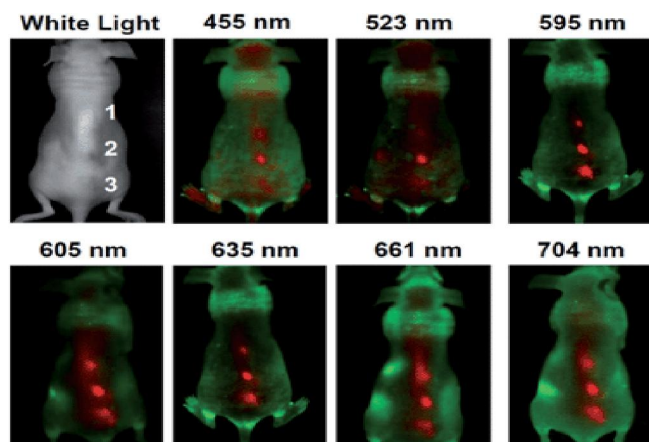


Figure 4.1 *In vivo* fluorescence images of a CQDs-injected mice. The images were taken at various excitation wavelengths. Red and green represent fluorescent signals of the CQDs and the tissue auto fluorescence, respectively. Adapted with permission.

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### 4.2. Biosensor-

CQDs have been used as biosensor carriers for their high solubility in water, flexibility in surface modification, nontoxicity, excitation-dependent multicolor emission, excellent biocompatibility, good

cell permeability, and high photo stability. The CQDs-based biosensors can be used for visual monitoring of glucose, cellular copper, phosphate, iron, potassium, Ph., and nucleic acid.

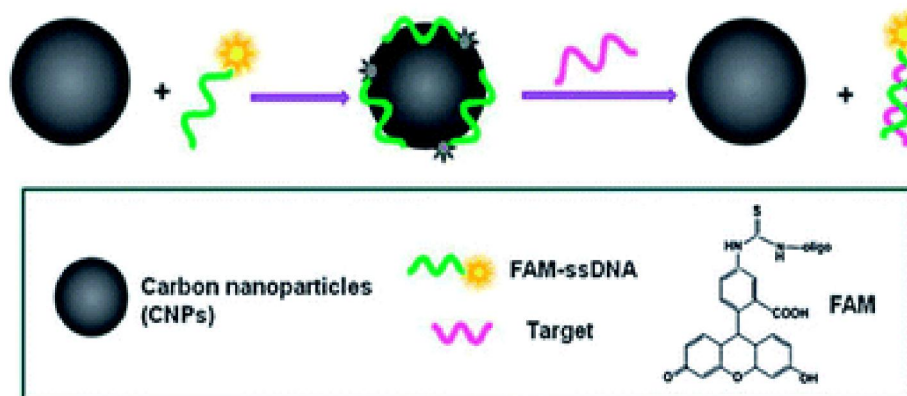


Figure 4.2 A schematic (not to scale) illustrating the CQD-based fluorescent nucleic acid detection. Adapted with permission.

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### 4.3 Organic solar cells (OSCs)-

A simple and effective method to prepare CQDs and their polymer based composites was developed and the CQDs with an excitation wavelength

independent PL can self-assemble in solution but not agglomerate in the solidified composite.<sup>[32]</sup> Accordingly, the composite with CQDs luminesce and exhibits a luminescent down-shifting (LDS) property.

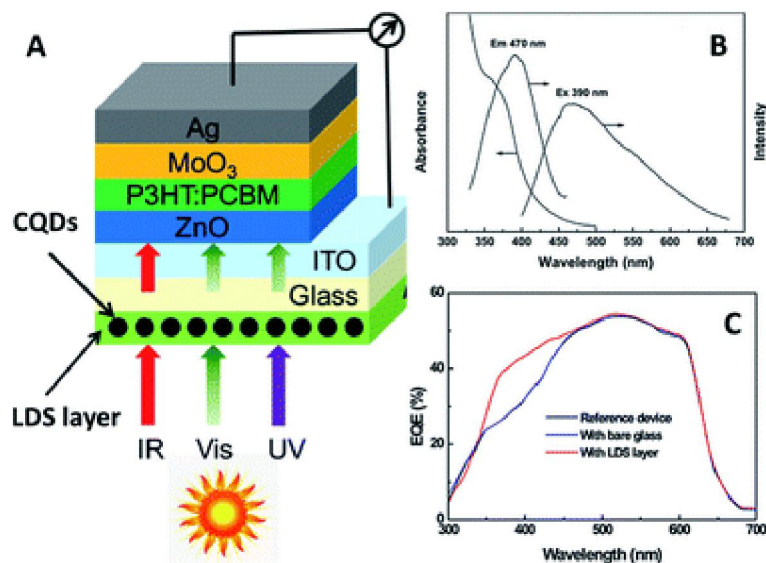


Figure 4.3 Schematic architecture of the BHJ solar cell with LDS layer. (B) UV-vis absorption and PL spectra of the CQDs filled

Polysiloxane composite (□20 μm thick) coated on glass. (C) Wavelength dependences of external quantum efficiency of the

P3HT:PCBM-based solar cell with and without the LDS layer. Adapted with permission. Copyright 2014, Elsevier.

#### 4.4 Light-emitting devices (LED)-

CQDs are an emergent material for LEDs due to their stable light emitting, low cost and eco-friendliness. Nitrogen-rich CQDs show broad and bright visible light under UV illumination that would be worth utilizing in phosphor applications.<sup>[33]</sup> Large-scale (20 × 20 cm) free-standing luminescent films of the CQDs embedded PMMA matrix were fabricated.

The polymer matrix can not only provide mechanical support but also disperse the CQDs to prevent solid-state quenching. The obtained films are cost-effective, fully flexible, easily scalable, thermally stable, eco-friendly, and mechanically robust, and they show great potential in large-scale flexible solid-state lighting systems.

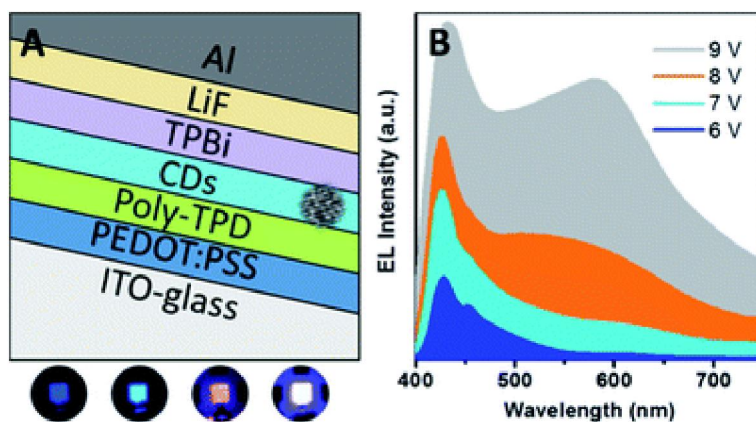


Figure 4.4 Sandwiched structure of LEDs using CQDs as a single emitting layer. (B) Electroluminescence (EL) spectra and true colour photographs of the blue, cyan, magenta, and white emissions. Adapted with permission. Copyright 2013, American Chemical Society.

#### 4.5 photo catalyst-

Advanced materials for electrocatalytic and photoelectrochemical (PEC) hydrogen evolution

reaction (HER) are central to the area of renewable energy. CQDs-modified P25 TiO<sub>2</sub> composites (CQDs/P25) were prepared *via* a facile, one-step



Hydrothermal/solvothermal reaction. <sup>[34]</sup>CQDs/P25 exhibited improved photocatalytic H<sub>2</sub> evolution under irradiation with UV-Vis and visible light ( $\lambda > 450$  nm) because of the CQDs acting as an electron reservoir to improve the efficient separation of the photo induced electron-hole pairs of P25 and a photo sensitizer to sensitize P25 into a visible light response structure for H<sub>2</sub> evolution.

**Conclusion:** carbon quantum dots (CQDs) are small carbon nanoparticles with size less than 10 nm. They were first obtained during the purification of single-walled carbon nanotubes through preparative electrophoresis. Carbon is commonly a black material, and was generally considered to have low solubility in water and weak fluorescence. Quantum dots are semiconductor nanocrystals that inherently fluoresce, at specific wavelengths in the visible range. The light emitted by these dots depends on wavelength of light used for excitation. The emission is size dependent, i.e. the smaller the size of dots the better their photoluminescence efficiency. Wide attention has been focused on carbon-based quantum dots because of their good solubility and strong luminescence, for which they are referred to as carbon Nano lights. We studied that CQD can be synthesized using various techniques and than we can also characterized CQD. Different characterization techniques give different results like size of CQD, Fluorescence nature of CQD etc.

Hydrothermal/solvothermal synthesis route based on water system is considered to be one of the simplest and most cost-effective methods owing to its cheap apparatus, simple manipulation, low energy consumption, good selectivity and preparation can be achieved in a single step without complex control. This method has been used to prepare CQDs by many researchers.

### 1.1 Proposed work

#### (a) Instruments required

Muffle furnace, tubular furnace, nitrogen cylinder, Pressure tube, silica boat, Spin coater, solar simulator, UV-lamp, Sonicate.

#### (b) Chemical and material

Nanofilter, ITO sheets, Citric acid, PSS, M-2070, PVP, TEOS, TIP, PEdotPSS, PAni, DMF, EtOH, MeOH, Isopropyl alcohol.

#### (c) STEPS

- Preparation of CQD with the help of Citric acid as precursor and polymer as dispersion media.
- Formation of polymer film and CQD over Petridis for study of optical behavior.
- Coating over ITO sheets for study of electronic properties with conducting AFM.
- Possibilities and implementation of Device preparation and applications.

#### CQD/Polymer matrix preparation

5 ml, 1 wt% CQD Solution in water is mixed with 5 ml, 1 wt% PVA in water and stirred for 20 minute in room temperature.

Then solution is dispersed on a Petri Dish and kept for evaporation of water in water bath at 80 °C for 10 hours. A very thin yellow layer is formed of uniform thickness.

#### Photo conductivity measurement

For light source **solar simulator** is used and further conductivity is measured by using an **electronic multimeter**.

#### Photo voltage Generation

Electron-hole pairs are separated by the action of a built-in electric field represented by the spatial bending of the bands. On light absorption, the energy of the photon is transferred to an electron in the semiconductor CQD, elevating it to the conduction band and leaving behind a hole in the valence.

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