

# **Optical and Structural Properties of Green Synthesized Graphene Quantum Dots**

A DISSERTATION

SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE  
AWARD OF THE DEGREE

OF

MASTER OF SCIENCE

IN

PHYSICS

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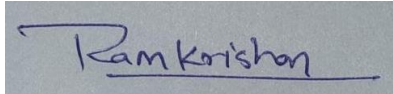
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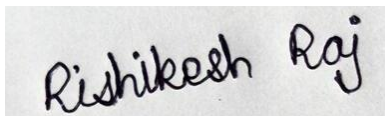
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## **SUPERVISOR CERTIFICATE**

I, hereby certify that the Project Dissertation titled “**Optical and Structural Properties of Green Synthesized Graphene Quantum Dots**” which is submitted by Ram Krishan Saini, Roll No. 2K21/MSCPHY/39 and Rishikesh Raj, Roll No. 2K21/MSCPHY/40 to the Department of Applied Physics, Delhi Technological University, Delhi in partial fulfilment of the requirement for the award of the degree of Master of Science, is a record of the project work carried out by the student under my supervision. To the best of my knowledge, this work has not been submitted in part or full for any Degree or Diploma to this University or elsewhere.

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WORD COUNT

**6434 Words**

PAGE COUNT

**32 Pages**

SUBMISSION DATE

**May 27, 2023 2:18 PM GMT+5:30**

CHARACTER COUNT

**36927 Characters**

FILE SIZE

**2.5MB**

REPORT DATE

**May 27, 2023 2:19 PM GMT+5:30**

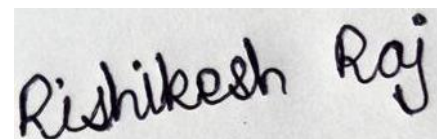
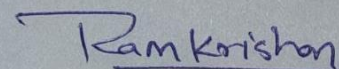
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## **ACKNOWLEDGEMENT**

I would like to express my indebtedness and deepest sense of regard to my supervisor, Dr. Mohan Singh Mehata, Assistant Professor, Department of Applied Physics, Delhi Technological University for providing his incessant expertise, inspiration, encouragement, suggestions, and this opportunity to work under his guidance. I am grateful for the constant help provided at every step of this project by Mr. Vinay Yadav and Mr. Aryan and all other lab members of Laser Spectroscopy Laboratory, Department of Applied Physics, Delhi Technological University. I am also thankful to my family and colleagues for their invaluable support, care and patience during this project. Lastly, I would thank Delhi Technological University for providing such a wonderful opportunity of working on this project.

## ABSTRACT

Hydrothermal synthesis of Graphene quantum dots (GQDs) was done using corn powder as a carbon source and water as a solvent. A high-resolution transmission electron microscope shows uniform size distribution with an average diameter of 42 nm. Fourier transform infrared analysis of synthesized GQDs indicates the presence of C=C bond, O-H groups, and aromatic rings. The absorption spectrum of GQDs shows a sharp band at around 227 nm, corresponding to the  $\pi$ - $\pi^*$  transition, and a shoulder peak at 283 nm assigned to the n- $\pi^*$  transition. The band gap of produced GQDs corresponding to 283 nm is 4.07 eV. When the GQDs are excited at 350 nm, the photoluminescence (PL) spectrum with a peak maximum of 450 nm was observed. The band maximum shifted blue of about 100 nm when excited with the band edge of absorption. Hence the PL is excitation dependent. The GQDs could be used as a potential candidate for hazardous metal ions detection in drinking water, and as a catalyst in degrading hazardous dye in wastewater treatment.



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## LIST OF SYMBOLS AND ABBREVIATIONS

Symbol/Index	Meaning/Abbreviation
GQDs	Graphene Quantum Dots
HR-TEM	High Resolution Transmission Electron Microscope
PL	Photoluminescence
0D	Zero-dimensional
2D	Two-dimensional
$\pi^*$	Anti-bonding $\pi$ -orbitals
CDs	Carbon dots
QY	Quantum Yield
GO	Graphene Oxide
FTIR	Fourier Transform Infrared Analysis
UV	Ultraviolet
h	Planck's constant
$\nu$	Frequency

## **CHAPTER 1**

### **INTRODUCTION**

#### **1.1. Nanotechnology:**

Nanotechnology is a rapidly growing field dealing with the study and modification of matter at the nanoscale, which is approximately 1 to 100 nanometres. At this scale, the physical, biological and chemical properties of substances change dramatically, resulting in unique properties and potential applications [1]. Nanotechnology has the potential to transform from electronics and energy to medicine and environmental science [2].

One of the significant applications of nanotechnology is in electronics. Nanoscale materials, such as graphene and carbon nanotubes, are being used to create faster, smaller, and more energy-efficient electronic devices [3]. The electric properties of these materials are unique which makes them ideal for use in electronic components, such as transistors, sensors, and memory devices. Nanotechnology is being used to create new materials with unique properties that can be used in various applications. For example, the creation of quantum dots, which are nanoscale semiconductor particles, has opened up new possibilities in areas such as electronics, optics, and quantum computing [4-5].

In the field of medicine, nanotechnology has the potential to revolutionize the diagnosis and treatment of diseases. Nanoparticles can be designed to target specific cells or tissues in the body, allowing for more precise drug delivery and reduced side effects. Nanoparticles can also be used to create new diagnostic tools that are more sensitive and accurate than current technologies. For example, gold nanoparticles can be used to detect cancer cells in the early stages [5-7].

Nanotechnology is also being explored for its potential applications in energy production and storage. Nanoscale materials, such as quantum dots and nanowires, can be used for creating solar cells and batteries with better efficiency. Nanotechnology can also be used for improving the efficiency of existing energy technologies, such as fuel cells and wind turbines [7].

In material science, nanotechnology is being used to create new materials with enhanced properties. For example, carbon nanotubes and graphene are being used to create stronger, lighter, and more conductive materials that can be used in various applications, from electronics to aerospace. Nanoparticles can also be used to create new coatings and films with unique properties, such as self-cleaning or anti-corrosive properties.

In engineering, nanotechnology is being used to create new devices and systems with enhanced performance. For example, nanoscale sensors and actuators can be used to create more precise and efficient control systems for various applications, from manufacturing to robotics. Nanotechnology is also being used to create new energy technologies, such as solar cells and fuel cells, that can be more efficient and cost-effective than current technologies [8-10].

In environmental science, nanotechnology is being used to develop new solutions for water treatment, air purification, and pollution control. Nanoparticles can be used for removing contaminants from water and air, and nanomaterials can be used to create more efficient catalysts for chemical reactions. Nanotechnology is also being used to develop new sensors and monitoring systems that can detect and track environmental pollutants at the nanoscale [10-12].

Nanotechnology is being used to develop new diagnostic and therapeutic tools that can target specific cells or tissues in the body. Nanoparticles, such as liposomes and

dendrimers, can be used to deliver drugs or genetic material, reducing the side effects of traditional treatments. Nanotechnology is also being used to develop new sensors and imaging techniques that can detect and monitor biological processes at the nanoscale.

Despite its many potential applications, nanotechnology also raises concerns about its potential impact on the environment and human health. As such, it is essential to regulate and oversee the development and use of nanotechnology to ensure that its use is safe and responsible [13-14].

Overall, nanotechnology has the potential to revolutionize various fields and create new possibilities for science, technology, and society. However, it is essential to balance the potential benefits with the potential risks and ensure that the development and use of nanotechnology are safe and responsible.

## **1.2. Graphene Quantum dots (GQDs)**

GQDs, being to a novel category of low-dimensional materials that exhibit distinctive physical and chemical properties. This makes them particularly appealing for diversified applications, such as electronics, biology, energy, and environmental science. A relatively recent addition to the family of carbon-based materials is GQDs. They are minute pieces of graphene with dimensions under 100 nm. They possess features that are a result of both GQDs and carbon dots [1-2]. GQDs are essentially a sort of zero-dimensional (0D) material that can be thought of as incredibly tiny graphene fragments [3-5]. They stand out from their close relatives, carbon dots, by containing a graphene structure inside each dot, excluding its size. As a result, GQDs exhibit certain exceptional behavior of graphene, making them an object of significant research interest [6-7].

In recent years, scientists have successfully developed GQDs using the same principles as two-dimensional (2D) graphene. GQDs that show quantum confinement and edge

effects are typically made up of one or more layers. These substances have a variety of appealing characteristics like biocompatibility, high surface area, good stability and adjustable cytotoxicity, tunable PL, small molecular size, chemical inertness and ease of surface grafting and functionalization [1,3,8–14].

Fluorescent nanomaterials known as GQDs have recently been discovered. They have unique optical features and great potential for use in bioimaging-based disease detection and treatment. This is brought about by their 0D structure, which also contributes to solubility, biocompatibility, and chemical stability. They are especially appealing for biomedical applications because of these characteristics [15], [16].

In electronics, GQDs have been studied as potential components in solar cells [17–19], photodetectors [20–21], and field-effect transistors [22–26], due to their photo response and high charge carrier mobility. They have been utilized in biomedicine as biosensors and for targeted medication delivery because of their biocompatibility and substantial surface area for chemical functionalization [27–36]. In energy, they have been studied as catalysts for fuel cells and hydrogen evolution reactions, and in environmental science, they have served as adsorbents for organic pollutants [37–45].

The  $\pi$ - $\pi^*$  transition of C=C makes carbon dots (CDs) as well as GQDs an efficient photon absorber within a small-wavelength range. Normally, they typically exhibit significant optical absorption between 260 and 320 nm, with considerable absorption reaching the visible spectrum. In general, CDs are more effective than GQDs at absorbing long wavelengths. Many GQDs show a peak within 270–390 nm, in contrast to CDs, which may be brought on by the C=O's transition ( $n$ - $\pi^*$ ). Functional groups and surface passivation cause changes in both materials' absorption properties [46–52]. The tunable photoluminescence (PL) features deriving from quantum confinement phenomena are the

most intriguing aspect of both carbon nanodots [9]. The PL intensity fluctuates together with the excitation wavelength, and the redshifts nearly span the visible spectrum as a result. The most thorough and convincing model to describe it is the quantum confinement effect. Surface emissive traps cause bare CDs' poor PL quantum yield (QY). For boosting their brightness, a surface passivation layer is therefore needed. Hence, a surface passivation layer is needed to boost their brightness. Because of their multilayer structure and increased crystallinity, GQDs often provide higher QY than bare CDs. The high QY PL of GQDs is a result of their crystallinity and layer structure. Electrical conductivity is significantly affected by quantum confinement and an edge effect in GQDs thanks to the possibility of high-speed electron mobility. GQDs have high electron mobility and a fast reaction rate, which make them excellent choices for sensing applications [6], [12].

### **1.3. Synthesis method of GQDs**

Bottom-up and top-down are two categories of GQD synthesis methods differing in their reaction mechanisms. In first strategy, large amounts of graphene are exfoliated into smaller particles using physical or chemical techniques like mechanical milling, oxidative cleavage [53,54], nanolithography [55–57], hydrothermal or solvothermal [58–61], microwave irradiation [62-64], laser ablation [65], sonication assisted [66], electrochemical [67,68], or chemical exfoliation [69,70]. This method can result in GQDs with a wide size distribution and a lot of functionalization, but it might also cause oxidation and structural damage.

In the second strategy, organic molecules are controlled pyrolyzed or carbonized, or controlled  $sp^2$  carbon structures are synthesized from organic polymers. This process entails the growth of GQDs from precursors like carbon, metal, or organic precursors.



Chemical vapour deposition can be used for developing graphene on metal substrates and then transferring it for exfoliation. This method can result in GQDs with controlled sizes and compositions, but it might also introduce undesirable impurities and defects. This method produces quantum-sized dots by first carbonizing tiny molecules that have undergone dehydration. There are many different techniques to carry out this synthetic process, including hydrothermal [71], plasma hydrothermal [72], microwave [73], combustion [74], pyrolysis in acid [75], microwave-assisted hydrothermal [76], microreactor-based synthesis [77], etc.

#### **1.4. Eco-Friendly Synthesis of Graphene Quantum Dots: Green Approaches and Raw Materials**

The conventional methods for the synthesis of GQDs often involves toxic chemicals, which can pose risks to human health and the environment. Therefore, researchers have been exploring alternative methods for synthesizing GQDs using eco-friendly raw materials and green approaches [2].

Eco-friendly raw materials are those that are natural, renewable, and sustainable, and can be obtained without damaging the environment. These include plant-based materials, such as fruits, vegetables, and biomass waste, which can be converted into graphene oxide (GO) through a simple oxidation process. The GO can then be reduced to GQDs using eco-friendly reducing agents, such as ascorbic acid, sodium borohydride, or green tea extract. Green solvents, such as water or ethanol, can be used to dissolve the precursors and facilitate the synthesis process [78-79].

Green approaches to GQD synthesis involve the use of non-toxic and environmentally friendly materials and methods that minimize the use of hazardous chemicals and reduce waste generation. These include hydrothermal, microwave-assisted, electrochemical, and

photochemical methods, as mentioned earlier. These methods have several advantages over conventional methods, including reduced toxicity, lower cost, and higher sustainability. Furthermore, the resulting GQDs can have improved biocompatibility and can be used in bioimaging, drug delivery, and biosensing [16].

The development of eco-friendly raw materials and green approaches for GQD synthesis is an important step towards sustainable and responsible nanotechnology. By reducing the use of toxic chemicals and waste generation, these approaches can help to minimize the environmental impact of nanotechnology while also improving the safety and efficacy of GQDs in various applications. Moreover, these approaches can promote the use of renewable and sustainable resources, which is crucial for addressing global challenges such as climate change and resource depletion [2-16].

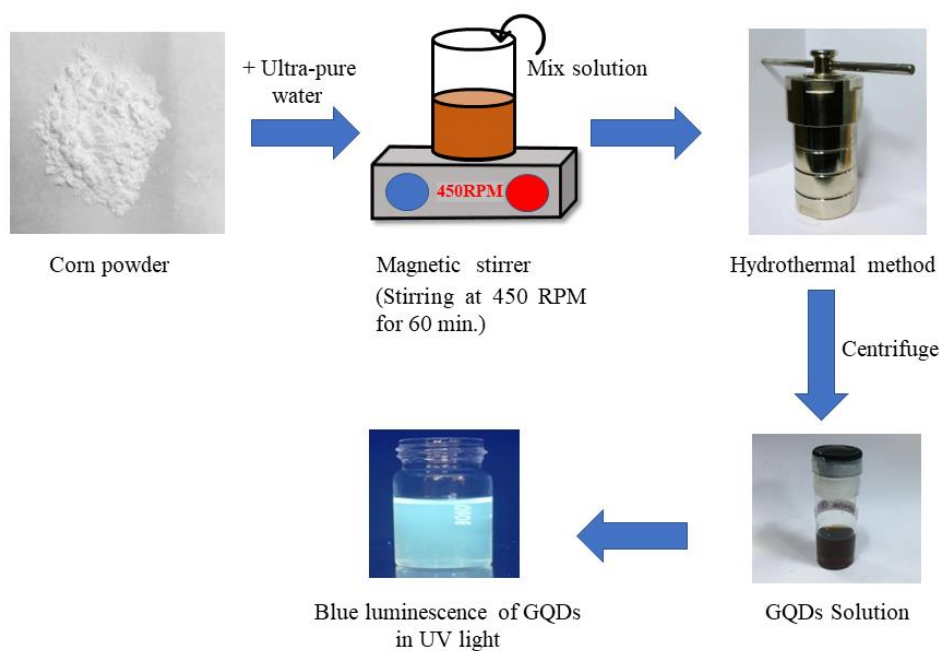
Green synthetic routes are being used as a recently growing research area in the field of nanotechnology. In the preparation of GQDs, strong acids or organic solvents are required and green production involves non-toxic reagents but it still has to confront some challenges; that's why, synthetic routes which are not harmful to the environment and do not involve complicated processes should be discovered and developed.

## CHAPTER 2

### SYNTHESIS & CHARACTERIZATION TECHNIQUES

#### 2.1. SYNTHESIS OF GQDS

An innovative and non-toxic precursor (corn powder) was used in the hydrothermal process to create GQDs. Initially, 50 ml DI water and 0.5 g corn powder were mixed and stirred, afterward, it was subsequently placed into an autoclave made of stainless steel and heated for eight hours at 180°C. It was subsequently placed into an autoclave made of stainless steel and heated for eight hours at 180°C. The brown solution was obtained, which was further filtered or GQDs were separated from the solution by rapid centrifuge at 7500 rpm for 15 minutes [78,79].



**Figure 2.1.** Schematic diagram of the synthesis route of GQDs using eco-friendly and cost-effective route

## 2.2. CHARACTERIZATION TECHNIQUES

TALOS (Accelerating voltage 200 kV) (Fig. 2.1), was used for TEM images, while FTIR spectroscopy within the range of 400 - 4000  $\text{cm}^{-1}$  was conducted with Perkin Elmer Two-Spectrum FTIR spectrometers (Fig.2.2) of the obtained GQDs. The UV-Vis spectroscopic data were recorded by a Perkin-Elmer Lambda-750 dual-beam spectrometer (Fig. 2.3). Steady-state PL and PL-excitation measurements were done using Fluorolog-3 spectrofluorometer from Horiba Jobin Yvon (Fig. 2.4), having equipment (photomultiplier tubes and xenon lamp). The measurements were done with a cuvette made of quartz having 10 mm of the optical path.



**Figure 2.2.** Photograph of TALOS thermo-scientific instrument (Acc. Vol. 200 kV)



**Figure 2.3.** Photograph of Perkin Elmer Two-Spectrum FTIR spectrometers



**Figure 2.4.** Photograph of Perkin Elmer Lambda 750 UV/Vis/NIR spectrophotometer



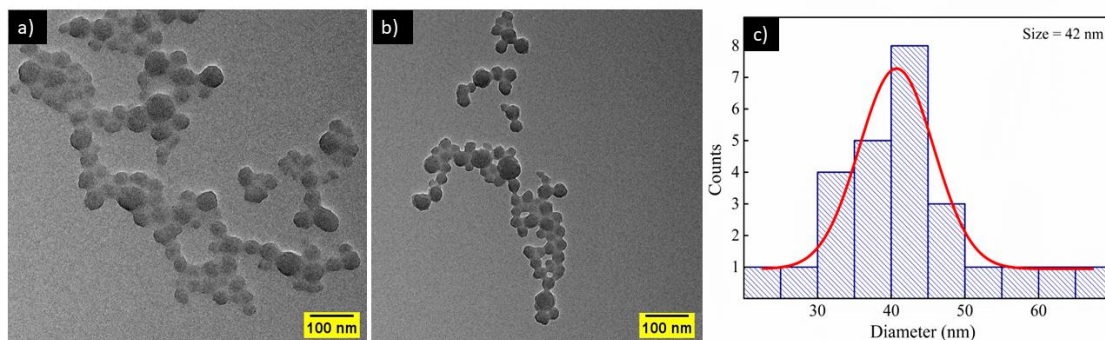
**Figure 2.5.** Photograph of Horiba Jobin Yvon Fluorolog-3 spectrofluorometer

## **CHAPTER 3**

### **RESULTS AND DISCUSSION**

#### **3.1. TEM Analysis**

The GQDs were subjected to TEM analysis to investigate their size, shape, and uniformity. The GQDs were then drop-cast onto a carbon-coated grid after that it was left for drying before TEM imaging. Fig. 3.1 displays TEM images of the GQDs having a spherical shape and the diameter is within 20 -50 nm. (Fig. 2 c) confirms an average size of 42 nm, having 5 nm of standard deviation [80–82]. ImageJ software was used further for analyzing GQDs' size distribution. It revealed that the size of GQDs is consistent and there is a small range of variation in their sizes. The observed size range is as per previous reports on GQDs, and its shape as observed from TEM images is consistent with spherical morphology, as previously reported for GQDs [83]. The uniformity of GQDs as observed from the TEM images is following the uniformity observed in its size distribution analysis. These results are consistent with previous reports on GQDs which highlight their potential for use in numerous applications, including bioimaging, energy storage, and catalysis. However, it should be noted that there are potential limitations to TEM analysis, including sample preparation and imaging artifacts, which should be taken care of while interpreting the results [84].

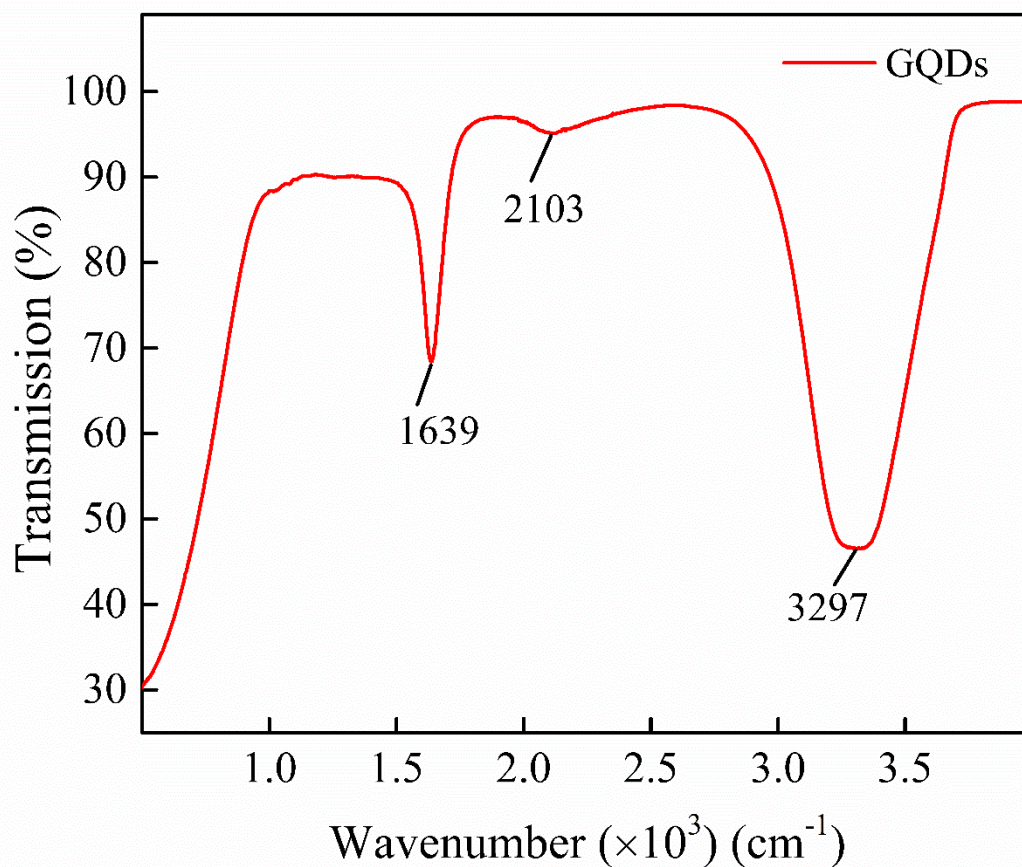


**Figure 3.1.** TEM images of GQDs at the magnification of 100 nm (a &b) along with particle size distribution (c).

### 3.2. FT-IR Analysis

The synthesized GQDs were investigated using FTIR, especially to find out functional groups, bonds and chemical composition, and the observed FTIR spectrum is shown in Fig. 3.2. The broad band with a peak at  $3297\text{ cm}^{-1}$ , may be attributed to O-H stretching vibration of hydroxyl groups. A weak band at  $2103\text{ cm}^{-1}$  is also observed, which may be attributed to the aromatic compounds. A band at  $1639\text{ cm}^{-1}$  may be attributed to the C=C stretching vibration of carbonyl groups [85]. The FTIR spectrum of the GQDs indicates various group's presence on their surface and this could be the reason behind the distinctive characteristics and potential applications of GQDs. The hydroxyl group's presence suggests that the GQDs may have good water solubility and biocompatibility. Overall, this analysis validates the functional group's presence on GQDs' surface and also offers an understanding of their chemical makeup. These results can inform further investigations into the properties and potential applications of GQDs [86–89].



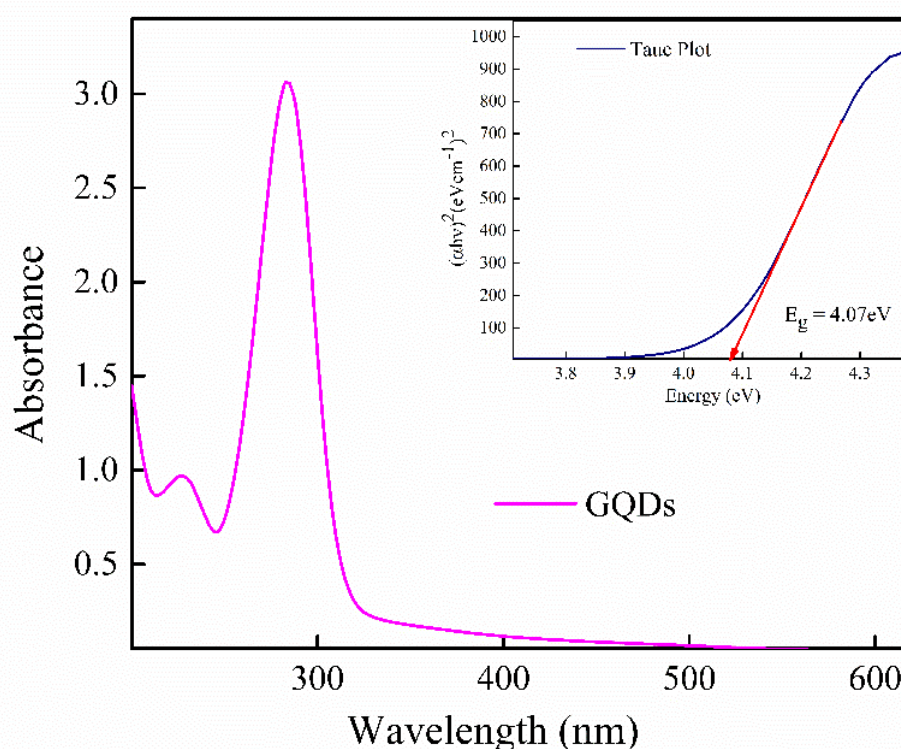


**Figure 3.2.** FTIR spectrum of green synthesized GQDs

### 3.3. Absorption Spectrum

The absorption spectrum of GQDs typically shows strong absorbance in the UV-Vis region because of  $\text{sp}^2$  hybridization of carbon bonds in the graphene layer. The exact shape and position of the absorption vary depending on their structure and functionalization as well as the solvent used for dispersing the GQDs. In general, smaller GQDs exhibit stronger and narrow peaks compared to larger GQDs. Additionally, the functionalization of the GQDs with various chemical groups can also affect their absorption properties [91,92].

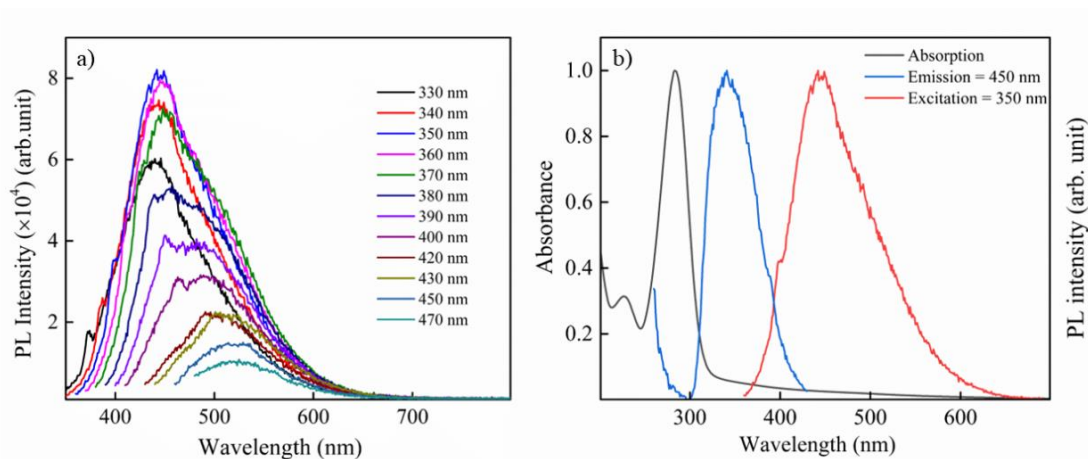
The GQDs absorption spectrum (Fig. 3.3) exhibits two distinct excitonic absorption peaks at approximately 227 and 283 nm as a result of the  $\pi-\pi^*$  and  $n-\pi^*$  transitions of C=C and C=O groups, respectively [48,90–94]. The longer wavelength band which arises as 283 nm appeared prominently. Tauc's plot was used for calculating the optical band gap and it is described as a plot of the converted absorption coefficient  $(\alpha h\nu)^r$  versus the photon energy ( $h\nu$ ). Here,  $h$  is Planck's constant,  $\alpha$  is absorption coefficient and  $\nu$  is light frequency. The appropriate optical band gap can be estimated after determining the value of  $R$ , which is  $1/2$  for materials with an indirect band gap and  $2$  for those with a direct band gap. (Fig. 3.3) When  $r = 2$ , displays a good linear fit, supporting that the synthesized GQDs are direct band gap materials ( $r = 1/2$  does not provide a good linear fit). From Fig. 3.3, we observed the band gap energy ( $E_g$ ) to be 4.07 eV, and this was accomplished by determining the point at which a line, extrapolated from the range of the curve, intersects the x-axis [95,96].



**Figure 3.3.** UV-Vis absorption spectrum and Tauc plot of  $(\alpha h\nu)^2$  vs  $h\nu$  (inset) of synthesized GQDs

### 3.4. Photoluminescence Spectra

Figure 3.4(a) displays the PL spectra of GQDs within the 330–470 nm wavelength region. The PL of the GQDs is another appealing quality although the mechanism behind the PL phenomenon is still not completely understood, it is thought to involve free zig-zag sites with a triplet state resembling a carbene and surface flaws in the functional groups of the GQDs, quantum effects, edge structure, doping and electron-hole recombination [97,98]. Fig. 3.4(b) shows a broad emission at 450 nm with the excitation wavelength at 350 nm. As the excitation wavelength was increased, there was a rise in the PL intensity before it began to decrease [50]. Notably, the intensity of the PL was the weakest when 330 nm wavelength was used for excitation, and it was the highest when 450 nm wavelength light was used. GQDs emitted an intense blue light when exposed with a wavelength of 365 nm and the band maximum shifted by about 100 nm when excited with the band edge of absorption, indicating that the PL is excitation-dependent, which might be due to size distribution and different emitting centers [99–102].



**Figure 3.4.** PL spectra with varying excitation wavelengths from 330 to 470 nm (a) and a combination of absorption spectrum (black line), PL-excitation spectrum (blue line) with 450 nm PL, and PL spectra with 350 nm excitation (red line) of the GQDs (b).

## **CHAPTER 4**

### **CONCLUSION**

A green method has been used to successfully create GQDs, utilizing corn powder as a precursor. The GQDs exhibited a spherical shape with an average size of 42 nm. The presence of hydroxyl and carbonyl groups, as well as aromatic compounds, was confirmed using FTIR. The GQDs showed promising optical properties with two distinct excitonic absorption bands and intense blue PL. Extensive research is needed to fine-tune the characteristics of GQDs to maximize their potential for different uses, such as bioimaging, sensing, and optoelectronics.

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## CONFERENCE RECORD

### CERTIFICATE OF PARTICIPATION

	<b>ATMA RAM SANATAN DHARMA COLLEGE</b> <b>(University of Delhi)</b> Accredited Grade 'A' By NAAC All India 7 <sup>th</sup> Rank in NIRF (MHRD)	
Certificate No: AFMD-2023/OP/045		
<b><i>2<sup>nd</sup> International Conference on</i></b> <b><i>"Advanced Functional Materials &amp; Devices" (AFMD-2023)</i></b>		
<b>Organized by</b> <b><i>IQAC &amp; Department of Physics, ARSD College</i></b>		
This is to certify that Prof./Dr./Mr./Ms. <b>Rishikesh Raj</b> from <b>DELHI TECHNOLOGICAL UNIVERSITY</b> has <b>presented</b> his/her research work as <b>Oral Presentation</b> in <b>2<sup>nd</sup> International Conference on "Advanced Functional Materials and Devices" (AFMD-2023)</b> organised by IQAC & Department of Physics, ARSD College, University of Delhi during 13-15 <sup>th</sup> March 2023 via online mode.		
 <b>(DR. SHANKAR SUBRAMANIAN)</b> CONVENER	 <b>(DR. VINITA TULI)</b> CONFERENCE CHAIR	 <b>(PROF. GYANTOSH KUMAR JHA)</b> PRINCIPAL

## PROOF OF SCOPUS INDEXING

### AFMD-2023 Invitation

After the successful completion of AFMD-2021, it gives us immense pleasure to invite you to attend the 2<sup>nd</sup> International conference on Advanced Functional Materials and Devices (AFMD-2023) to be organized online during March 13-15, 2023. This conference is being organized in succession with previous event held in March 2021. The principal motive of AFMD-2023 is to provide a common platform for all the researchers, academic personnel's, scientist, research students from all over the world to brainstorm, discuss and share their new achievements, ideas, results and new innovations regarding new functional materials for device purposes.

**Technical Sessions:** The symposium will comprise of plenary, invited and contributed papers. Contributed papers will be presented orally as well as during poster session covering recent developments in the areas listed above.

**Presentations:** Keynote lectures will be of 45 minutes duration. Duration of invited talk and oral presentation will be 30 mins. and 15 mins. respectively.

**Publications:** Abstract book will be published. Selected peer reviewed papers will be published in Scopus / Web of science indexed International journal/ Book Proceedings of repute.

### Scope of symposium

(Not limited to the following):

- Sensors, Transducer, flexible electronics
- Fuel cells, solar cells and energy storage devices
- Materials for Energy Harvesting Applications
- Bulk and composite materials
- Materials for Biomedical Applications
- Systems for Environmental studies
- Applications of Carbon Nanomaterials
- Materials for Photonics and Optoelectronics
- Applications of Magnetic Materials
- Materials with Multifunctional Properties
- Theoretical methods for nanomaterials and nanotechnology
- Physics and application of heterostructures and superlattices
- Biocompatible systems for medical applications
- Advanced materials for Military applications
- High energy and particle physics

Submit abstract @

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### Important Dates

**Abstract Submission Opens:** Dec 10, 2022

**Abstract Submission Ends:** Feb 10, 2023

**Acceptance Notification:** Feb 20, 2023

**Registration Opens:** Jan 01, 2023

**Conference Commences:** March 13, 2023

### Registration

Category	Early bird registration (Before 01/03/2023)		After 01/03/2023	
	Indian (INR)	Foreign (USD)	Indian (INR)	Foreign (USD)
Academician/ Researcher	1000	75	1500	100
Students	500	50	750	75

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