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**University of Anbar**  
**College of Science**  
**Department of Chemistry**



**Bio synthesis of carbon quantum dots from lemon juice and  
study of its applications**

**A Thesis submitted to**  
**The Council of the College of Science -University of Anbar as a**  
**Partial**  
**Fulfillment of the Requirements for the Degree of Master in**  
**Chemistry**

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## **Dedication**

To my beloved father who has a good heart, May Allah give him a long life.

To my dear mother and the light of my eyes, May Allah protect her.

To the winds of my life in adversity and prosperity my brothers and sisters

To derby companion my fiancé dear

And everyone who encouraged me and helped me to accomplish this work

I dedicate this humble work.

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## **Abstract**

Carbon quantum dots (CQDs) are the latest generation of carbon nano materials in applications with several attractive properties. In this study, is used lemon as a natural source is the third Citrus species most widely cultivated world wide. Lemon fruits' health benefits are undeniable and scientifically established to help prevent cancer, cardiovascular disease, high blood glucose , cholesterol levels, obesity and has been related these benefits its high amount content of bioactive elements such as dietary fibers, polyphenols, micronutrients, vitamins, acids, mineral salts, essential oils, ascorbic acid, antioxidants substances, and antimicrobials for preparing the introduced carbon quantum dots powder (CQDs) using the one-pot simple hydrothermal method, because this method is considered safe, fast, and inexpensive when compared to chemical and physical methods, which are polluting for the environment, have high costs, and require a lot of energy to complete.

Transmission electron microscopy (TEM), Scanning electron microscope (SEM), X-ray diffraction XRD, Fourier transform infrared spectroscopy (FT-IR), ultraviolet-visible spectroscopy (UV-Vis), were utilized to explore the morphology, chemical content, and spectroscopy properties for carbon quantum dots (CQDs). Where transmission electron microscopy analysis showed that the CQDs are approximately spherical and have nearly uniform dispersion with a diameter of (2-8) nm, the images of the scanning electron microscope display nanoparticles with the average diameter (5 - 12) nm .The UV-Visible spectra showed bands around 288 nm for CQDs, and the infrared absorption spectrum showed various peaks ranging from (400 - 4000)



cm<sup>-1</sup> used to identify the functional groups responsible for reducing and capping of CQDs.

The applied of carbon quantum dots are investigated The antibacterial effectiveness of CQDs was investigated against Gram positive bacteria such as *Staphylococcus lentus*, *Enterococcus faecalis*, *Kokerococcus Kristina*, and *Staphylococcus aureus*, as well as Gram negative bacteria such as *Escherichia.coli stander*, *Escherichia coli*, and *Pseudomonas*. The results showed that CQDs exhibited better antibacterial efficiency against *Kokerococcus Kristina* (with inhibition zone was 35mm) comparing to other bacterial.

The sensitivity parameter , reaction time and recovery time of the manufactured CQDs gas sensor detector, is determined using NO<sub>2</sub> and NH<sub>3</sub> gases with different temperatures (200,250,300) ° C .The sensitivity factor (S %) at various temperatures calculated which was found to be around 79 % (200 ° C) and 22 % (300 ° C) for NO<sub>2</sub> and NH<sub>3</sub>, respectively. The detection time were equal to 23.35 s for NO<sub>2</sub> gas and 33 s for NH<sub>3</sub> gas. the recovery time for NO<sub>2</sub> was calculated as 11.93 s and the response time was 14.7s at 200 C. while the response time for NH<sub>3</sub> gas was 11.21 s and the recovery time was 6.11 s at 250 ° C. These temperatures were chosen due to the fact that the CQD sensors was optimal at these degrees.

Hydrophilic & Hydrophobic of carbon quantum dots were measured by contact angle system. The contact angle for CQDs is 9.534° reffering that CQDs have the possess high water solubility

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## List of Symbols and Abbreviations

Symbols and Abbreviation	Full name
CDs	Carbon dots
CQDs	Carbon quantum dots
GQDs	Graphene quantum dots
ASA	The advertising standards authority
PAC	Powders activated carbon
GAC	Granular activated carbon
FAC	Fibrous activated carbon
CAC	Commercialized activated carbon
CNTs	carbon nanotubes



PL	Photoluminescence
NIR	The near infrared region
QY	Quantum yield
DEG	Di ethylene glycol
HTC	Hydrothermal carbonization
PEC	Photo electrochemical
S	Sensitivity
$\tau_{res}$	Recovery time
MPED	Microwave plasma-enhanced decomposition
GP	Garlic peels
UMVD	Ultrasonic mist vapour deposition
UV	Ultraviolet
FT-IR	Fourier transform infrared
XRD	X-ray diffraction
FE-SEM	Field emission scanning electron microscopy (FE_SEM).
TEM	Transmission electron microscope

## 1. Introduction

Fabrication and use of carbon materials has a history of over 3000 years, starting with ink and pigments. From thereon, development of carbon structures has grown significantly targeting numerous applications. With the discovery of fullerene and carbon nanotubes, the versatility of carbon material has further expanded into the fields of energy and environment sectors<sup>1</sup>. Carbon nanomaterials are a novel form of carbon that has gained attention over the years owing to the unique combination of mechanical, chemical, optical, thermal and electrical properties. Thus, they have been used in different fields of environmental, biomedical and energy sectors as materials for catalyst support, fuel cells, drug delivery and gas storage<sup>2</sup>. Carbon dots (CDs) are a type of carbon-based nanomaterials in the range of 2-10 nanometers that have gained interest due to their physicochemical properties, such as tunable photoluminescence and high biocompatibility<sup>3</sup>. In addition to the direct raw materials, the synthesizing techniques of quantum dots involve chemical vapour decomposition, arc discharge and laser ablation methods which utilize catalysts composed of heavy metals like cobalt, iron, nickel and titanium. Thus, synthesis of such nano structures can involve toxic and/or costly materials and harsh operational conditions<sup>4</sup>. Therefore, developing high quantum yield CDs has become very important. Carbon dots,<sup>5</sup> are microscopic carbon nanoparticles with varied particle, surface passivation methods<sup>6-9</sup>, as first described by Sun et al. in 2006<sup>5</sup>. They are typically created by introducing defects onto the surface of the carbon core through a variety of functionalization processes, such as laser ablation of graphite, carbonization of plants, pyrolysis of small molecules, and even unusual materials. While

surface functionalization groups can be derived from a variety of polymers or monomers containing amino groups, and amino groups are preferred over other options such as hydroxyl groups because they improve the optical properties of carbon dots<sup>(6-9)</sup>. Graphene quantum dots (GQDs), on the other hand, were first synthesized by Eda et al. in 2010<sup>(10)</sup>, and theoretically are small fragments of single-layer graphene sheets or the equivalent configuration of conjugated-islands in a single-layer graphene sheet without an interlayer quenching effect, and can be derived from both carbon dots (CD) and graphene. Because of the limitations of characterization methods and the less-than-obvious connection between the two, it is not surprising that what papers refer to as carbon dots are more properly GQDs or vice versa<sup>(11)</sup>. Figure (1) shows the difference between CQDs and GQDs. Natural sources become as green precursors widely accepted, which have significant benefits in biomedicine because they are easy, repeatable, environmentally acceptable, and cost-effective for C-dot synthesis. As a result, much work has gone towards synthesizing C-dots from available natural materials. Apple juice, strawberries, and orange juices with different quantum yields can be used to make C-dots. Herein, we use nanotechnology to modify the atomic or molecular structure of lemon juice and production CQDs at an economic cost not exceeding the cost of raw materials and energy used in the process of industry<sup>(12-14)</sup>.

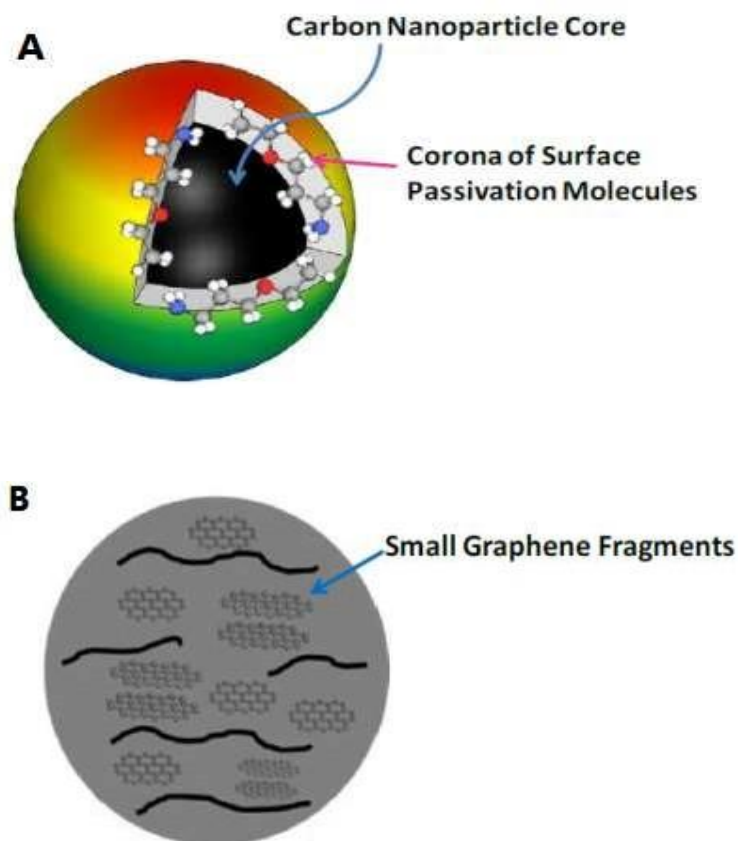


Fig.1: Sketch illustration of A- CQD, B- GQDs)<sup>15</sup>.

## 1.1 .Green Chemistry

Green chemistry is the application of a set of principles in the design, manufacture and application of chemical products and that reduce or eliminate the use of hazardous substances in their production. While green chemistry emerged in the 1990s to address the growing number of health and environmental issues caused by hazardous chemicals and substances because some of these health and/or environmental issues were the result of limited or lacking knowledge about toxicity, bioaccumulation and environmental toxicity<sup>(16)</sup>. Recently, in materials science, “green” synthesis has received a lot of attention as a dependable, sustainable, and environmentally friendly method of producing a wide range of nanomaterials, including carbon

quantum dots. Green synthesis is regarded as an important tool for reducing the harmful effects associated with traditional methods of synthesis for nanoparticles commonly used in laboratories and industry using natural extracts<sup>(17)</sup>, such as glucosamine<sup>(18)</sup>, ascorbic acid<sup>(19)</sup>, saccharides<sup>(22)</sup>, watermelon peels<sup>(21)</sup>, pomelo peels<sup>(22)</sup>, orange juice<sup>(23)</sup>, strawberry juice<sup>(24)</sup>, sugar cane juice<sup>(25)</sup>, and chicken eggs<sup>(26)</sup>. Carbon quantum dots can be synthesized from material low cost and green, such as lemon which is the third citrus species most widely cultivated worldwide. Lemon fruits' health benefits are undeniable and scientifically established to help prevent cancer, cardiovascular disease, high blood glucose and cholesterol levels, and obesity.

And has been related these benefits its high amount content of bioactive elements such as dietary fibers, polyphenols, micronutrients, vitamins, acids, mineral salts, essential oils, ascorbic acid, antioxidants substances<sup>(27)</sup>, and antimicrobials<sup>(28)</sup>. Ascorbic acid is found in abundance in lemons. Humans are unable to synthesize because they lack a functional L-Gulono-gamma-lactone oxidase enzyme, which catalyzes the biosynthetic pathway's last step. According to the advertising standards code (ASA), the requirement, which is estimated to be 90 mg per day for males and 75 mg per day for women, must be obtained through diet. Citric acid (2-hydroxy-1, 2, 3-propanetricarboxylic acid) is also abundant in lemons, it is a weak tri carboxylic acid that is naturally concentrated in citrus fruits. Citric acid is commonly used as a food additive to add acidity and a sour flavor to foods and beverages<sup>(29)</sup>.

## **1.2. Activated Carbon**

Activated carbon, also called activated charcoal, is a form of carbon processed to have small, low-volume pores that increase the surface area. Activated carbon is made from high-carbon-content trash in the environment. As raw materials for the production of activated carbons, cellulosic and coal materials have been used. Physical activation and chemical activation are the two methods for producing activated carbon that can be used in water purification operations. And because of its larger surface area, micro porous capabilities, and chemical complexity of its exterior area, activated carbon has a high potential for adsorbing heavy metals. There are two types of stimulated activated carbon: the H-type and the L-type <sup>(32)</sup>. When injected into water or treated with strong acids, H-type carbon acquires positive charges and is classified as hydrophobic. The L-type carbon is a stronger solid acid than the H-type carbon, which has a negative charge in water and can neutralize strong bases and hydrophilic compounds. Activated carbon is classified into four types based on its physical appearance. Powders (PAC), granular (GAC), fibrous (FAC), and cloth are all examples of materials (CAC). Commercialized activated carbon (CAC) is now widely used all over the world <sup>(31)</sup>.

## **1.3. Nanotechnology**

Nanotechnology refers to any technology that is implemented at the nano scale and has real-world applications. It is defined as the control or restructuring of matter at the atomic and molecular levels in the 1 to 100 nm size range. Where the properties of matter at the nano scale are different from those at greater scales. When a material's

dimensions are lowered from a big size, its qualities remain constant at first, then alter somewhat. When the size is reduced to less than 100 nm, dramatic changes in properties can occur. Nanotechnology includes the creation of nano materials, the application of physical, chemical, and biological systems at scales ranging from individual atoms or molecules to submicron dimensions, and the incorporation of the resulting nanostructures into larger systems. It has had an impact on society and the economy comparable to that of semiconductor technology, computer technology, and cellular and molecular biology in the twentieth century. Many current environmental, medical, and industrial problems have been addressed by incorporating nanotechnology into larger systems, such as smart materials, nanomanufacturing, electronics, drug delivery, energy and water, biotechnology, information technology, and national security. Nanotechnology will have a large impact on our economy and society. It symbolizes the modern industrial revolution. It has progressed into a general-purpose technology with applications in a wide range of industrial sectors Figure (1.2) nano-scale of some materials in nature (32).

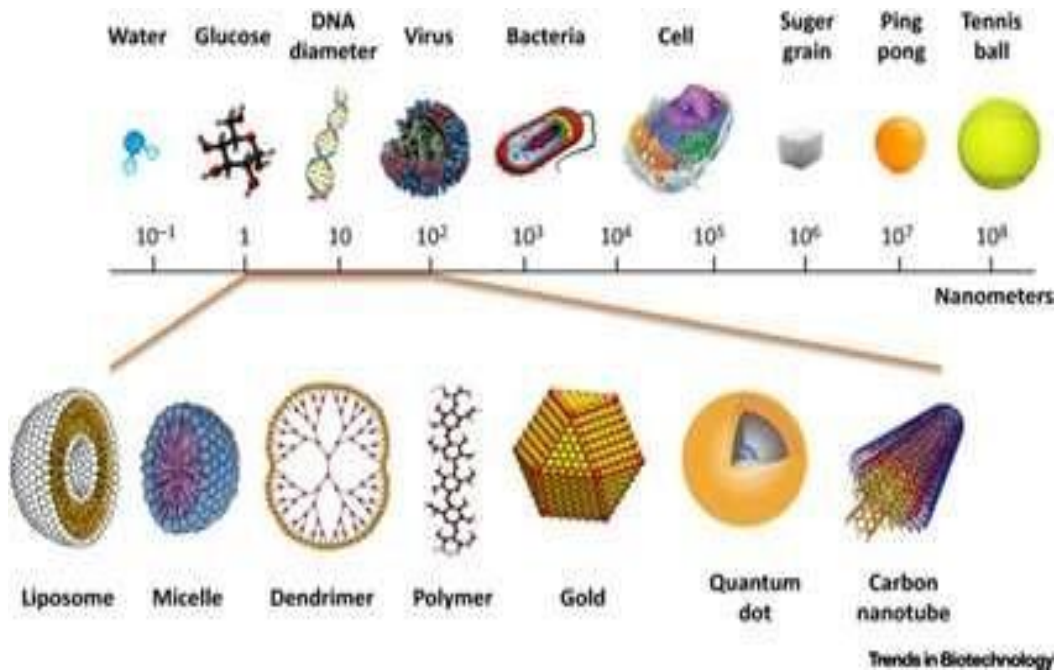


Figure 1.2: Nano-scale of some materials in nature <sup>(33)</sup>.

## 1.4. Carbon nanostructures

Materials with structural features (clusters, crystallites, or molecules) with dimensions in the range of (1-100) nm are known as nano structured carbon-based materials. All allotropic carbon changes are generated on the nano scale, regardless of how they are made. The term "nanostructured modification of carbon" is well-established in the chemical and physical literature, and it is frequently applied to fullerenes, nanotubes, and other carbon-based structures <sup>(34)</sup>. Scientists are intrigued by carbon nanostructures because of their unusual physical, chemical, optical, and electrical capabilities, as well as their bio compatibility. They are successfully used in sensing, biomedical, and biological applications. Electronic properties and electron transport of carbon nanostructures are examples of unusual properties of carbon. Carbon nanostructures embrace a wide variety of carbon allotropes and a large number of shapes and sizes development of new



carbon nano-forms has its origin in the discovery of fullerenes thirty years ago by H. Kroto, R. Smalley, and R. Curl who received the Nobel Prize in 1996 for this discovery thus opening the race for the discovery of other amazing carbon nanostructures such as carbon nanotubes (CNTs), graphene and carbon dots (C-dots), as well as other less-explored carbon nano forms such as graphene quantum dots (GQDs), nano diamonds, carbon nano onions, and that exhibit huge diversity in structure<sup>(35)</sup> .

## **1.5. Classified of carbon nanomaterials**

Carbon nanostructures are classified based on the number of dimensions that are not restricted to the nano scale range, i.e. Zero-dimensional (0D) nano materials such as fullerene, carbon quantum dots<sup>(36)</sup> , one-dimensional (1D) nano particles such as nanotubes and two-dimensional (2D) such as graphene is layered materials<sup>(35)</sup> , and three-dimensional (3D) nano materials such as the nanostructured bulk<sup>(37)</sup> (Figure. 1.3). So:

- a.** All dimensions of zero-dimensional (0D) nano materials are measured at the nanoscale (no dimensions are larger than 10 nm). Where quantum dots are the most prevalent zero-dimensional nanomaterial.
- b.** Two dimensions are outside the nano scale in two-dimensional nano materials (2D). Examples of this class Graphene, nano films, nano layers, and nano coatings, which have plate-like structures.
- c.** Nanomaterials that are three-dimensional (3D) are materials that are not bound by the nanoscale in any dimension. This category includes bulk powders, nanoparticle dispersions, nanowire bundles, and multi-nanolayers.

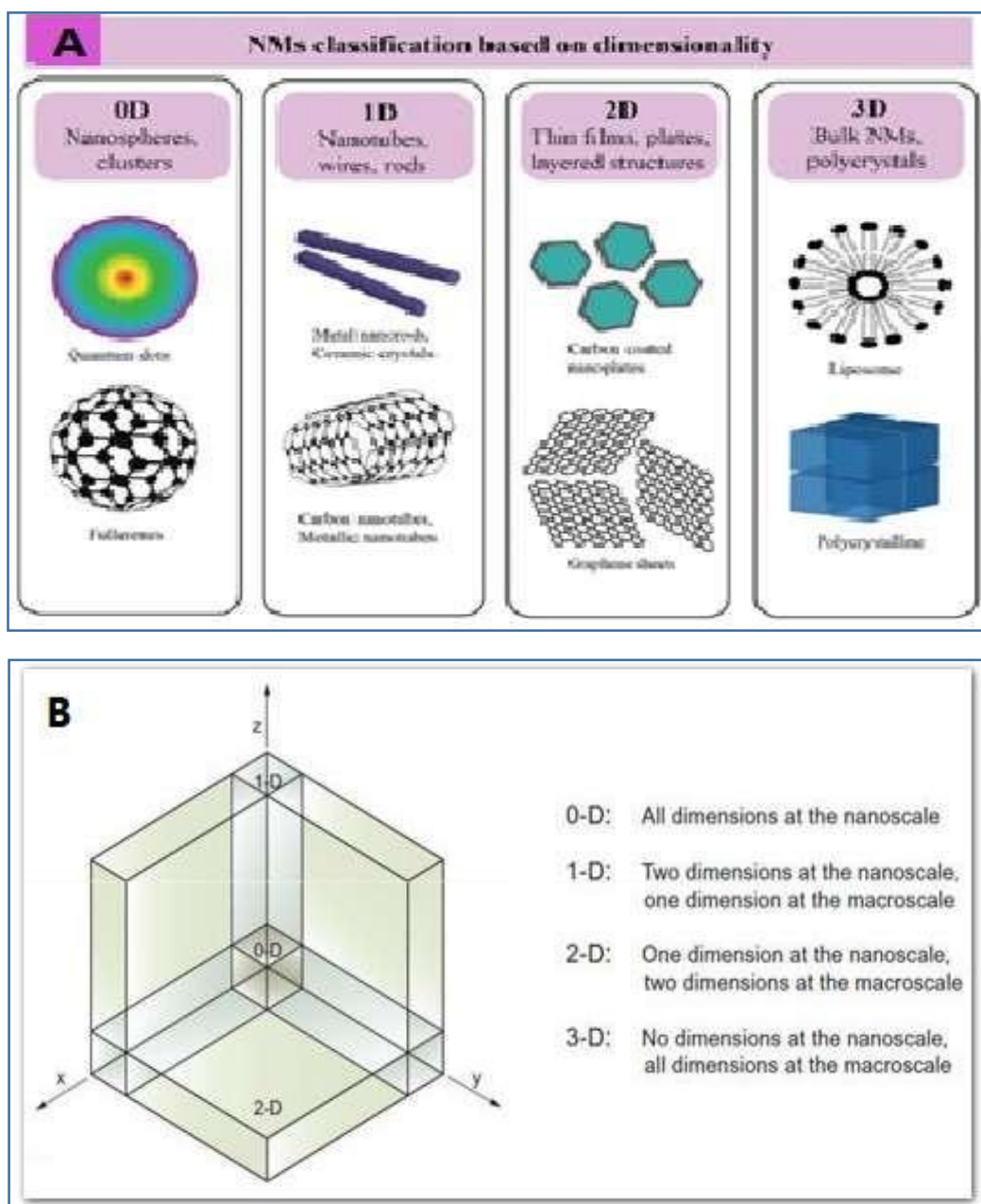


Figure 1.3: (A) Classification of carbon nanostructure with examples of each category and (B) of nano scale dimensions <sup>(38)</sup>.

## 1.6. Carbon quantum dots

Carbon dot (C-dots) is a comprehensive term for several nanosized carbon materials and also known as carbon quantum dots. Basically, all nanosized materials that consist of mainly carbon skeleton can be called C-dots. C-dots always possess at least one dimension smaller than 10 nm in size and fluorescence as its characteristic properties. Carbon quantum dots, often known as carbon dots, have earned a huge name in the field of materials research. Unlike semiconductor quantum dots like C-dots Te and C-dots Se, which have some toxicity, where C-dots have emerged as a new advancement in medicine and theranostics due to their exceptional biocompatibility, typical optical properties, nontoxic precursors as carbon sources, high aqueous solubility, and easy surface passivation. Another enticing feature of C-dots is their photoluminescence (PL) in the near infrared region (NIR), which suggests that they could be used to treat malignancies using photothermal therapy<sup>(39-41)</sup>. Figure 1.4 shows the chemical structure of CQDs.

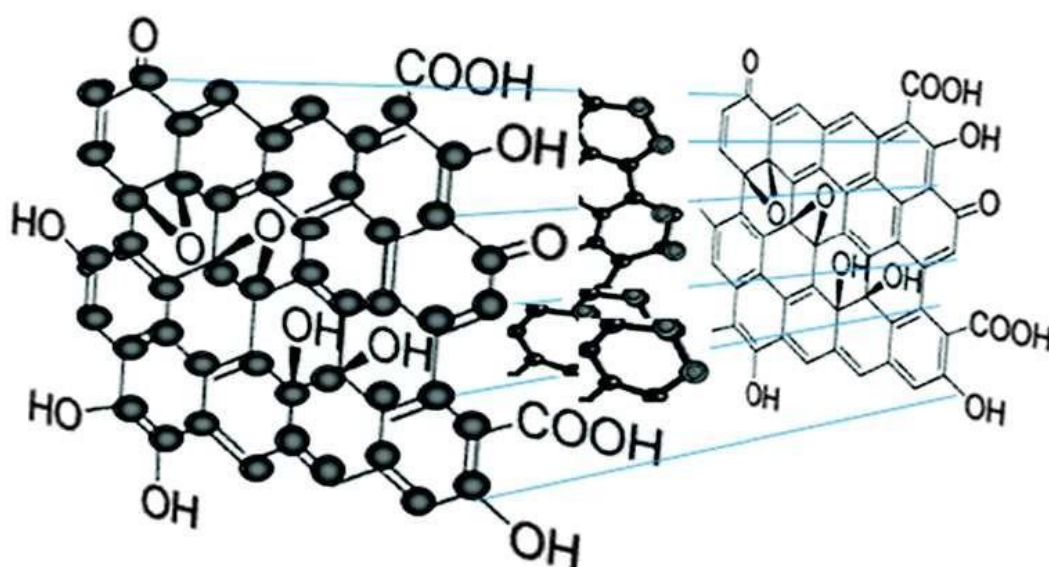


Figure 1.4: Chemical structure of CQDs<sup>(42)</sup>.

## 1.7. Synthesis of carbon quantum dots

Many methods for preparing CQDs have been presented over the last decade, which can be classified as "Top-down" and "Bottom-up" approaches, and they can be adjusted during preparation or post-treatment, as shown in figures. (1.5) and (1.6) .When preparing for CQDs, there are three things to keep in mind <sup>(43)</sup>:

(i) Carbonaceous aggregation can be avoided during carbonization by employing electrochemical synthesis, restricted pyrolysis, or solution chemistry techniques.

(ii) Post-treatments such as gel electrophoresis, centrifugation, and dialysis can improve size control and uniformity, which is important for homogeneous properties and mechanistic studies.

(iii) Surface properties that are critical for solubility and specific applications.

Which can be adjusted during or after therapy.

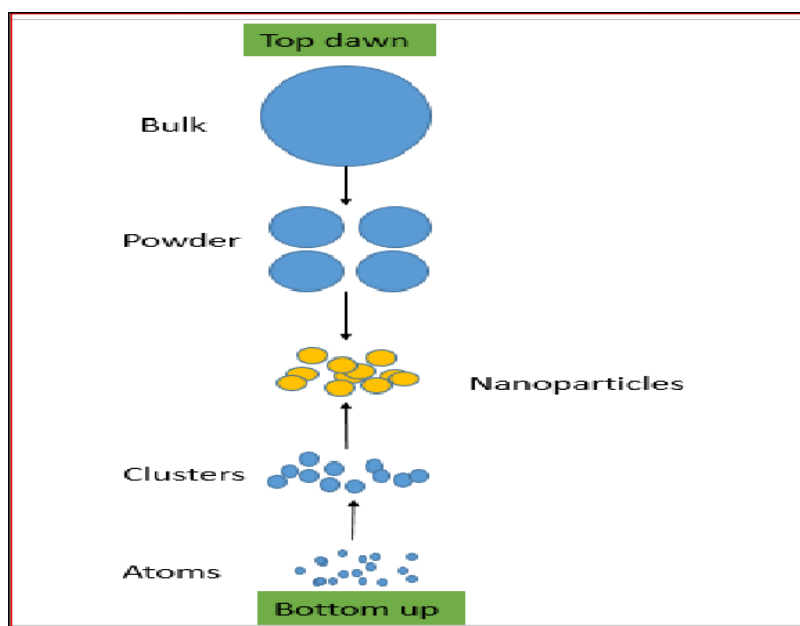


Figure 1.5: Procedures employed in the synthesis of nanoparticles using bottom up and top down process <sup>(44)</sup>.

### 1.7.1 Bottom-up methods

Bottom-up, or self-assembly, approaches to nanofabrication use chemical or physical forces operating at the nano scale to assemble basic units into larger structures. Inspiration for bottom-up approaches comes from biological systems, where nature has harnessed chemical forces to create essentially all the structures needed by life. In the bottom-up method, C-dots are normally formed in three steps:

- i. Carbonaceous aggregation during carbonization, which can be avoided through electrochemical synthesis, limited pyrolysis, or solution chemistry.
- ii. Size control and uniformity, which are critical for uniform properties and mechanistic studies and can be improved with post- treatments such as gel electrophoresis, centrifugation, and dialysis.
- iii. Surface properties that are important for solubility and specific applications and can be adjusted during or after preparation.

Bottom-up methods are simpler to implement and can be utilized to produce CQDs on a wide scale. Bottom-up methods rely in the synthesis for CQDs on the use of molecular precursors, such as polymerization and carbonization of carbon-containing molecules [like natural materials and agricultural by-products<sup>(45,46)</sup>].

### 1. Irradiation by microwaves

Microwave irradiation of organic compounds is a quick and low-cost method of producing CQDs. Where green luminous CQDs were produced in one minute under microwave irradiation using sucrose as the carbon source and di ethylene glycol (DEG) as the reaction media. DEG-stabilized CQDs (DEGCQDs) might be widely diffused in water and appear transparent<sup>(47)</sup>.

## 2. Hydrothermal/ Solvothermal treatment

Hydrothermal carbonization (HTC) <sup>(48)</sup>, also known as solvothermal carbonization, is a low-cost, environmentally friendly, and nontoxic method for synthesizing new carbon-based compounds from a variety of precursors. Typically, an organic precursor solution is enclosed and reacts at high temperature in a hydrothermal reactor. CQDs were created using a variety of precursors, including glucose <sup>(49)</sup>, citric acid <sup>(52)</sup>, chitosan <sup>(51)</sup>, banana juice <sup>(52)</sup>, and protein <sup>(53)</sup>. Mohapatra et al. used hydrothermal treatment of orange juice followed by centrifugation to produce highly photo luminescent CQDs with a QY of 26% in a single step <sup>(54)</sup>. Because of their high photo stability and low toxicity, these CQDs with diameters of 1.5–4.5 nm were used in bio imaging. Liu et al. described a one-step synthesis of amino-functionalized fluorescent CQDs by hydrothermal carbonization of chitosan at 180C for 12 hours <sup>(55)</sup>. It's worth noting that the amino-functionalized fluorescent CQDs can be used right away as new bio imaging agents. Solvothermal carbonization followed by extraction with an organic solvent is a common method for preparing CQDs <sup>(56)</sup>. Heat treatment in high-boiling-point organic solvents was followed by extraction and concentration of carbon-yielding compounds. Bhunia et al. created two types of CQDs from carbonized carbohydrates: hydrophobic and hydrophilic with diameters less than 10 nm. The hydrophobic ones were created by combining different amounts of carbohydrate with octadecyl amine and octadecene, then heating them for 10–30 minutes at 70–300 C. The hydrophilic ones are created by heating a carbohydrate aqueous solution over a wide pH range. Hydrophilic CQDs with yellow and red emissions are produced by

mixing an aqueous carbohydrate solution with concentrated phosphoric acid and then heating at 80–90 C for 60 minutes <sup>(57)</sup>.

### **3. Ultrasonic-assisted method**

The ultrasonic-assisted method for C-dot synthesis has the advantages of being low-cost and easy to use. C-dots can be created by ultrasonically treating a solvent and carbon source mixture. Modifying experimental conditions such as ultrasonic power, reaction time, solvent and carbon source proportions, and so on can control the properties of C-dots. Park *et. al.* A simple ultrasonic treatment-based approach for large-scale synthesis of water-soluble C-dots from food waste-derived carbon sources was described, and 120 g C-dots were synthesized from 100 kg of food waste mixes. For in vitro bio imaging, the generated C-dots have high water solubility, photo stability, photoluminescence, and low cytotoxicity. Furthermore, byproducts produced during the synthesis of C-dots from food waste may aid in seed germination and plant growth <sup>(58)</sup>.

#### **1.7.2 Top-down methods**

The bulk material is broken down into nano-sized structures or particles in a top-down method. Top-down synthesis techniques are expansions of micron-sized particle production techniques. Top-down techniques are naturally simpler, relying on bulk material removal or division, or bulk fabrication process downsizing, to produce the required structure with sufficient attributes. The most significant flaw in the top-down method is the inaccuracy of surface structure. CQDs prepared by a top-down method exhibited superior light-activated cell cytotoxicity<sup>(59)</sup>. In the top-down approach, CQDs are synthesized by breaking the large carbon materials into smaller ones<sup>(63)</sup>. such as

graphite<sup>(62)</sup> , candle soot<sup>(61)</sup> , carbon rod<sup>(62)</sup>.by employing the arc discharge<sup>(63)</sup> laser ablation<sup>(64)</sup> , chemical oxidation<sup>(65)</sup>.

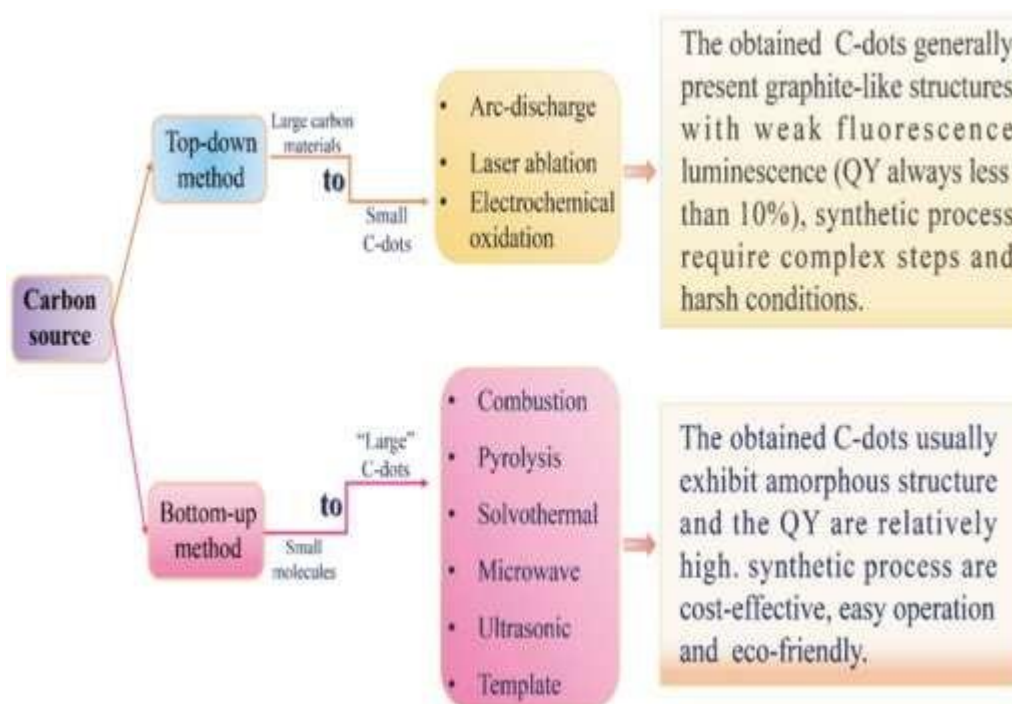


Figure 1.6: The top-down and bottom-up methods to C-dot synthesis (QY refers to fluorescence quantum yield) <sup>(66)</sup>.

## 1. Electrochemical carbonization

Electrochemical soaking, which uses various bulk carbon sources as precursors, is a powerful method for producing CQDs <sup>(67)</sup>. However, electrochemically carbonizing tiny compounds to CQDs has only been reported in a few reports. Zhang and colleagues propose making CQDs by electrochemically carbonizing low-molecular-weight alcohols <sup>(68)</sup>. The working and auxiliary electrodes were constructed from two pt sheets, while the reference electrode was a calomel electrode set on a freely adjustable luggin capillary. The alcohols were converted into CQDs after electrochemical carbonization under basic conditions. The sizes and graphitization degrees of these CQDs increase as their applied potential increases.



The resulting amorphous core CQDs have excellent excitation- and size-dependent PL properties without the need for time-consuming purifying and passivation operations. It's worth noting that the quantum yields (QYs) of these CQDs can reach 15.9 percent. CQDs can be created using a variety of tiny molecular alcohols that are non-toxic to human cancer cells <sup>(69)</sup>.

## **2. Ablation by laser**

Sun and colleagues created CQDs by laser ablation of a carbon target at 900 C and 75 kPa in the presence of water vapour and argon as a carrier gas <sup>(70)</sup>. The synthesis of fluorescent CQDs by laser irradiating a suspension of carbon materials in an organic solvent was described by Du *et al*, <sup>(71)</sup>. By using organic solvents, the surface states of the CQDs can be adjusted to provide variable light emission. Based on control measurements, the origin of the luminescence was attributed to surface states associated with the ligands on the surface of the CQDs. Li *et al.* proposed a simple laser ablation approach to manufacture CQDs using nano-carbon materials as the starting material and a simple solvent as the liquid medium. In a typical process, 0.02g of nanocarbon material was dispersed in 50 mL of solvent (such as ethanol, acetone, or water). After ultrasonication, 4 mL of the suspension was placed in a glass cell for laser irradiation. An ND: YAG pulsed laser with a second harmonic wavelength of 532 nm was used to irradiate the suspension. After laser irradiation, the solution was centrifuged to obtain the supernatant containing the CQDs <sup>(72)</sup>.

### **3. The arc discharge**

CQDs are made by rearranging carbon atoms obtained from decomposition of bulk carbon precursors in the anodic electrode as a derivation of gas plasma created in a sealed reactor employing an arc discharge. As a result, using electric current, the temperature in the reactor could be raised to around 4000 K, allowing for the generation of high-energy plasma. Finally, CQDs were created utilizing the arc discharge method <sup>(73)</sup>.

### **1.8. Applications of carbon quantum dots**

Carbon Quantum Dots have great applications in different fields due to their ease of functionalization and inexpensive synthesis. These applications can be classified as follows:

#### **1.8.1 Applications in biomedicine and biotechnology**

Carbon dots, a new member of the carbon family with a size less than 10 nm, have piqued the interest of researchers in recent decades due to their unique properties, which include simple and inexpensive synthetic methods, low toxicity, the ability to modify the surface, excellent photoluminescence, and excellent water solubility <sup>(75)</sup>. Because of their unique properties, carbon dots have been widely used in a variety of scientific fields. The primary properties of carbon dots are phosphorescence and fluorescence, which make them ideal for in vivo and in vitro bio sensing and imaging. Drug delivery, photocatalytic reactions, photodynamic, and photo thermal therapy could all benefit from it. However, some issues arise from the use of metal ions in carbon dots, which are typically poisonous and environmentally harmful, whereas classic carbon dots are non-toxic and significantly safer, allowing for good biological and environmental compatibility

<sup>(75)</sup>. It can also be used as an antimicrobial agent. CQDs can successfully interact with a variety of viruses to prevent infection <sup>(76)</sup>. CQDs containing amino groups or boronic acid, for example, may alter the entrance of the herpes simplex virus type 1. These CQDs can be utilized to combat one of today's most dangerous pathogenic human illnesses (corona virus). It could be due to human coronavirus-229E entry inhibition, which is caused by CQD boronic acid activities interacting with the HCoV229E S protein via pseudo-lectin-based interactions. The findings assist scientists' efforts to replace antiviral drugs currently in use (e.g., interferons and ribavirin). These agents had a variety of adverse effects, including short-term memory loss, decision-making inhibition, disorientation, and extrapyramidal symptoms. More effort should be put into examining clinical trials in depth for materials that could be used as therapeutic candidates, as this is one strategy to combat complex and life-threatening disorders <sup>(77)</sup>. CQDs have been utilized as an antibiotic against a variety of bacteria, including *Pseudomonas aeruginosa*, *E. coli*, and *Staphylococcus aureus*, as well as for microbe imaging <sup>(78)</sup>.

### **1.8.2. Diodes that emit light**

In a variety of applications, CDs and CQDs have the potential to replace phosphor-based white light emitting diodes. Quantum dot-based LEDs have higher efficiency, higher quantum yield, better stability, and lower toxicity than phosphor-based LEDs that use rare earth elements and common metal components (e.g., cadmium and lead). As a result of these fascinating properties, researchers have studied strategies to employ CDs and GQDs for both phosphor-based and electroluminescence-based LEDs <sup>(79,80)</sup>.

### 1.8.3. Flexible and wearable electronic devices

Because of their compatibility with flexible substrate methods, GQDs and CQDs have been successfully used in the fabrication of a wide range of flexible electronics, including super capacitors, photo sensors, nonvolatile memories, and light emitting (or electro chromic) devices. CQDs have lately been found to be widely used in advanced super capacitor applications. CQD anchoring to nanostructures of traditional super capacitor electrodes has proven to be a successful method of improving super capacitor performance <sup>(81,82)</sup> .

### 1.8.4. Hydrogen fuels

Producing hydrogen fuel from renewable sources has remained a challenge in meeting global energy demands. The generation of hydrogen fuel from green CQDs and GQDs is a promising method of producing clean energy. Because of their abundance of elements and resistance to photo bleaching, CQDs and GQDs are intriguing alternatives to standard semiconductor quantum dots. Because of their broad and customizable absorption ranges and high absorption coefficients, CQDs and GQDs are extremely useful in the fabrication of photo electrodes for photo electrochemical (PEC) hydrogen generation via water splitting. Several hetero structures containing CQDs and GQDs have been attempted, and when combined with one-dimensional nanostructures such as TiO<sub>2</sub>, Si, ZnO, and Cu<sub>2</sub>S nano rods, these provided distinct advantages. CQD-decorated hetero junctions were used for PEC water oxidation <sup>(83)</sup>. In contrast, Zeng *et al.* described the layer-by-layer assembly of nitrogen-doped GQD monolayers on 1D ZnO nano rods for PEC water splitting. The intrinsic negatively charged surface of hydrophilic N-GQDs was critical in their growth on hydrophilic ZnO nanowire surfaces <sup>(84)</sup>.

### **1.8.5. Solar cells**

Quantum dot solar cells (QDSCs) are emerging as a viable candidate due to the unique and adaptable properties of quantum dots (QDs), such as variable band gap and high absorption coefficient. Quantum dots are widely used in QDSCs as sensitizers<sup>(85)</sup>. Carbon dots, an emerging carbon-based nanomaterial, have been actively researched and used in a variety of industries<sup>(86)</sup>, because of their distinctive optical and electrical properties, water solubility, high biocompatibility, low toxicity, and chemical inertness<sup>(87)</sup>. One of the CD applications is being investigated as a QDSC sensitizer. Mirtchev *et al.* Used water soluble CDs as sensitizers for QDSCs, yielding a power conversion efficiency (PCE) of 0.13%<sup>(88)</sup>.

### **1.8.6. Gas Sensor**

Electronic devices that detect and identify various types of glasses are known as gas sensors (also known as gas detectors). They can also be characterized as devices that respond reversibly and quantitatively to the presence of a gaseous analyte by altering the physical characteristics of the sensor while being monitored by an external instrument. They're typically employed to detect and monitor dangerous or explosive gases. Gas sensors are used to detect smoke and carbon monoxide in houses, as well as to detect gas leaks in factories and manufacturing facilities. Advances in gas sensing devices are critical for reducing air pollution and controlling human health, where Semiconductor gas sensors have been developed. Quantum dots, which have electron-hole pairs confined in all three dimensions, offer new insights into material properties. Quantum dot chemical sensor research has emerged as one of the most rapidly expanding areas of modern sensing technology. Quantum dot structures have

shown promising sensing capability, implying that they are evolving into a new class of materials for use in chemiresistive devices. In contrast, QD-based structures are only now being integrated into monitoring systems. The findings suggest that more research is needed to fully understand the impact of synthesis techniques and added materials on quantum dot sensing capability. Furthermore, because the band gap in quantum dots changes with size, the response and selectivity of the materials should be investigated<sup>(89)</sup>, CQDs are employed as a sensitive part because of their physical and chemical qualities. It was also utilized as a dopant to boost the sensor's performance<sup>(90)</sup>. A simple drop-casting deposition process was used to create a GQD synthesis employing hydrothermal method based optical gas sensor. For 1000 ppm CO<sub>2</sub> gas, the predicted gas sensor response in the wavelength of 310 nm was roughly 50%. When GQDs sensing films exposed to CO<sub>2</sub> gas at room temperature, displayed a significant optical absorption shift, which was consistent with our earlier theoretical analysis<sup>(91)</sup>. When doing any gas sensor experiment, the following are the average parameters to consider:

### **1.8.6.1. Sensitivity**

The sensitivity (S) of a sensor is its response to the introduction of a certain gas species. The most general definition of sensitivity used to solid state chemiresistive gas sensors when exposed to a reducing or oxidizing gas component is a change in electrical resistance (or conductance) compared to the original state. The background gas composition, relative humidity level, sensor temperature, oxide microstructure, film thickness, and gas exposure period are all elements that affect sensitivity<sup>(92)</sup>. Equations (1.1) and (1.2) indicate a popular way of reporting S<sup>(93)</sup>:

$$S = \frac{[\Delta R]}{R^\circ} \times 100\% = \frac{R_{gas} - R_{air}}{R_{air}} \times 100\% \quad \dots\dots (1.1)$$

$$S = \frac{[\Delta G]}{G^\circ} \times 100\% = \frac{G_{gas} - G_{air}}{G_{air}} \times 100\% \quad \dots\dots (1.2)$$

Where R represents electrical resistance and G represents electrical conductance, and the subscript (air) represents the initial dry air state, and the subscript (gas) represents the introduction of the analytic gas.

### **1.8.6.2. Response and recovery times**

A gas sensor's response time ( $\tau_{res}$ ) is defined as the time it takes for the sensor to achieve 90% of its maximum/minimum conductance value after the reducing/oxidizing gas is introduced <sup>(94)</sup>. Similarly, when the flow of reducing or oxidizing gas is removed, the recovery time ( $\tau_{res}$ ) is defined as the time required to return to within 10% of the original baseline <sup>(95)</sup>.

### **1.8.7. Hydrophilic and hydrophobic**

The Merriam-Webster dictionary defines hydrophilic as “of, pertaining to, or having a great affinity for water.” This refers to a substance's capacity to mix well, dissolve, or be drawn to water. While hydrophobic is described as “resistant to or preventing wetting” by the same definition. Hydrophobic is opposite of hydrophilic. When liquids come into touch with hydrophobic substances, water is urged to bead up and roll off the surface, practically forcing the item away like a magnet does with metal objects. Self-cleaning glass is an excellent example of anything that is hydrophilic. This unique glass has been created and thinly covered with a nanoscale coating. Rather than

allowing water to condense into water droplets that roll off the glass, this innovative nanotechnology allows tiny water molecules to glide across the surface in a sheet, cleaning dirt and other debris away. The wettability of a material's solid surface is determined by its contact angle. Contact angles are influenced by a variety of factors, including surface geometry, roughness, contamination, and deformation. With such sensitivity, this very macroscopic measurement can detect impacts on a very small scale. It is the angle formed by the tangent to the solid surface and the tangent to the liquid-fluid interface at the contact line of the three phases on the liquid side (the denser liquid side if there are two liquids). The contact point, as shown in (Figure 1.7), is the place where two tangent lines connect.

There are many different types of contact angles that can be used to illustrate both equilibrium and non-equilibrium situations (Figure 1.8):

- Young contact angle: The contact angle that is calculated from the description of the Young equation.
- Ideal contact angle: The contact angle on an ideal surface.
- Actual (local) contact angle: The contact angle that exists locally at any point along the contact line.
- Apparent (global) contact angle: The macroscopic contact angle as measured empirically.

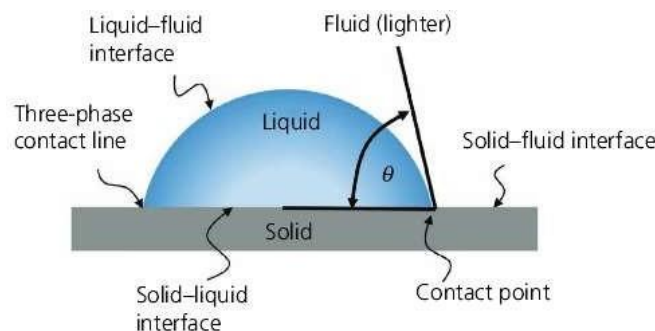


Figure 1.7: Represents the contact angle ( $\theta$ ) of a sessile drop.



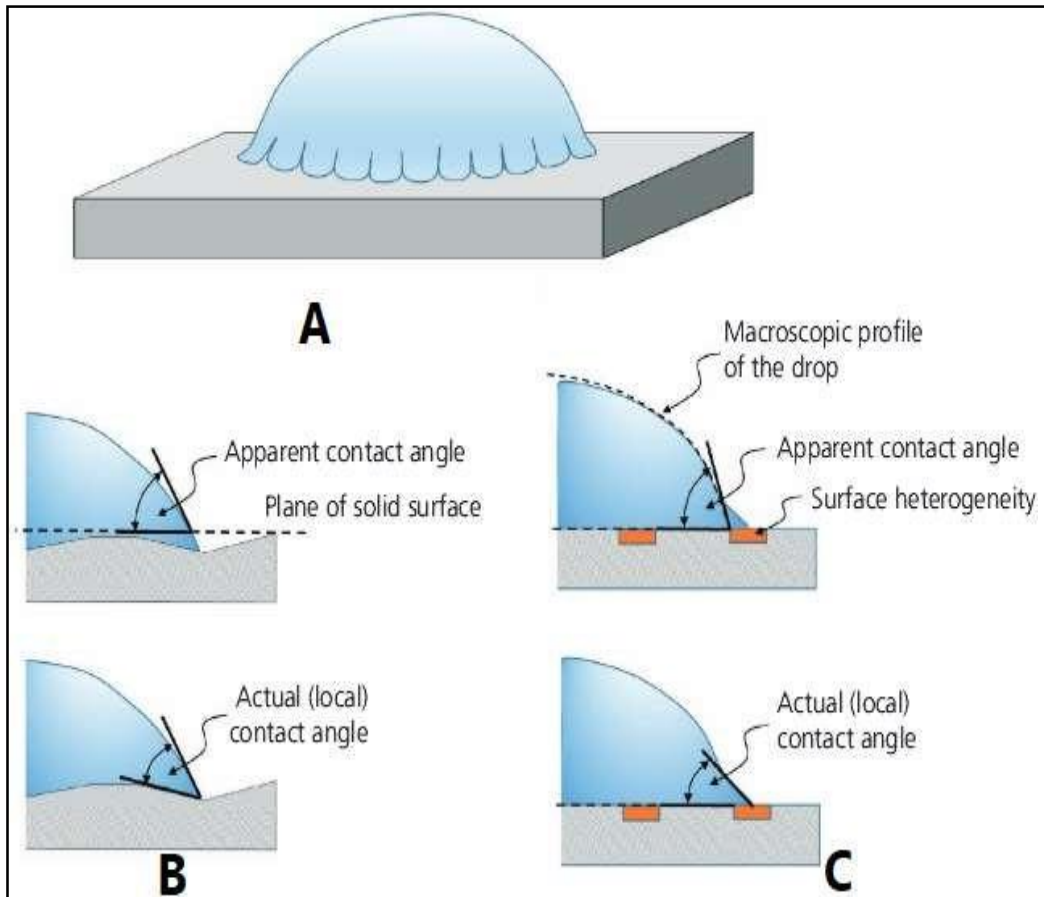


Figure 1.8: A hypothetical liquid drop on real solid surface; A) illustration of corrugated drop base; B) apparent and actual contact angles on a rough surface; C) apparent and actual contact angles on a heterogeneous surface<sup>(96)</sup>.

For a smooth surface, follow these steps:

- Hydrophilic surface – characterized by Young contact angle for water that is smaller than 90 degrees
- Hydrophilic surface: Any liquid with a Young contact angle less than  $90^\circ$  is considered hydrophilic. (Figure 1.9).

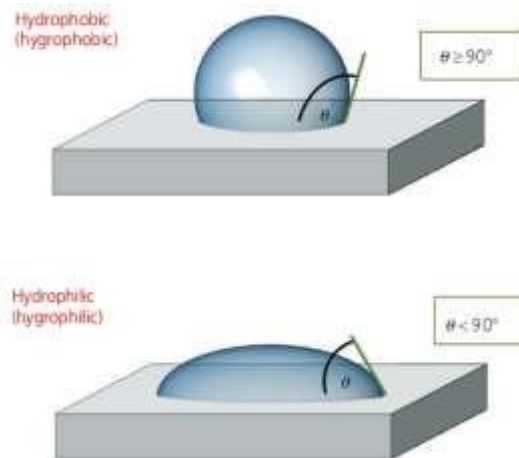


Figure 1.9: A drop of liquid on a hydrophobic (hydrophobic) and a hydrophilic (hydrophilic) surface.

## 1.9. Literature survey

In 2018, Nandhini Arumugam and Jongsung Kim used hydrothermal treatment to make fluorescent carbon quantum dots (CQDs) from broccoli juice for selective detection of Ag<sup>+</sup>, with a limit of detection (LOD) of 0.5M, showing that the synthesized CQDs can be used as an Ag<sup>+</sup> sensing instrument <sup>(97)</sup>.

In 2021, Li, Z, *et.al*, studied the Green formation of carbon quantum dots from agricultural waste without any addition of chemical reagent such as corn stalk shell by hydrothermal approach in near-critical water. The as-prepared CQDs can be employed in the detection and bioimaging domain <sup>(98)</sup>.

In 2020, Dager, A, *et.al*, produce CQDs used Microwave plasma-enhanced decomposition (MPED) process was used to produce CQDs. The result showed exploitation to make the luminescent bio-nano hybrid systems. Which includes conjugation lysozyme protein crystal with CQDs <sup>(99)</sup>.

In 2018, Amin, N,*et.al*, The production of carbon quantum dots (CQDs) from date kernels without the need of chemical reagents was investigated using a hydrothermal treatment method. The significant affinity of phosphate groups for Fe<sup>+3</sup> was used for it was applied for green photoluminescence on-off-on sensors. The detecting system was built by quenching the fluorescence of N-CDs with Fe<sup>+3</sup> via electron transfer, and then inverting the quenching action with Fe<sup>+3</sup> to ZA. In addition, utilizing osteoblast-like cells MG-63 as a model system, the biocompatibility of N-CDs produced from date kernel was tested <sup>(100)</sup>.

In 2021, Hu, X., *et.al*, Carbon quantum dots (CQDs) from orange peel were prepared by microwave method for fluorescence detection of *Escherichia coli* in milk, which classify as one of the most dangerous food borne pathogens, even at a very low concentration via milk where The obtained result showed fluorescent intensity of the CQDs-MNPs was decreased with addition of *E. coli* <sup>(101)</sup>.

In 2019, Qi, H., *et.al*, investigated nitrogen-doped carbon quantum dots (N-CQDs) produced from rice residue and glycine as carbon and nitrogen sources using a one-step hydrothermal method. The results of TC using fluorescence method were in good agreement with the standard Ultraviolet–visible (UV–VIS) approach in real water samples <sup>(102)</sup>.

In 2017, Cheng, C., *et.al*, produce CQDs was used natural cellulose and tested with live-cell fluorescent images for the potential intracellular probes. Raman mapping technique was also applied to detect the CQDs' distribution in vitro and clarify the entering procedure of CQDs into cells besides confocal microscopy <sup>(103)</sup>.

In 2020, Chaudhary, N., *et.al*, examined the highly fluorescent carbon quantum dots from banana juice using a simple one-step hydrothermal technique that did not include any surface passivation, oxidizing agent, or inorganic salt. With this system, copper ions in water can be identified selectively. The built fluorescence sensor, which had a LOD of 0.3 g/mL, identified copper ions in the concentration range of 1800 g/mL <sup>(104)</sup>.

In 2017, Carbon quantum dots were created from natural chia seeds by Jones, S. S., *et al.* Its carbohydrate composition, which includes carbon, hydrogen, and oxygen, suggests that it might be used as a bulk carbon source for bottom-up C-dot synthesis utilizing heat treatment alone. A green approach was developed for the scalable synthesis of carbon dots with long-range emission for use in bio imaging, photo thermal therapy, and multifunctional sensing systems <sup>(105)</sup>.

In 2017, Harroun, S. G., *et.al*, synthesizes carbon quantum dots from polyamines as the initial raw natural organic material and by applying simple pyrolysis methods. CQDs showed enhanced antimicrobial activity against tested Gram-positive and Gram-negative bacteria <sup>(106)</sup>.

In 2019, Khan, Z. M., *et al* investigated nitrogen doped fluorescent carbon quantum dots (N-CQDs) from red lentils. The sensor's stability was evaluated under the influence of ionic concentration and pH, and the outcome demonstrates this. It shows that the sensor is stable in high-salt conditions and that pH has no effect on it. This type of sensor has the potential to play a big role in controlling iron levels and monitoring human health <sup>(107)</sup>.

In 2020, Boruah, A., *et al.* From waste biomass sources such as sugarcane bagasse (SCB), garlic peels (GP), and taro peels, blue-emitting luminous carbon quantum dots were created using an ultrasonic assisted wet-chemical oxidation technique (TP). Anusuya Boruah and colleagues tested three CQDs generated from three distinct biomass wastes in a comparative study. The CQDs were found to be

extremely water soluble, with a strong blue fluorescence under UV light and a quantum yield of approximately 4-27 percent <sup>(108)</sup>.

In 2019, Yahyazadeh, E., *et al.* To make the product, a simple thermal process was used. Carbon dots were created from citric acid and glycine precursors, and they were utilized to assess mercury (II) levels in water samples. For the determination of Hg (II) ions, a sensor was constructed and optimized. The sensor had a high selectivity for Hg (II) ions and was effectively utilized to measure Hg (II) ions in mineral water samples <sup>(109)</sup>.

In 2020, Pandiyan, S., *et al.* To make carbon quantum dots from sugarcane bagasse pulp, surendran pandiyan and colleagues employed a hydrothermal technique. CQDs had a quantum yield of 17.98%, and their antibacterial activities were tested in aquatic conditions against Gram-positive (*Benthesicymus cereus* and *Staphylococcus aureus*) and Gram-negative (*Pseudomonas aeruginosa*, *Vibrio cholerae*, and *Escherichia coli*). The findings, on the other hand, imply that synergistic sugarcane industrial waste CQDs could be used as bioimaging and therapeutic compounds <sup>(110)</sup>.

In 2016, Wang, H., *et al.*, Nitrogen-doped carbon dots were created by pyrolyzing citric acid and ammonia directly. The excitonic absorption of nitrogen-doped carbon dots is proportional to the nitrogen doping concentration. The N-doping can be easily changed by changing the mass ratio of reactants. The manufactured green nitrogen-doped carbon dots solar cell obtains the best power conversion efficiency of 0.79 percent under AM 1.5 G one full sun irradiation, which is the maximum efficiency for carbon dot-based solar cells <sup>(111)</sup>.

In 2016, Lee, K., *et al*, Graphene-GQD chelate-based transparent supercapacitors on flexible substrates were created to construct transparent power supply units. The devices have a high degree of transparency (92 percent at 550 nm), a high energy storage efficiency (9.09 F/cm<sup>2</sup>), a fast contact time (8.55 ms), and notable stability <sup>(112)</sup>.

In 2017, Jian, X., *et al*, A poly pyrrole composite was employed with CQDs via electrochemical deposition as an alternate method of producing electrode materials for transparent solid-state supercapacitors. The flexible supercapacitors have a specific capacitance of 315 mF/cm<sup>2</sup>, a current density of 0.2 mA/cm<sup>2</sup>, and a cycling stability of 2000 cycles <sup>(113)</sup>.

In 2020, Raeyani, D., *et al*, It was said to be a GQD-based optical gas sensor made using a hydrothermal process with graphite intercalation compounds. Due to the lack of various chemicals and the use of the basic drop-casting approach, both synthesizing and sensor production procedures are very inexpensive and straightforward to make. When exposed to CO<sub>2</sub> gas at ambient temperature, GQD sensor films showed a considerable shift in optical absorption. The high and partially reversible response obtained suggests that it could be used as an optical gas sensor to detect CO<sub>2</sub> <sup>(114)</sup>.

In 2016, Long, L. M., *et al*, Humidity sensors were made from synthetic graphene quantum dots (GQDs) and spin-coated composite thin films of GQDs, PEDOT: PSS, and CNT (GPC). The recovery time of the GOD/CNT/PEDOT: PSS (GPC) sensors was reduced from 70 seconds (0.4 weight percent CNT) to 60 seconds (0.8 weight percent CNT) and 40 seconds (1.2 weight percent CNT) <sup>(115)</sup>.

In 2020, Yu, Z., *et al*, To make a ZnO/CQDs composite, carbon quantum dots (CQDs) were produced using a hydrothermal technique and subsequently doped into ZnO using a grinding procedure. According to a gas sensitivity test of the ZnO/CQDs composite, the sensor had a high NO response (238 for 100 ppm NO) with response and recovery durations of 34 and 36 seconds, respectively <sup>(116)</sup>.

In 2019, Wei, Y., *et al*, CQDs were produced in a microwave reactor, and a sensor based on CQDs was built with a glassy carbon electrode (GCE) to detect ascorbic acid selectively (AA). High sensitivity ( $44.13 \text{ A}^{-1} \text{ M}^{-1} \text{ cm}^{-2}$ ,  $9.66 \text{ A}^{-1} \text{ M}^{-1} \text{ cm}^{-2}$ ) correlate to linear ranges of  $0.01^{-3} \text{ mM}$  and 4-12 mM, respectively, with a low detection limit of 10 M to AA <sup>(117)</sup>.

In 2020, Zhao, D., *et al*, The solid-phase synthesis of long-wavelength-emitting HCDs (yellow-emitting,  $\text{em} = 541 \text{ nm}$ ) obtained a quantum yield of 30%, utilizing L-cysteine hydrochloride anhydrous and citric acid as carbon sources and dicyclohexylcarbodiimide as a dehydrating agent, and reacting at 180 C for 40 minutes. The photoluminescence ability of the HCDs created utilizing the new solid-phase technique is persistent and extremely efficient, indicating that they will have a promising future in a variety of domains <sup>(118)</sup>.



In 2021, Peng, H., *et al*, Chemical etching and organic carbon dots were used to create very hydrophobic surfaces (OCDs). Coating the surface of etched-Al with OCDs at a concentration of 0.01mg/mL or higher aids in the formation of a continuous hierarchical micro-nanostructure. In addition to the usual fluorine or silicon modification, the results show, an unique way to produce extremely hydrophobic surfaces of materials using a low-cost and ecologically friendly OCD coating <sup>(119)</sup>.

In 2016. Hydrophobic graphene quantum dots (C<sub>12</sub>-GQDs) were synthesized using a simple chemical exfoliation process by Zhang, B., *et al*, The results show that the prepared C<sub>12</sub>-GQDs when coated on the glass substrate have the contact angles of 97° (C<sub>12</sub>-GQDs<sub>Hexane</sub>) and 98 ° (C<sub>12</sub>-GQDs<sub>Toluene</sub>). So, the wettability can be changed from GQDs (hydrophilic) to C12-GQDs (hydrophobic) <sup>(120)</sup>.

In 2021, Zirak, M., *et al*, Carbon quantum dots (CQDs)-ZnO composite thin films were used to examine the hydrophilic properties. The composite was prepared by ultrasonic mist vapour deposition (UMVD) method. The results show super-hydrophilic surface after annealing (470 ° C) with contact angle of 3° <sup>(121)</sup>.

## 1.10. Aim of the present study

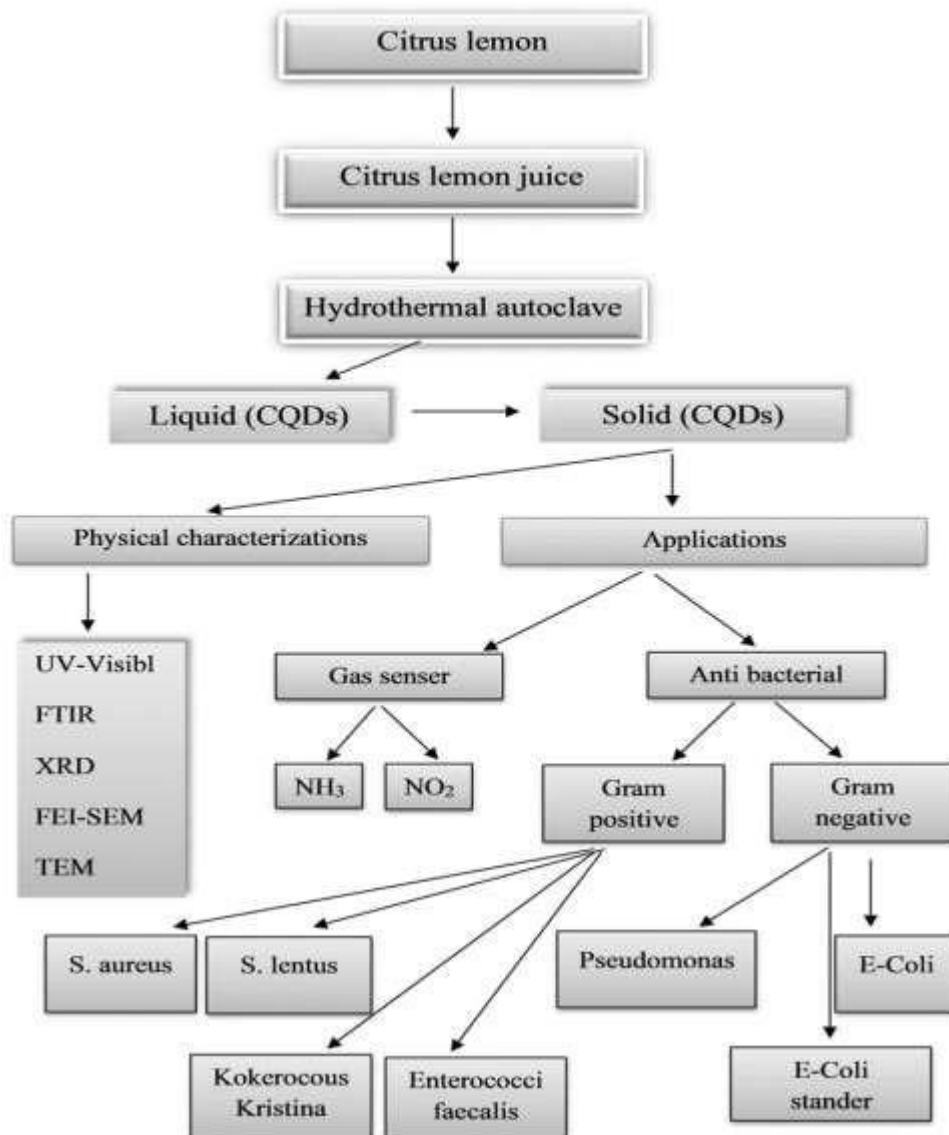
The main objective of this study is:

1. Using Lemon juice to produce carbon quantum dots, because of its low cost, simple, repeatable, and environment friendly.
2. One-step hydrothermal carbonization (HTC) process can be used to synthesis CQDs. HTC is a facile synthesis process that uses low temperature profiles ( $\leq 250$  ° C).
3. Study the effectiveness of the obtained CQDs in different applications (Anti-microbial such as (*Escherichia Coli Stander*, *Staphylococcus lentus*, *E-Coli*, *Staphylococcus aureus*, *Kokerocous Kristina*, *Enterococci faecalis*, and *Pseudomonas*), gas sensor such as ( $\text{NO}_2$  ,  $\text{NH}_3$ ), and Hydrophilic or Hydrophobic).

## 2. Experimental part

### 2.1 Introduction

In this chapter, the equipment utilized as well as the processes for making carbon quantum dots (CQDs) from lemon juice are discussed. Furthermore, in order to determine their application the produced CQDs have been tested for antibacterial, gas sensor, and hydrophilic and hydrophobic properties



Schem (2.1). Diagram of the system work.

## 2.2 Chemicals

Chemicals used in the experiment are tabulated in table (2.1) below.

Table (2.1).Chemicals used in the experiments

No.	Chemical Materials	Chemical Formula	Supplier From	Purity %
1	Muller Hinton Agar	-----	Thomas Baker INDIA	----- -

## 2.3. Equipments:

In this study equipments are used as illustrated in Table (2.2)

Table (2.2): Equipment and their manufacturers that were used in this study.

N0	Equipment	Production Company	Model
1	Transmission electron microscope (TEM)	Tecnai_G2ES1111, FEI	TecnaM
2	Field emission scanning electron microscopy (FE_SEM)	FEI CORPORATE HEADQUARTERS	Quanta 200 FEG
3	X-Ray diffraction (XRD)	Shimadzu X_ray Diffractometer	XRD_600 0

4	Fourier transform infrared (FTIR)	Bruker company	VERTEX 70 With Hyperion Scanning,
5	Ultraviolet-visible spectroscopy (UV_VIS)	Keysight Technologies Inc. United states	Cary 100
6	Electrical oven	homemade	India
7	C W Furnace 1200	CARBOLITE	England
8	Magnetic hotPlate stirrer	M101020 03	India
9	Drying oven	Memmert	Germany
10	Centrifuge	Human LabHSI85R	Germany
11	Laminar flow hood	K&K Scientific Supplier	Korea
12	Hydrothermal autoclave reactor	.....	Iraq- Baghdad

## 2.4. Hydrothermal autoclave reactor

Quantum Dot Carbon synthesis was by The hydrothermal autoclave reactor the manufactured ,It is also known as Hydrothermal synthesis reactor, digestion or pressure melting bombs, hydrothermal synthesis reactor, high temperature and pressure digestion vessels, Teflon Lined Autoclave, Hydrothermal Autoclave Reactor is use to carry hydrothermal reaction at high pressure and high temperature. and which consists of high-quality a stainless steel cylindrical test chamber

with durable, reliable structure; the sealing effect being stable long term without leakage. with a diameter of 7.5 cm and a height of 10 cm, and a thermal Teflon is corrosive resistant placed inside the reactor of volume 100 ml with a diameter of 6 cm and a height of 9 cm and a tight lid to close the reactor to prevent any heat from leaking outside the reactor to complete the preparation process



Figure 2.1: Hydrothermal autoclave reactor.

## 2.5 Raw Material

Lemon was purchased from local grocery stores as a raw material in the synthesis process of carbon quantum dots. Where the citrus is a fluid plant of the Rutaceae family. Approximately 140 genera and 1300 species are present in the genus citrus. The "term" lemon comes from ancient French called "limon." The term "limone," Arabic "lamun" or "Imun" and Persian "Imun" have distinct names in Italy too. Generic word 'lime' to citrus fruit is connected to Sanskrit nimbu. Many other lemon fruit names are available. Flavonoids, acids, caffeine, pectin and minerals are the primary elements of the chemical composition<sup>(122)</sup>.

## 2.6 Method

### 2.7. Green synthesis of carbon quantum dots (CQDs) from lemon juice

Transfer 100 mL of extract lemon juice (ivory white solution after squeezing and filtering, for the pulp is completely removed and then placed in an electric oven for half an hour at 70 ° C for 30 minutes to remove all impurities) into a Teflon-lined stainless steel autoclave for hydrothermal treatment at 180°C for 8 hours. The autoclave was naturally cooled to room temperature after the process. The ivory white fluid transformed to a dark brown solution during the hydrothermal process, showing the production of C-dots. These carbon dots were then filtered with filter paper before being dried in an 80° C drying oven for 30 minutes <sup>(123)</sup>.



Figure 2.2: Scheme of preparing CQDs from lemon juice.

## **2.8 Characterization of carbon quantum dots (CQDs)**

The main goal of these tests is to look at the structure and properties of the carbon quantum dots that have been created. Used. (TEM, FE-SEM, XRD, UV-Vis, and FTIR) to characterize it.

### **2.8.1. Transmission electron microscope (TEM)**

TEM analysis was carried out in Turkey to obtain information about the microchemistry, and crystal structure on a microscale. Where TEM used to characterize and understand the structure of carbon quantum dots generated from lemon juice, using an FEI technical G2 Split Biotic transmission electron microscope at (120 kv).

### **2.8.2. Field emission scanning electron microscopy (FE\_SEM)**

The shapes and diameters of carbon quantum dots generated from lemon juice were investigated using an FE- SEM (model Quanta 200FEG, in Iran ) set to run at various magnification levels (120 KV).

### **2.8.3. X-Ray diffraction (XRD)**

X –Ray diffraction measurements were accomplished in Iraq- Baghdad using Micro Max 007HF DW. The diffracted power of the Cu K $\alpha$  radiation ( $\lambda=1.54060\text{\AA}$ , 40 Kv and 30 mA) was measured in  $2\theta$  range from  $20^\circ$  to  $80^\circ$ , the maximum power is (1.2 Kw).



#### **2.8.4. Fourier transform infrared (FTIR)**

Spectra was measured the FTIR in College of Education for Pure Sciences, University of Anbar using (model VERTEX 70 With Hyperion Scanning) device for investigating the change of functional groups of the CQDs. The samples were grounded and mixed with KBr. The resultant powder was grounded and pressed into thin pellets and the absorbance was measured.

#### **2.8.5. Ultraviolet-Visible spectroscopy (UV-Visible)**

The optical properties of CQDs were analyzed using UV-Vis spectroscopy in the College of Science, University of Anbar (Cary 100, Keysight Technologies Inc) by monitoring the electron spectra of the samples employing a Shimadzu UV-1800 UV-Vis spectrophotometer. The spectral bandwidth ranged from (200 - 1100) nm at a wavelength resolution of 1 nm, while quartz cuvettes were applied for the measurements, over a path-length of 10 mm. The device was fully controlled by UV Probe software package.

### **2.9 Applications**

#### **2.9.1. Anti-bacterial activity**

Using an agar well diffusion method in a clean aseptic room, the antibacterial effectiveness of CQDs was investigated against Gram positive bacteria such as *Staphylococcus lentus*, *Staphylococcus aureus*, *Kokerocous Kristina*, and *Enterococci faecalis*, as well as Gram negative bacteria such as *Escherichia Coli Stander*, *E-Coli*, and *Pseudomonas* <sup>(110)</sup>. Before culture, 20 mL of Muller-Hinton was put into sterilized Petri dishes. A sterile wire loop was used to collect stock

cultures of bacterial species <sup>(124)</sup>. Prepared bacterial inoculums were swabbed across the surface of the nutrient agar medium (growth medium) with a sterile cotton swab to preserve equal dispersion of the germs across the plate surface. Using a sterile point <sup>(110)</sup>, wells with a diameter of 6 mm were bored into the agar plates <sup>(124)</sup>. Carbon quantum dots (prepared by dissolving 20mg of CQDs in 20mL of distilled water) were placed in the well and cultured for 24 hours at 37 °C. One by one, the inhibitory zone (mm) created in the Petri dish was observed <sup>(110)</sup>. This work was done in Al-Ramadi Teaching Hospital, Laboratory Departments.

### **2.9.2. Gas sensor system**

The sensitivity parameter, namely the reaction time and recovery time of the manufactured CQDs gas sensor detector, is determined using an appropriate setup. Figure 2.2 shows the gas sensor testing equipment, which consists of a cylindrical stainless steel test chamber with a diameter of 30 cm and a height of 35 cm. The chamber has an effective volume of 6594 and an inlet for allowing the tested gas to flow in and an air admittance valve for allowing ambient air to flow out after it has been evacuated. Electrical connections to the heater, K-type thermocouple, and sensor electrodes are made via a multi pin feed through at the chamber's base. To manage the operating temperature of the sensor.

The heater consists of a hot plate and a K-type thermocouple inside the chamber. The fluctuation of the sensor current while exposed to air-NO<sub>2</sub> and NH<sub>3</sub> gas mixing ratios is recorded using a PC-interfaced digital multimeter of type UNI-T UT81B and a Laptop PC. Through a flow meter and needle valve configuration, the mixing gas is fed from zero air and test gas. To obtain the true sensitivity, the

mixing gas is delivered through a tube over the sensor inside the test chamber, this test was conducted in the Department of Chemical Engineering-University of Technology.



Figure 2.3: (A) Gas sensor testing system; (B) Gas sensor sample setup.

### 2.9.3. Sensor testing procedure

The test setup's operation is demonstrated in the following points:

1. The sensor is put on the heater after the test chamber is opened. The test container was closed after the appropriate electrical connections between the pin feed through and the sensor were made using conductive aluminum sheet.
2. Set a 6 volt bias voltage between the electrodes' two sides.
3. Turn on the rotary pump to empty the test chamber to about 1 mbar, then use a temperature controller to adjust the sensor to the correct working temperature. The schematic diagram of the electrical circuit for gas sensing measurements is shown in Figure 2.3.
4. Adjust the air and tested gas flow rates with the needle valves, then install the volumetric concentrations of 1, 2, and 3 percent test gas to the air.
5. A PC-connected digital multimeter of the type UNIT-UT81B is used to measure current fluctuation.
6. The digital multimeter records the air flow biasing current, then turns on the testing gas ( $\text{NO}_2$ /or  $\text{NH}_3$ ) for a few seconds to stabilize the current, then switches off the test gas to record the recovery time.

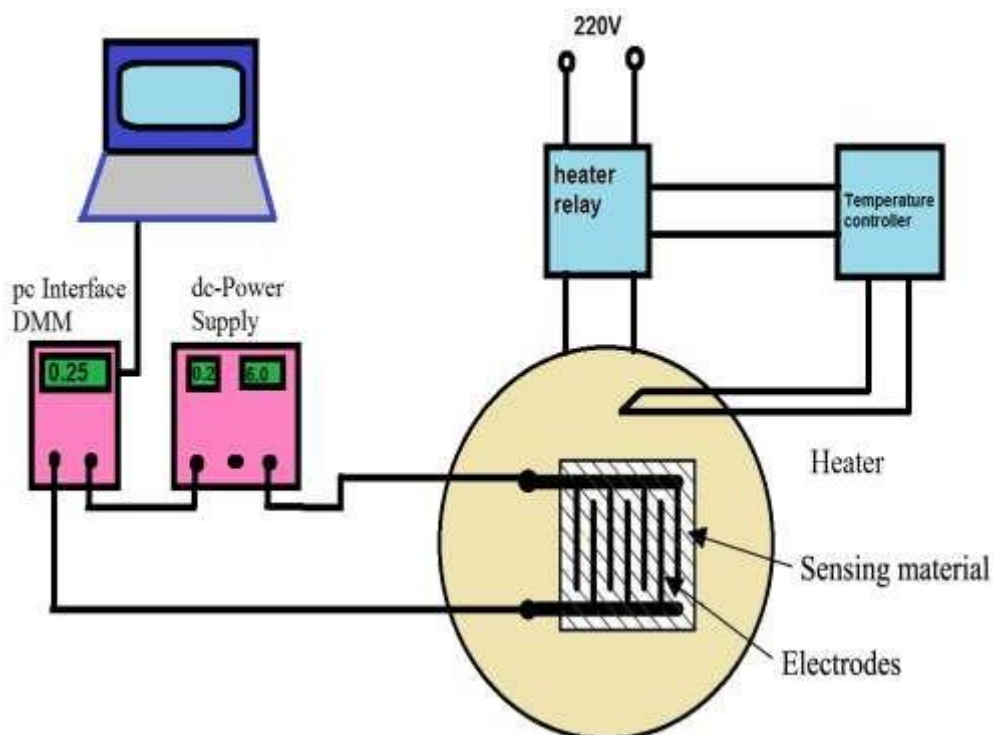


Fig 2.4: Gas sensing and electrical circuit setup schematic diagram.

#### 2.9.4. Hydrophilic and hydrophobic properties

Carbon quantum dots layered on a glass slide altered its properties with thickness  $2.32 \mu\text{m}$ . To identify the hydrophobic of the sample, water contact angles were measured by employing the sessile drop method<sup>(125)</sup> the contact angle value, which is the angle between the water droplet and the sample surface, was calculated by contact angle system, which used the DSA100 software. The Mechanism of contact angle measurement was shown in Figure (2.4).

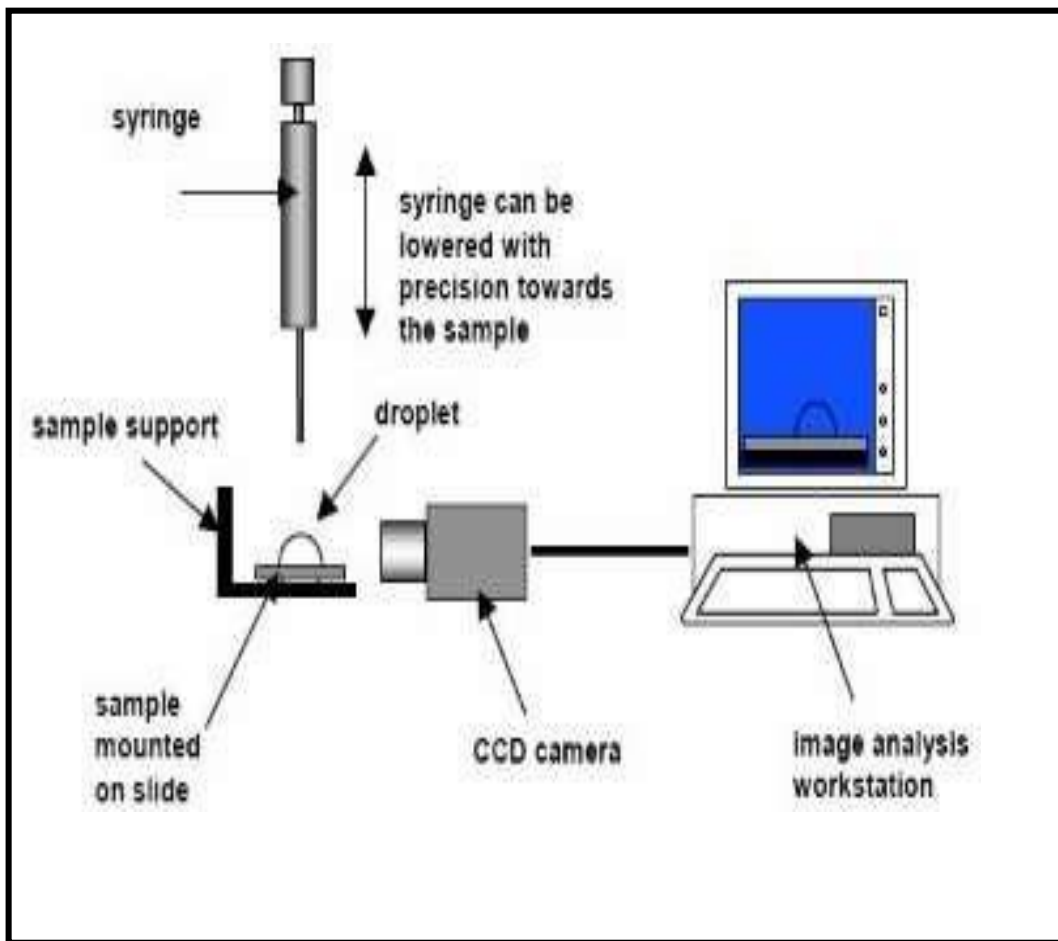


Figure 2.5: Mechanism of static contact angle measurement <sup>(126)</sup>.

### 3. Results and Discussions

#### 3.1 Carbon quantum dots preparation from raw materials

Carbon quantum dots preparation from raw materials, where methods implied in this study to prepare the CQDs compared to that of previous classical methods. Where a solid powder of carbon quantitative points (CQDs) preparation was prepared from lemon juice, by hydrothermal carbonization, using incineration furnace at the temperature to 180 ° C for 8h.

#### 3.2 Carbon quantum dots identification

CQDS particles were identified using different methods:

##### 3.2.1 Transmission electron microscope (TEM) analysis method

The morphological characterization of CQDs was studied using a TEM image for CQDs generated from lemon juice at 182°C for 8 hours. The particle size and structure of CQDs are studied using TEM. CQDs are approximately spherical and have nearly uniform dispersion with a diameter of (2–8) nm, according to the study.

CQDs have an average diameter of 3.99 nm, according to the frequency histogram (Fig. 1.C). Equations (3.1) and (3.2) were used to calculate the average diameter of CQDs.

$$D = \pi r^2 \quad \dots \dots \dots \dots \dots \dots \dots \dots \quad (3.1)$$

Where r was computed using (2)

$$r = \sqrt{(A/\pi)} \quad \dots \dots \dots \dots \dots \dots \dots \quad (3.2)$$

Where,  $r$  = Diameter

$A$  = the average area

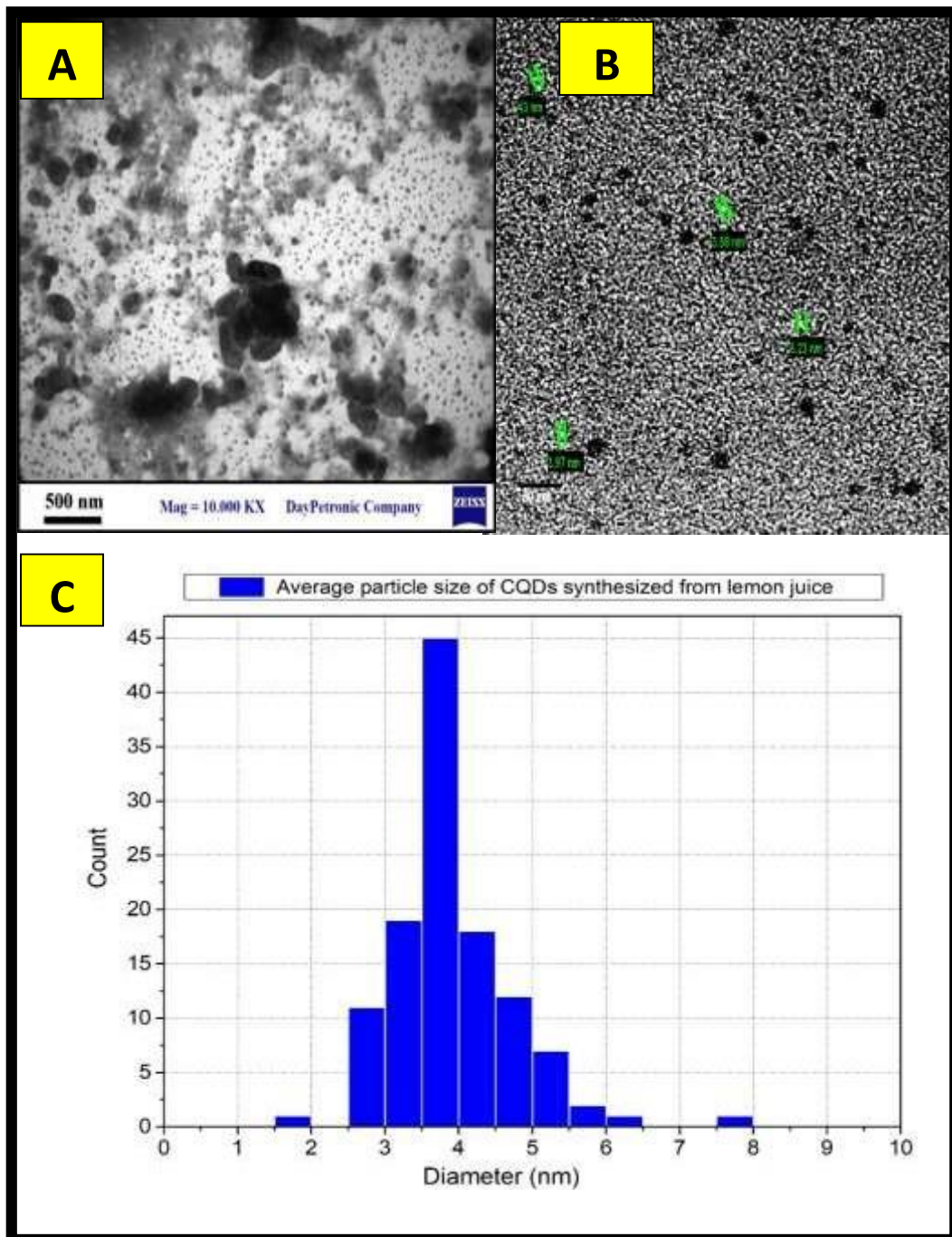


Figure (3.1): A., B. TEM micrograph of CQDs, C. Diameter count distribution of CQDs.



### 3.2.2. Field Emission Scanning Electron Microscope (FE-SEM)

FE-SEM analysis is used to investigate the structural morphology of CQDs. The surface of CQDs synthesized from lemon juice using a hydrothermal technique appeared atypically in FE-SEM micrograph (3.2).

FE-SEM pictures revealed approximately spherical CQDs with diameters ranging from (5-12) nm at various magnitudes.

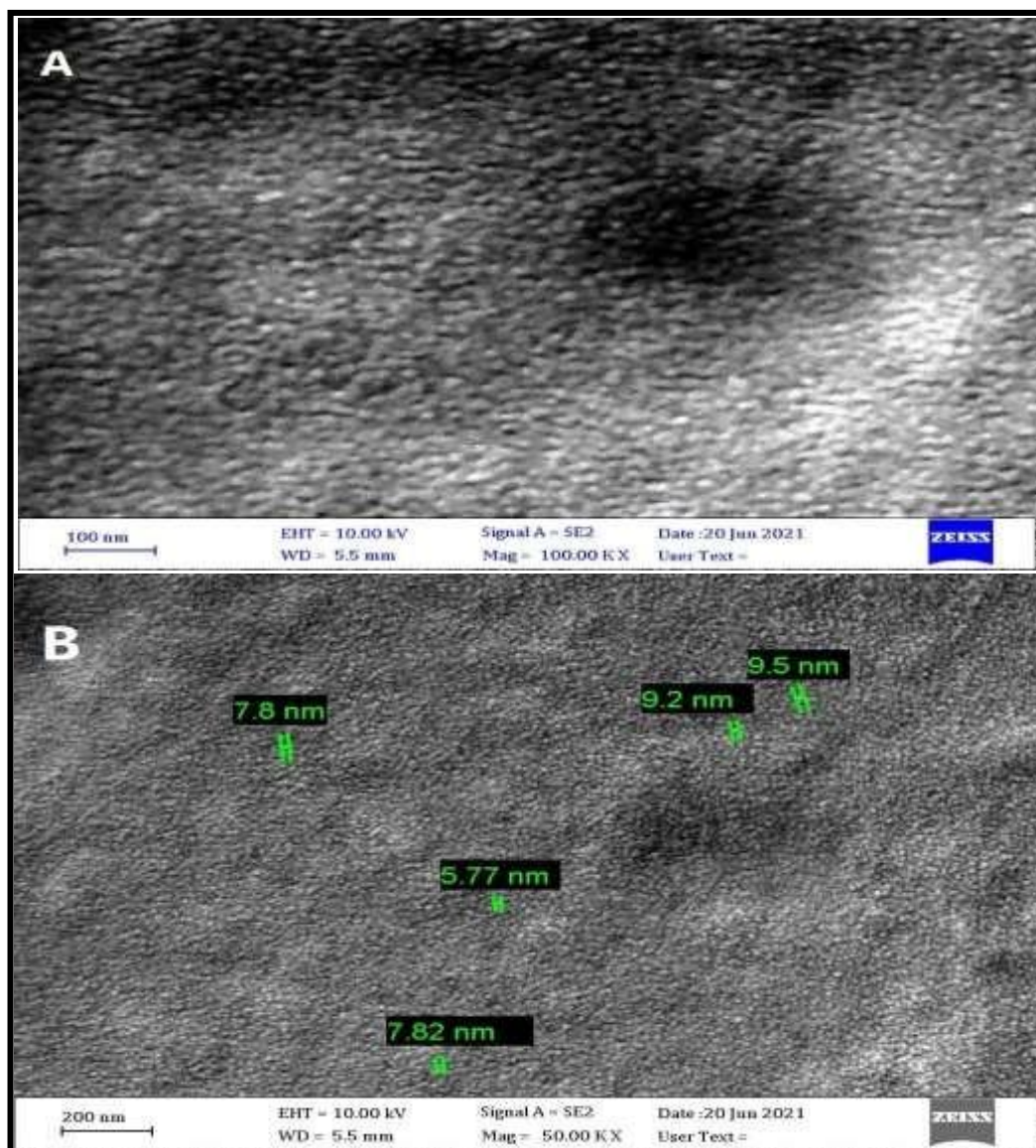


Figure (3.2): FE-SEM micrograph of CQDs synthesized in different magnification (A) 100 nm (B) 200 nm

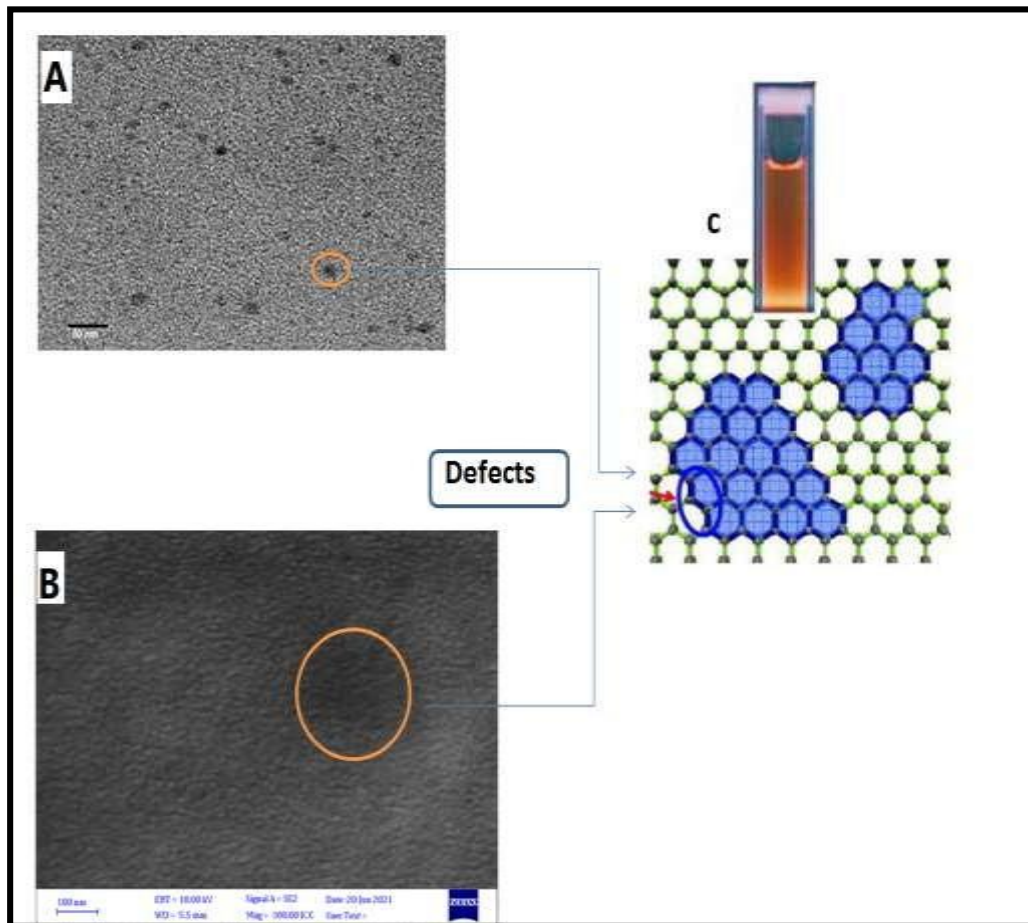


Figure (3.3): (A) TEM (B) FE-SEM images, and (C) isolated  $sp^2$  islands of CQDs <sup>(127)</sup>.

Figure (3.3) shows the TEM and FE-SEM images do not show discernible lattice structures of C-dots on the higher magnification, suggesting that C-dots are amorphous dots. Although many scientists have demonstrated the existence of a  $sp^2$  crystal carbon, most of the C-dots have weak crystallinity <sup>(128,129)</sup>. Similar to natural source-derived C-dots, lemon juice-derived C-dots also have amorphous nature in this study.

### 3.2.3. X-ray analysis (x-ray)

Figure (3.4) displays the XRD pattern of the synthesized CQDs. The diffractogram of CQDs shows many broad diffraction peaks and sharp diffraction peaks. Broad diffraction peaks of ( $2\theta = 10\text{--}30$ ) usually correspond to amorphous carbon and organic material and indexed to the (002) carbon lattice spacing <sup>(130)</sup>, and the sharp diffraction peaks indicate that the material is not ordinary carbon nanoparticles but carbon quantum dots with better crystallinity <sup>(131)</sup>. These peaks confirm the CQDs shape is hexagonal.

The grain diameter / or crystal size (D) can be calculated by the Debye–Scherrer equation: (3.3)

$$D = \frac{K\lambda}{\beta \cos(\theta)} \quad \dots \dots \dots (3.3)$$

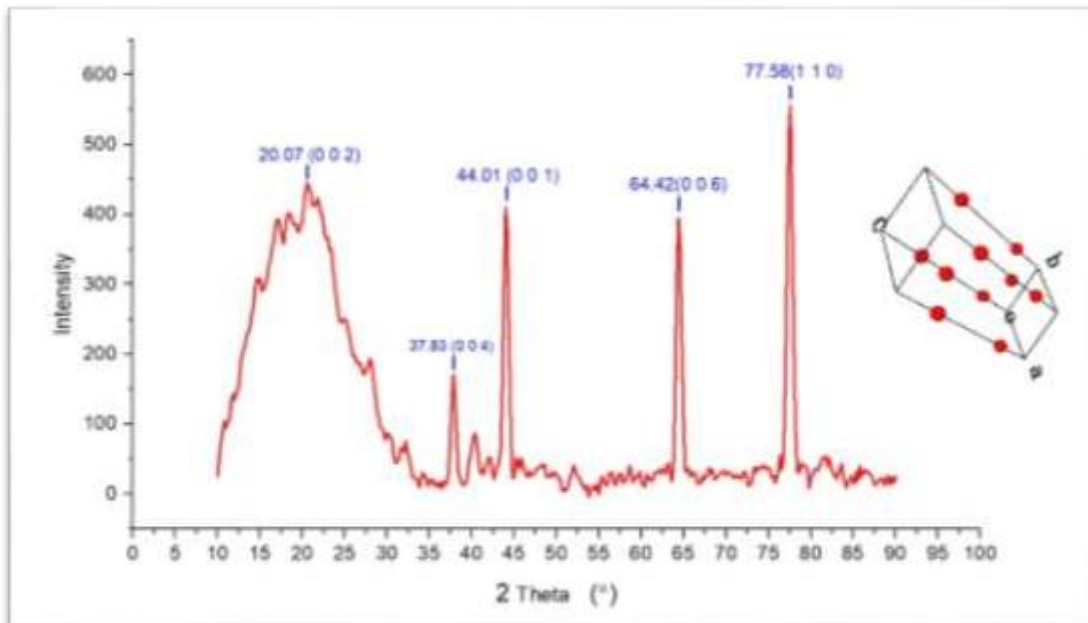


Figure (3.4): displays the X-ray analysis of CQDs.

Where  $K= 0.9$ ,  $\lambda$  is the wavelength of X-ray,  $\theta$  is the Bragg angle in radians, and  $\beta$  is the full width at half maximum of the peak in radians. From Debye–Scherrer equation, D is around 3.93 nm.

### 3.2.4 Fourier transform infrared (FT-IR) analysis

FT-IR spectroscopic investigation is evidence about the absorption bands to characterize the prepared CQDs. The presence of key functional groups on the surface of CQDs produced from lemon juice is discovered. Which accounts for their high water solubility. The peak at  $3401.61\text{ cm}^{-1}$  were attributed to stretching vibrations of O-H the absorption peaks at  $2979.05\text{ cm}^{-1}$ , indicate the existence of the aliphatic C-H bond. The sharp band observed at  $1712.47\text{ cm}^{-1}$  indicated the presence of C=O groups. And the bands in range of  $1301.55\text{--}1099.74\text{ cm}^{-1}$  illustrate the existence of C-O functional groups, as shown in Figure (3.5).

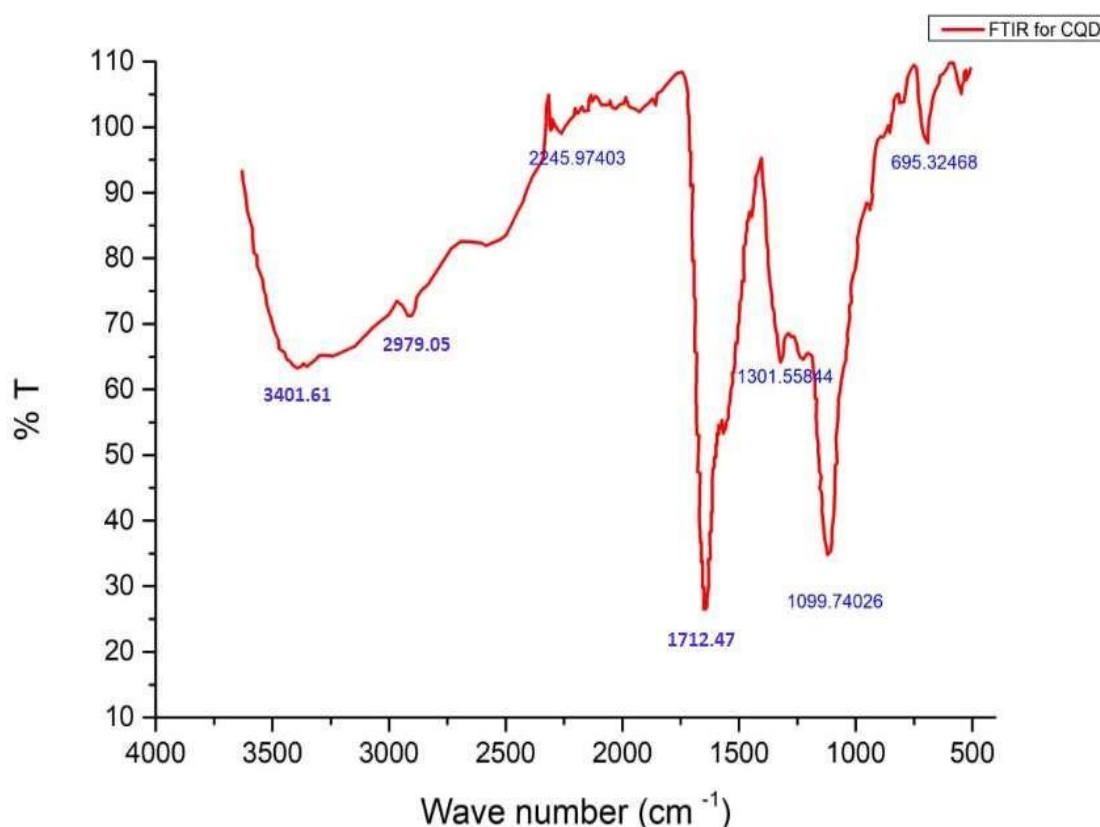


Figure (3.5): FT-IR spectrum of synthesized CQDs

### 3.2.5 UV-Visible spectroscopy of CQDs

The characterization of the prepared solution pale yellow for CQDs was studied using UV-Visible spectroscopy, which measured the UV-visible absorption spectra of the prepared CQDs in deionized water. Where the electronic spectrum of the synthesised CQDs has exhibit a broad peak at 288 nm, which shows the formation of CQDs. This result is compatible with Nguyen, T. N., *et.al*<sup>(132)</sup> as shown in Figure (3.6).

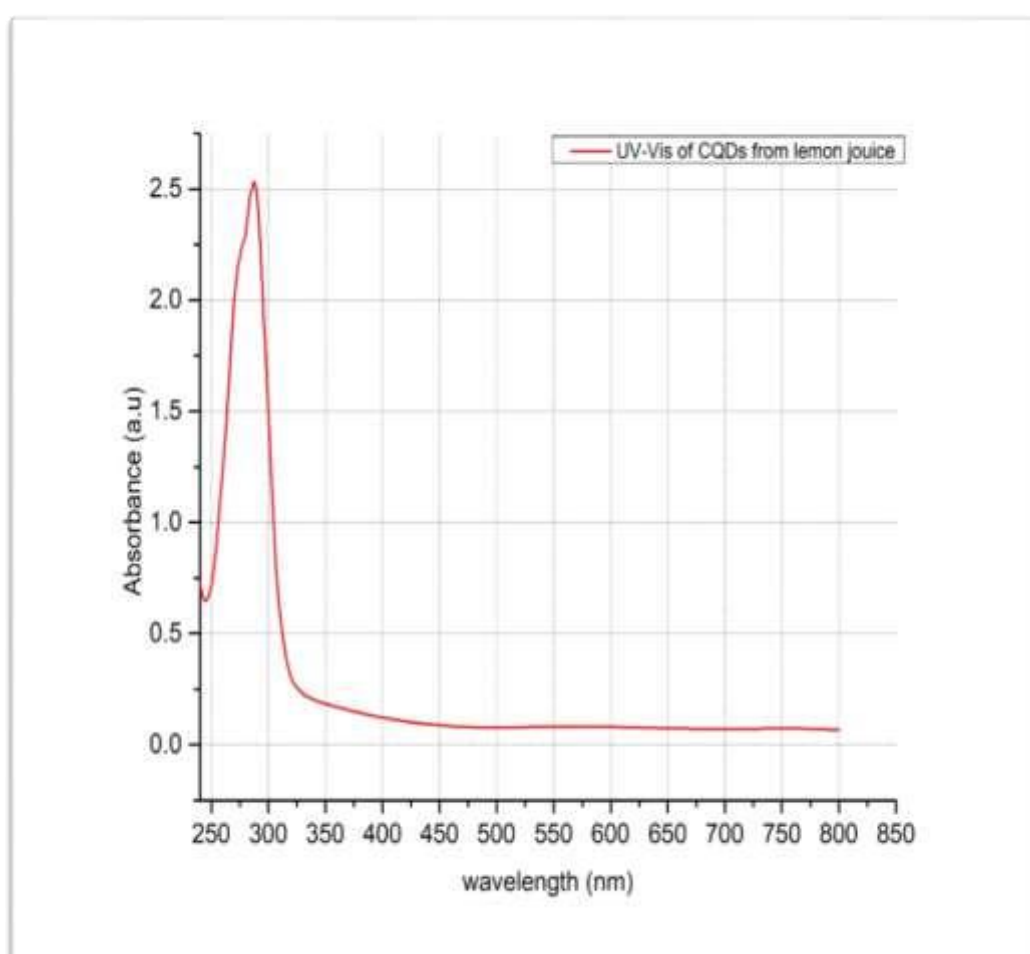


Figure (3.6): UV-Visible spectrum of CQDs.

### **3.3. Carbon quantum dots applications**

#### **3.3.1 Anti-bacterial activity**

CQDs are generally known as benign and non-toxic in vitro and in vivo. However, C-dots can efficiently generate ROS by activating oxygen in the air or water, resulting in the generation of hydroxyl free radicals ( $\text{OH}^\bullet$ ) and/or singlet oxygen ( $\text{O}_2$ ), which can destroy some of the cell's most critical macromolecules and lead to cell death. Intracellular protein inactivation, lipid peroxidation, mitochondrial dysfunction, and gradual cell membrane disintegration are all caused by reactive oxygen species (ROS), which leads to necrosis/apoptosis and cell death<sup>(133)</sup>. The antibacterial ability of the samples contained CQDs were determined in terms of the inhibition zone created on agar around the paper discs against different positive and negative gram as shown in (Figure 3.7). The diameter (D) of the inhibition zone for different bacterial were shown in (table1). Kokerocous Kristina had stronger antibacterial more than others. Results show that CQDs have anti-bacterial ability against Gram-positive bacteria comparing to Gram-negative bacteria. Because the permeability to the cell wall is easier in positive bacteria containing 90% peptide glucan and 10% other substances, and negative bacteria contain 10% glucan peptide.

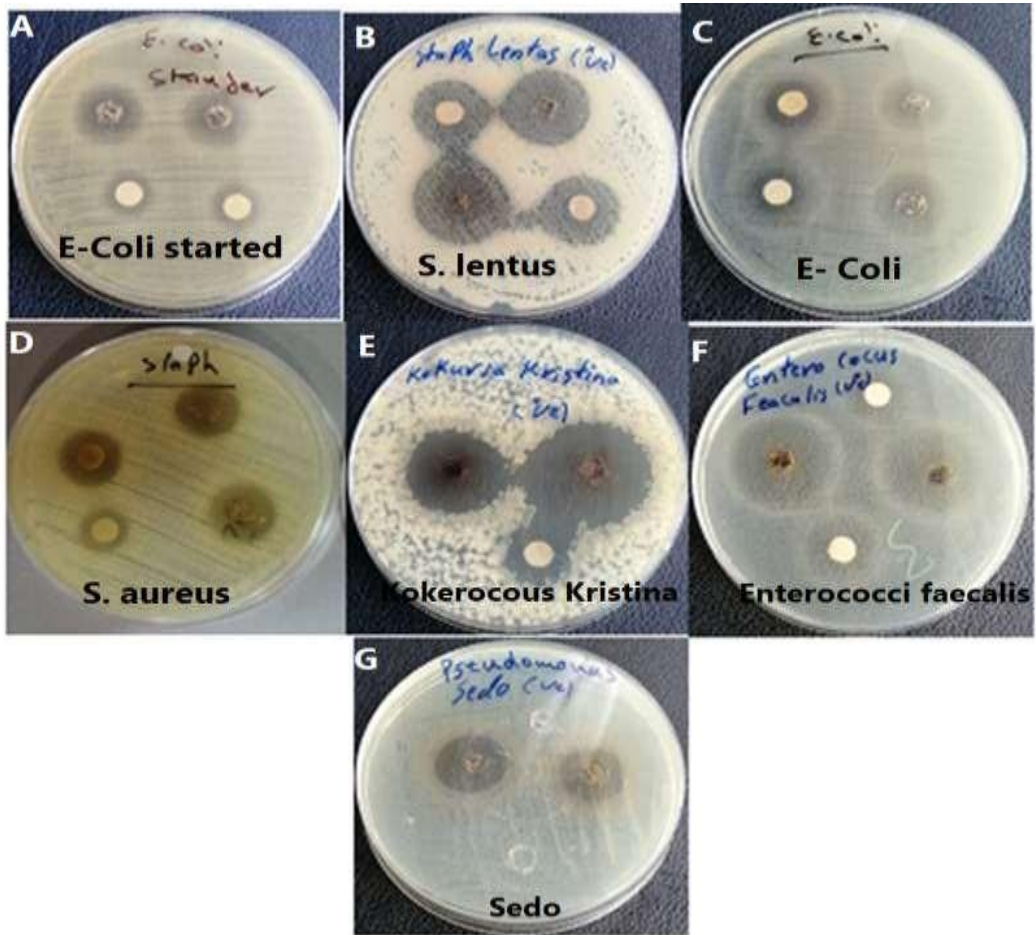


Figure (3.7): Antibacterial efficacy of CQDs against (A) *Escherichia Coli Stander*, (B) *Staphylococcus lentus*, (C) *E-Coli*, (D) *Staphylococcus aureus*, (E) *Kokerocous Kristina*, (F) *Enterococci faecalis*, (G) *Pseudomonas*

<b>Tested organism</b>	<b>Gram reaction</b>	<b>Inhibition zone (mm)</b>
<i>Escherichia Coli</i> <i>stander</i>	-Ve	17
<i>E-Coli</i>	-Ve	18
<i>Pseudomonas</i>	-Ve	21
<i>Staphylococcus</i> <i>lentus</i>	+Ve	28
<i>Enterococci</i> <i>faecalis</i>	+Ve	25
<i>Kokerocous</i> <i>Kristina</i>	+Ve	35
<i>Staphylococcus</i> <i>aureus</i>	+Ve	22

Table (3.1): Diameter of the inhibition zone for different bacterial of CQDs



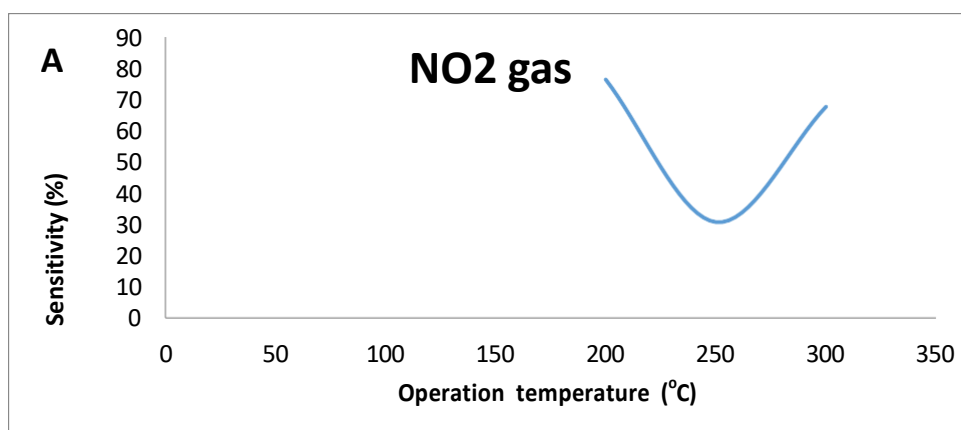
### 3.3.2 Gas sensor application

#### 3.3.2.1. Determination of operation temperature of the gas sensor:

The sensitivity of a chemical sensor is one of the factors that characterize its performance. Selectivity, stability, detecting limit, and dynamic range are all important characteristics. Resolution, response tuning, and recovery time are all factors to consider. In general, a chemical sensor that meets all of the criteria should be very sensitive.

The high temperature required for sensor operation (200–500 °C) is one of the most prominent shortcomings of CQDs gas sensors. As a result, the impact of operation temperature on thin film sensitivity was investigated with the goal of lowering the operating temperature to the lowest possible level.

Equation (1.1) was used to calculate the sensitivity factor (S %) at various temperatures, which was found to be around 79 % (200 C°) and 22 % (300 C°) for NO<sub>2</sub> and NH<sub>3</sub>, respectively, as shown in figure (3.8). The porosity, huge surface area, and high rate of oxidation may account for the gas sensitivity.



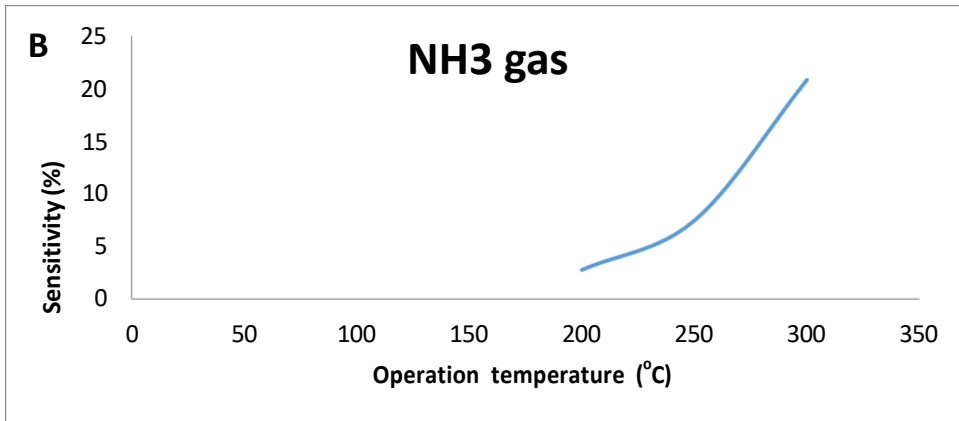
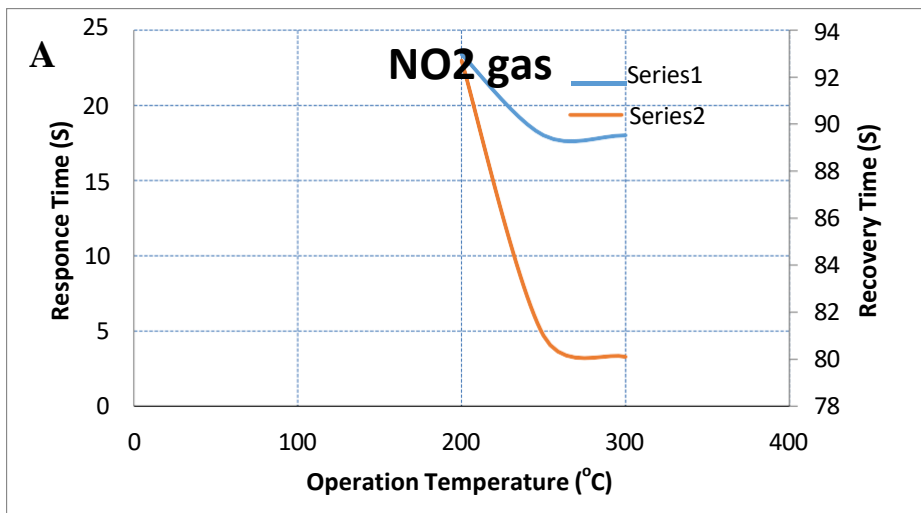


Figure (3.8): The constructed gas sensor's sensitivity varies with its working temperature (A)NO<sub>2</sub> gas, (B) NH<sub>3</sub> gas.

### 3.3.2.2 Response time and recovery time:

Pure CQDs' response and recovery times to NO<sub>3</sub> and NH<sub>3</sub>. The tests were carried out at a bias voltage of 3V and a temperature of (200-300) ° C, Figures (3.9).



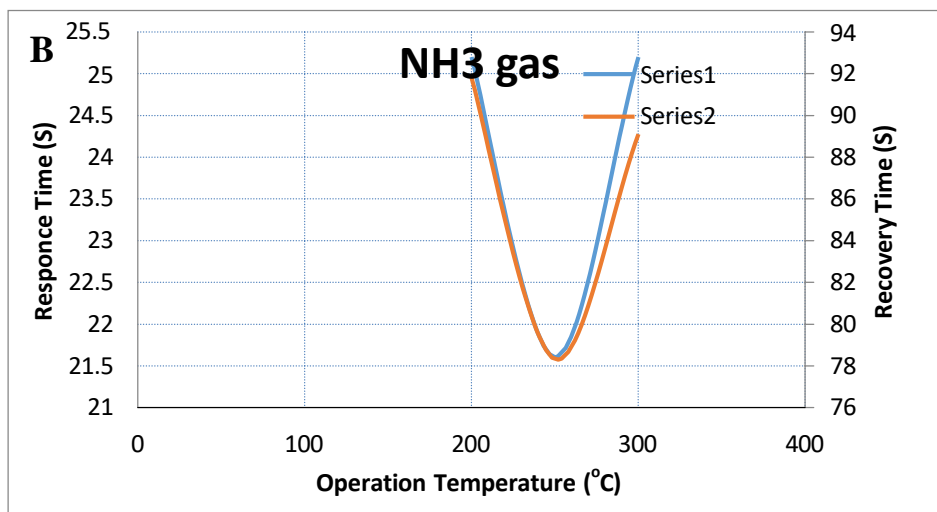


Figure (3.9): The variation of response time and recovery time with operation temperature of the prepared gas sensor.

Depicts the outcomes of response and recovery of CQD sensor with  $\text{NO}_2$  and  $\text{NH}_3$  gases. The detection time ( $T_{R(10\%)}$ ) is the time taken for sensor output signal to rise 10% above its initial value after applying the target gas in a step function equal to 23.35 s and 33 s for  $\text{NO}_2$  and  $\text{NH}_3$  respectively.

The response time, describes the time taken for the sensor output signal to reach 90% of its saturation value after applying the target gas in a step function calculated as 14.71 s for  $\text{NO}_2$  gas and 11.93s for  $\text{NH}_3$  gas, Figures (3.10).

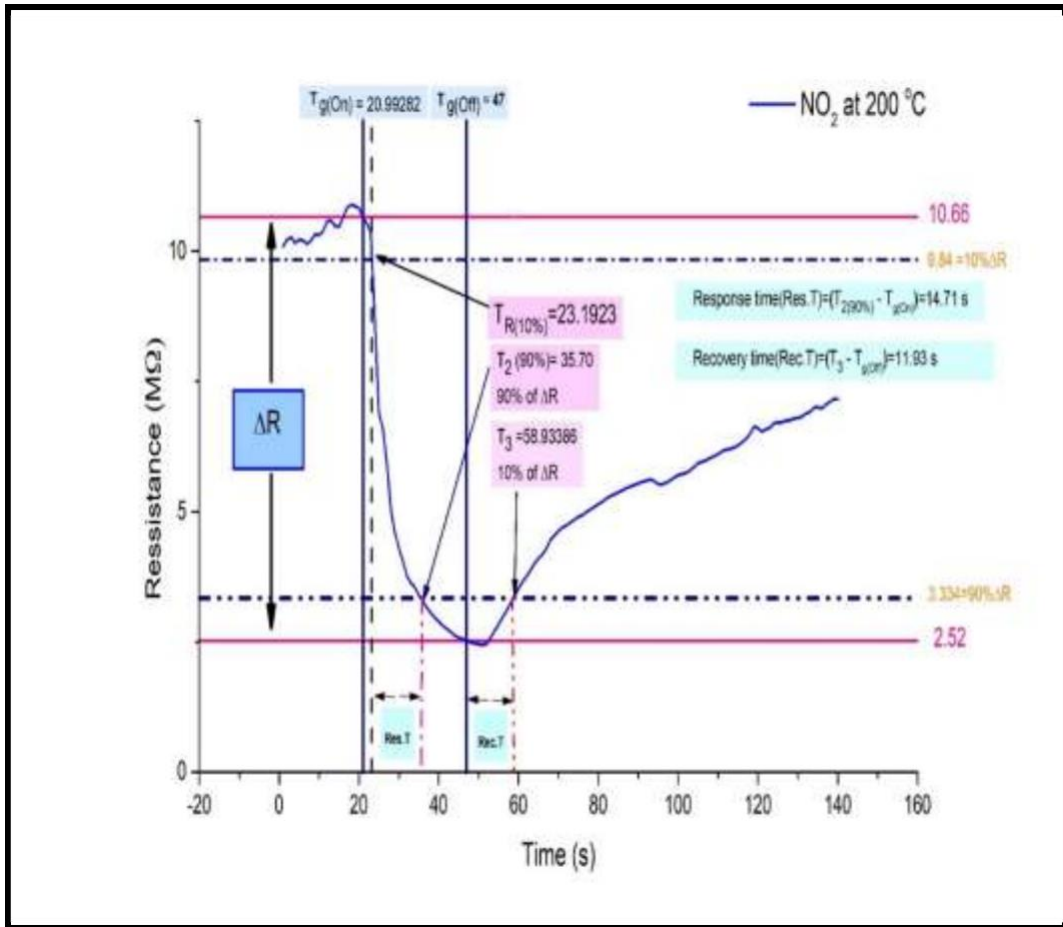


Figure (3.10): Response and recovery times of the constructed CQDs NO<sub>2</sub> gas sensor as a function of operating temperature at 200 ° C.

The recovery or decay time, is the time taken for the sensor output signal to drop to 90% of its saturation value after switching off the target gas in a step function (i.e., time taken for the sensor response to recover to within 10 % above its initial value) in this case; the recovery time for NO<sub>2</sub> was calculated as 11.21 s for NH<sub>3</sub> gas was 6.11 s at 250 ° C. these temperatures were chosen due to the fact that the CQD sensors was optimal at these degrees.

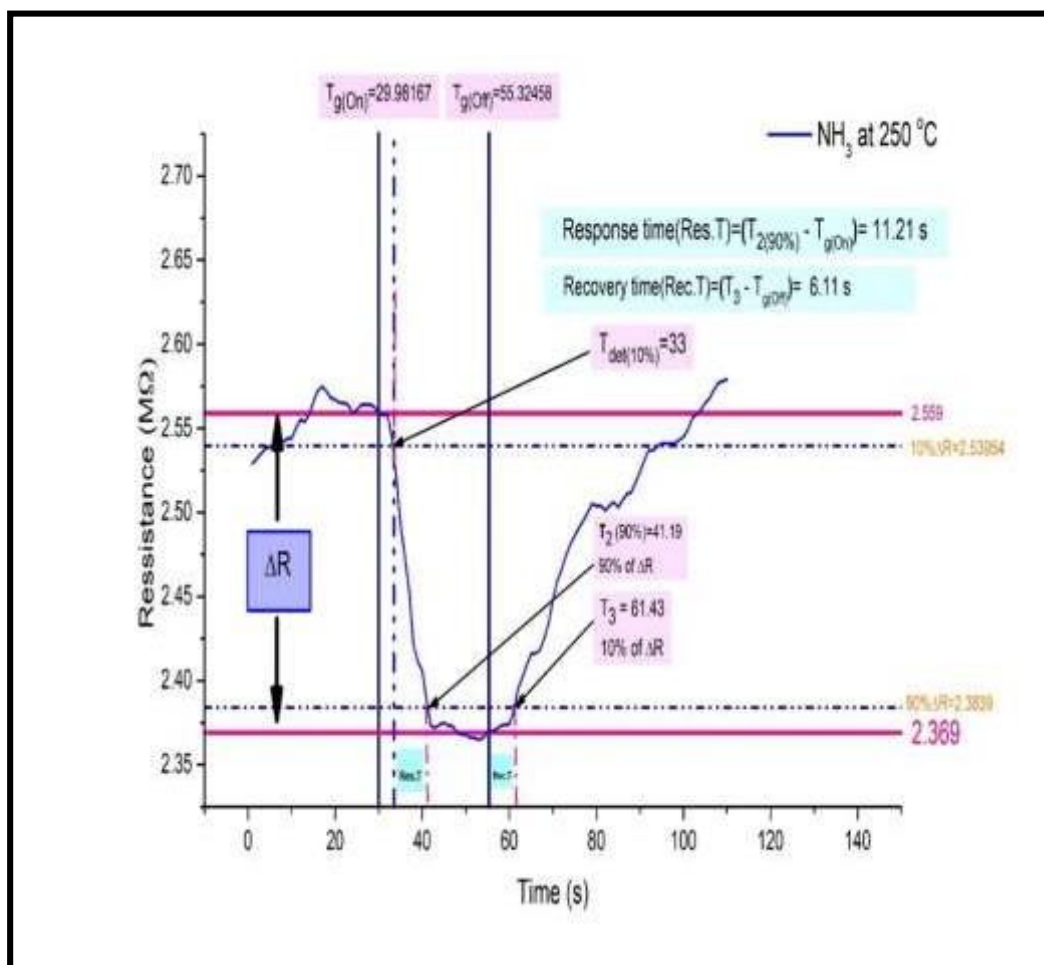


Figure (3.11): Response and recovery times of the constructed CQDs NH<sub>3</sub> gas sensor as a function of operating temperature at 250 ° C.

Table (3.2): Appreciation of the response time, recovery time and the sensitivity with operating temperature of CQDs

Response times	Recovery times	T (k)	The sensitivity (S %)	T (k)	Concentration of gas NO <sub>2</sub> (ppm)
14.71 s	11.93 s	473 k	79 %	473 k	193.7788

Table (3.3): Appreciation of the response time, recovery time and the sensitivity with operating temperature of CQDs

Response times	Recovery times	T k	The sensitivity (S %)	T k	Concentration of gas NH <sub>3</sub> (ppm)
11.21 s	6.11 s	523 k	22 %	573 k	71.61392

### 3.3.2.3. Gas sensing mechanism of CQDs.

The surface reaction between chemisorbed oxygen, oxidizing gases, and reducing gases is the basis for the gas sensing process. There are two types of oxygen adsorption on the film surface: physisorption and chemisorption. Chemisorption dominates at high temperatures. Activation energy is required for the transition from physisorption to chemisorption.

This can be done by raising the operating temperature. It has been found that as the temperature rises, the amount of oxygen adsorbed on the sensor surface increases<sup>(134)</sup>. In the temperature ranges of (200–300) ° C, molecular oxygen and atomic oxygen have a dominant influence on the electro physical and gas sensing characteristics of CQDs films. Where the oxygen is adsorbed on the carbon's surface, allowing for electron trapping. As a result, the charge carrier density is lowered, resulting in an increase in CQD resistance.

Figs (3.12) depicts the fluctuation in resistance with time of CQDs exposed to 3% NO<sub>2</sub> and 10% NH<sub>3</sub> in the air ambient fed into the testing chamber, with the bias voltage kept at (6V) at the sample's ideal operating temperature for each gas. The sensor resistance was monitored immediately against time, and it originally reached a steady state before the gas was opened to allow mixing with the air inside the

chamber. The resistance drops sharply until it reaches a stable condition, at which point the gas is turned off. The current case then reverted to its original state. The nature of the interaction between the gas molecules and the surface atoms of the sensing film determines a sensor's capacity to detect the presence of gas. The doping and defect structure of the surface have a significant impact on its reactivity.

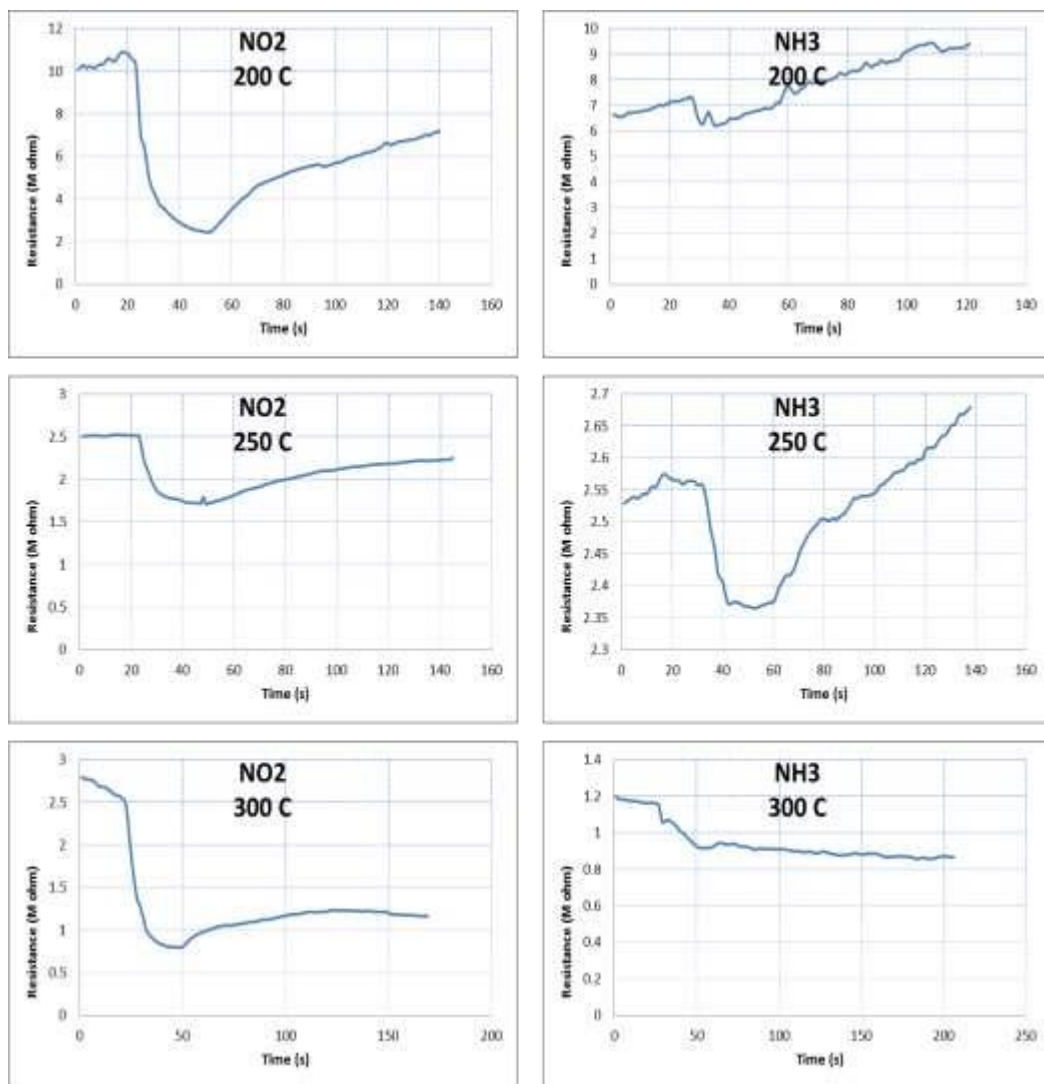


Figure (3.12): The variation resistance with time for different operation temperature of NO<sub>2</sub> and NH<sub>3</sub> gases for the gas sensor (0.2M).

### 3.3.3 Wettability of carbon quantum dots (CQDs)

The contact angles at the water–air interface were calculated to determine the wettability of CQDs, as illustrated in Fig. (3.13), where water droplets seemed to be quasi-spherical. The bare glass substrates are covered with our synthetic CQDs for this purpose. The contact angle for CQDs is  $9.534^\circ$ , indicating that the high hydrophilic is attributable to the presence of very hydrophilic oxygenated functional groups, and that the hydrophilic carboxyl group was used to connect the surface of CQDs. This approach yields CQDs with good water solubility<sup>(135)</sup>.

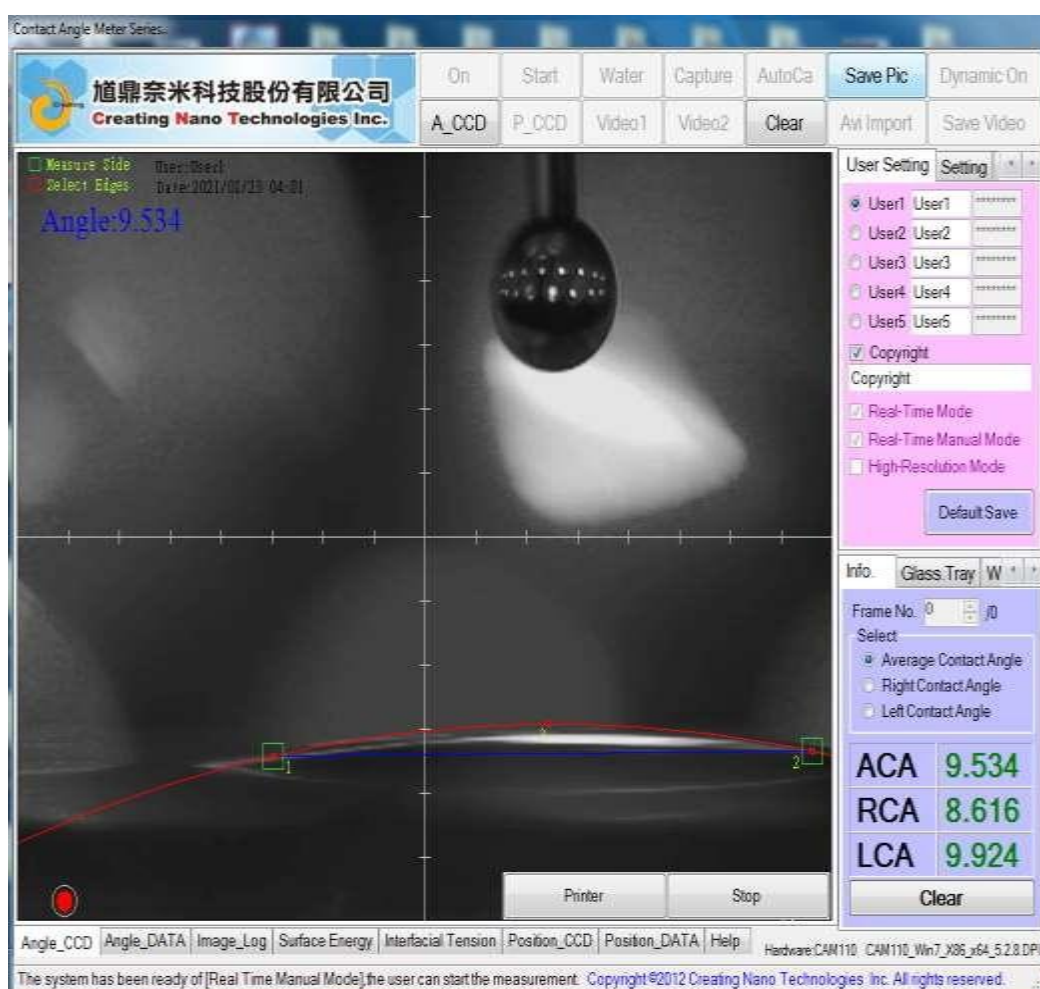


Figure (3.13): CQDs on bare glass were measured for their static contact angles in water.



## 3.4. Conclusions and recommendations

### 3.4.1. Conclusions

Throughout the results obtained by this work, it can be concluded that:

- 1- Manufacture of CQDs powder with in the nano scale by using lemon juice as a non-toxic source.
- 2- CQDs were successfully prepared from raw material using a one-step hydrothermal carbonization (HTC) procedure, as measured by TEM, FE-SEM, XRD, UV-vis, and FT-IR.
- 3- CQDs exhibited high antibacterial activity against types of bacteria (*Escherichia Coli Stander*, *Staphylococcus lentus*, *E-Coli*, *Staphylococcus aureus*, *Kokerocous Kristina*, *Enterococci faecalis*, and *Pseudomonas*).
- 4- The synthesized carbon quantum dots (CQDs) used as gas sensors, and that were responded well to NO<sub>2</sub> and NH<sub>3</sub> gases at different temperatures (200,250,300) ° C.
- 5- CQDs showed a good response and recovery times to NO<sub>2</sub> and NH<sub>3</sub> at temperatures 200 ° C and 250 ° C.
- 6- The contact angle for CQDs indicating that CQDs have high hydrophilicity and the CQDs with good water solubility.

### **3.4.2 Recommendations**

According to the results of the present study, various recommendations can be concluded as follows:

1. Raw materials such as (carbohydrate (glucose), strawberry juice, orange juice, sugar cane juice, chicken eggs and chitosan) and other sources can be further examined to investigate the probability of CQDs extraction.
2. High surface area of CQDs prepared can be combined with biologically active molecules as peptides and proteins for biosensor design.
3. Other natural products can be used as the source of the functional group (COOH, C=O).
4. Green trash can be exploited as a source of carbon quantum dots, resulting in innovative waste treatment technologies and a reduction in the financial and operational costs of garbage disposal.

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أبقال حببيات الكاربون الكمية من خالل فح الفعالية المضائية للبكتيريا لنقط

الكيمياء الغروية) كإضافة للبكتيريائية موجبة الجرام *Staphylococcus lentus* و  
*Staphylococcus aureus* و *Kokerococcus Kristina* و *Enterococcus faecalis*  
و *Escherichia coli* stander و *Escherichia coli* و *Pseudomonas* وأظطرت النتاصح أن CQDs صفاء مضائية  
للجراثيم أفضاء كإضافة *Kokerococcus Kristina* مع منطقة تاييط صال 35 مم (مقارنة  
بالبكتيريا الخرم).

حدمعلات التحايية وول رد الفع وول انقترجاع لكاش اةتسعار الغاز  
CQDs المصانعة بالانقترجاع و الازات  $\text{NO}_2$  و  $\text{NH}_3$  بالرجاءات حرارية مختلفة  
(222.252.322 درجة مئوية. % 222 Co) و بنا 22% (322 C) (لاي أوصي  
النيتروجين  $\text{NO}_2$  و  $\text{NH}_3$  بلق التوالي. صان و ل الكشاي و ل 23.35 ثانية لغاز  $\text{NO}_2$  و 33 ثانية لغاز  
 $\text{NH}_3$ . تم حاب و ل انقترداد ل  $\text{NO}_2$  بلق أة 11.93 ثانية و صان و ل انة التجابة 14.7 ثانية بن  
222 درجة مئوية بينما صان و ل انة التجابة لغاز  $\text{NH}_3$  هو 11.21 ثانية و ل انقترجاع صان 6.11  
ثانية بن 252 درجة مئوية. أن مجات CQD  
صال مالية بن هذال رجات الحرارية.

تم يال (قال الكم الكربونية المحبة للماء و الطارصاة للماء بواة الانقترجاع) ماال زاوية  
التلمس. زاوية التلمس لنقلا الكمية الغروية هي  $9.534^\circ$  مما يشاء لير هلق أن النقط الكمية  
الغروية تمثلو ابلية بالية للذوبان في الماء.



جمهورية العراق

وزارة التعليم العالي و البحث العلمي

جامعة الأنبار

كلية العلوم قسم

الكيمياء

التحضير الحيوي لنقاط الكربون الكمية من عصير الليمون ودراسة  
تطبيقاتها

رألة

مقمة هلق صلية العلوا- جامعة ا(بار  
وهي ج ء من متطلبات (ي) شطادي الماج تير في نلوا الكيمياء

من ب(الطالبة

أيات خميس فتيخان الفهداوي

بكلوريوس صيمياء ١٢٠١٨

إشراف

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صلية العلوا- جامعة ا(بار

أ.م.د عمار محمد حمزة  
م الطنة الم(ية- الجامعة التكنولوجية

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