PRODUCTION AND CHARACTERIZATION OF GRAPHENE QUANTUM DOTS

A DISSERTATION

SUBMITTED IN PARTIAL FULFILMENT OF REQUIREMENTS
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IN
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DECLARATION

I, **SHIVANG KUMAR MISHRA (23/MSCPHY/69) AND MAYANK SRIVASTAVA (23/MSCPHY/32)** hereby certify that the work which is presented in Dissertation-II entitled **"Production and Characterization of Graphene Quantum Dots"** in fulfilment of the requirement for the award of the degree of Master of Science in Physics and submitted to the Department of Applied Physics, Delhi Technological University, Delhi is an authentic record of my own, carried out during a period from July 2024 to May 2025 under the supervision of **Dr. Mohan Singh Mehata** .

The matter presented in this report/thesis has not been submitted by us for the award of any other degree of this or any other Institute/University.

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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. I further certify that the publication and indexing information given by the student is correct.

9/6/2025

Place: Delhi

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ABSTRACT

Graphene quantum dots (GQDs) have great potential due to their advantageous characteristics of non-zero band gap, highly tunable property, biocompatibility, and ability to accommodate functional groups and dopants. Herein GQDs were synthesized by bottom-up approach of cost-effective hydrothermal technique using grass waste as a feasible carbon source. The obtained GQDs were confirmed through different characterization techniques. The structural properties were studied by X-Ray Diffraction whereas morphology of GQDs were examined through Transmission Electron Microscopy. The Optical characteristics were studied using Absorption spectroscopy and Photoluminescence while Fourier Transform Infrared Spectroscopy confirms the presence of functional groups and chemical bonds within GQDs.

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LIST OF SYMBOLS AND ABBREVIATIONS

GQDs	Graphene Quantum Dots
PL	Photoluminescence
DI	Deionized
XRD	X-RAY DIFFRACTION
FTIR	FOURIER TRANSFORM INFRARED
HRTEM	HIGH RESOLUTION TRANSMISSION ELECTRON MICROSCOPY
EDX	ENERGY DISPERSIVE X-RAY
PMT	PHOTO MULTIPLIER TUBE

CHAPTER -1

INTRODUCTION

1.1 NANOTECHNOLOGY

Human fascination with small objects and the belief that they had greater potential led to the miniaturization of technology, resulting in the development of laptops and palmtops as alternatives to desktop computers, as well as microchips capable of controlling these devices. The world is now seeking increasingly compact and more efficient products than those available now.

Scientists assert that nanotechnology is expected to progress through four distinct generations of development. We are presently encountering the first or possibly second generation of nanomaterials. The initial generation of materials possesses characteristics attained by the integration of "passive nanostructures" [1].

The study of events that occur on a nanoscale scale is known as nanoscience. At least one dimension of a structure or material lies between 1 and 100 nanometres. The discussion that physicist Richard Feynman gave in 1959 is considered the catalyst for the development of nanotechnology. He imagined the possibility of synthesis through the direct manipulation of atoms in his work "**There's Plenty of Room at the Bottom."** The term "nanotechnology" refers to the process of manipulating matter to the extent that at least one dimension lies on the nanoscale. The technology in question is one that allows for the manipulation of matter on an atomic or molecular scale.

Nanotechnology is the process of developing and fabricating functional items at the nanoscale. We refer to the science that underpins nanotechnology as nanoscience. The interdisciplinary character of nanotechnology perpetuates innovation, providing various applications in medicine, energy efficiency, and environmental solutions. This highlights the capability of altering materials at the nanoscale to tackle intricate issues across many demanding fields. Nanoscale particles differ from macroscale particles in four distinct ways:

At the nanoscale, gravity forces become insignificant, but electromagnetic forces prevail. At the nanoscale, quantum physics surpasses conventional physics in describing motion and energy. Increased surface-to-volume ratio at the nanoscale, Random (Brownian) molecular motion gains significance at the nanoscale.

1.2 NANOMATERIALS

Nanomaterials are increasingly significant for the comprehensive advancement of humanity. Scientists use nanomaterials to develop instruments for diagnosing and managing pandemic diseases that occur globally.

In order to control the highly infectious disease that swept across multiple nations, Covid-19, scientists managed to use nanomaterials in 2019 [2].

Nanomaterials are utilized for many applications in construction and architecture. In 2020, around 800 building products utilizing nanotechnology were developed [3].

1.3 NANOPARTICLES

Nanoparticles consist of nanoparticles that have been constructed, metal-organic frameworks, and core/shell structures. Using organic and inorganic nanoparticles, polymers, and ceramics, various characteristics and enhanced chemical activity are achieved.

Nanoparticles vary in size, shape, dimension, and composition. Nanoparticles may possess zero dimensions if their three spatial dimensions are confined inside the nanometric scale. The term ‘three-dimensional’ implies that there are 0D, 1D and 2D elements within the dense, inter contacting surfaces, which are applicable to the nanometric grains in dense polycrystals or 3D porous nanostructures [4].

1.4 QUANTUM DOTS

The degree of confinement rises as QD size decreases, creating a higher energy exciton that raises the band gap energy. Emissions will be directed into the blue region with shorter wavelengths due to large energy of the band gap. The continuous energy levels spectrum of bulk material turns out to be discrete at the nanoscale [5].

QDs possess a substantial surface area, hence ensuring an elevated drug loading capacity and the ability to label the nanocarriers within biological systems. Quantum dots are chemically inert and gave positive response specifically for theragnostic drug administration [6].

“Quantum dot” is defined as a substance exhibiting quantum confinement effects in all dimensions. Quantum dots (QDs) are nanoparticles that demonstrate quantum confinement characteristics and exhibit PL emission that is dependent on size [7].

1.5 GRAPHENE

Graphene, this automatically thin carbon allotrope has ignited a revolution in materials science, captivating researchers across all disciplines. A flat layer of carbon atoms in a hexagonal pattern; graphene is the fundamental unit of graphitic carbon compounds. This produces a densely packed and robustly bound confer an exceptionally high tensile strength, Simultaneously, graphene is a lightweight, low-density material [8,9,10].

Despite its significant advantages it encounters inherent constraints like zero bandgap and relatively low absorptivity [11].

Graphite is primary source of graphene. Additionally, organic compounds serve as significant sources for graphene formation. The utilization of waste materials for the synthesis of graphene-like structures is significantly valuable for its environmentally sustainable and cost-effective approach [12]

Its fundamental character, which allows different functionalisation, making it a building block for nanomaterials such as GQDs [10].

1.5.1 GRAPHENE QUANTUM DOTS

Recently, GQDs have emerged as a prominent carbon-based material, attracting significant interest from researchers worldwide due to their distinctive and remarkable features to investigate and synthesize GQDs through rapid, straightforward and sustainable methods that minimize the production of poisonous by-products that may be detrimental to the ecosystem [13].

With their inexpensive precursors and basic manufacturing methods, GQDs are in high regard [14]. Quantum dots are like shape shifters; you can accordingly observe variation in properties just by tuning the size.

Researchers can functionalize graphene quantum dots (GQDs) due to high surface-to-volume ratio and sp^2 network, facilitates easy stacking. Scaling down from macroscopic to quantum confined nanostructures and edge induced events arising in the formation of a finite band gap leads to photoluminescence characteristics [15,16].

GQDs have emerged as a novel fluorescence system owing to their notable attributes, including low cytotoxicity, exceptional solubility, chemical inertness, and stability of PL [17].

Inorganic metal-based quantum dots have been documented during the past few decades and utilized as multifunctional biosensors. Following extensive research, these metal-based quantum dots have now been supplanted by graphene quantum dots. The superior biocompatibility and surface functionality render them suitable candidates for future functionalization after their use in bioimaging, sensing, and drug administration [18].

The quantum confinement effect determines energy required to promote electron-hole pairs from their ground state to elevated energy bands, which directly influences both the wavelengths of light that GQDs can absorb and the range of wavelengths they emit, with these optical characteristics being tied to the material's bandgap energy [19].

CHAPTER 2

NANOMATERIALS AND SYNTESIS METHODS

2.1 SYNTHESIS METHODS OF NANOPARTICLES

BOTTOM-UP: These aim to organize simpler elements into complex structures. Operate through physical effects or chemical activity at the nanoscale to construct fundamental units into more complex structures.

TOP-DOWN: These aim to fabricate tiny devices by utilizing larger ones to guide their construction. The predominant top-down fabrication method uses lithographic patterning techniques utilizing short-wavelength light sources. Diverse physical and chemical techniques have been developed for the creation of nanoparticles with specific shapes and sizes. However, these methods are neither economically viable nor environmentally sustainable. Green synthesis is advantageous due to its biocompatibility, low toxicity, and environmental friendliness. Furthermore, these technologies exhibit greater compatibility for medical applications than physical and chemical procedures, which may result in the adsorption of hazardous compounds on the surface of nanomaterials, thereby causing negative consequences when utilized for medical purposes.

2.2 GREEN SYNTHESIS OF GQD

A noticeable shift in research priorities highlights the role of biomass conversion in converting materials because of their splendid natural application varieties and currently seeking materials that are environmentally sustainable and cost-effective.

A bottom-up technique is superior to a top-down strategy, as the former yields fewer surface defects on GQDs and achieves a higher production yield. The bottom-up technique additionally encompasses the synthesis of GQDs using hydrothermal treatment [18].

Green-synthesized nanomaterials have superior structural characteristics, including high crystallinity, narrow size distribution, and exceptional monodispersed. Furthermore, recent advancements have concentrated on the recycling of biomass waste due to its detrimental effects on the environment. Consequently, the green synthesis method is gaining significance due to its low cost, sustainability, eco-friendliness, and capacity to reduce environmental impact [20].

2.3 BIO-WASTE AS A PRECURSOR

The utilization of renewable, cost-effective, and environmentally friendly biomass resources meets the urgent need for large-scale manufacturing of biomass-derived GQDs and promotes sustainable applications. Grass, as a component of biomass, represents a viable carbon source for GQDs. Consequently, the conversion of grass waste products into valuable substances is in high demand, especially as a precursor [21].

Consequently, biomass, characterized by its affordability, renewability, environmental sustainability, and high carbon content, emerges as a suitable precursor for the economical synthesis of GQDs. Various plants have distinct microstructures and compositions as a result of their diverse environments and variations. These disparities may affect the composition of their derivative carbon compounds [22]. In the past five years, green methodologies have emerged that utilize biomass waste and convert natural, cost-effective organic molecules for the synthesis of nanoparticles or quantum dots [23].

CHAPTER 3

SYNTHESIS PROCESS

3.1 HYDROTHERMAL METHOD

An organic solution serves as the precursor and is enclosed in a hydrothermal reactor under elevated temperatures [24].

The hydrothermal method is an efficient and speedy technique for synthesizing GQDs. This can ultimately be synthesized from various macromolecular or tiny molecular precursors via high temperature and pressure conditions [25].

Hydrothermal synthesis utilizes a sealed autoclave for high pressure and temperature, facilitating the formation of superior crystal shape.

Hydrothermal synthesis significantly affects the production and particle size of GQDs. DI water, utilized as solvent, is crucial for atom-economical processes [26].

3.2 MATERIALS USED

Grass waste was obtained near the science block of DTU, along with deionized water and a mechanical grinder.

3.3 SAMPLE SYNTHESIS

At the outset, Grass waste was subjected to boiling in distilled water and later grinded mechanically. Place it in an oven and dry it for 10 hours at 90^{o} C. Utilize a hand grinder to grind up the desiccated grass. Measure 60 ml of DI water in a beaker and add 0.2261 grams of ground grass. Subject it to sonication for 30 minutes at room temperature. Place 60 ml solution in a Teflon-coated autoclave and insert it into the hydrothermal apparatus. Maintain the hydrothermal in an oven at 200^{o}C for 9 hours. After completion of this method, centrifuge the resultant solution at 6000 rpm for 10 minutes, thereby isolating it from larger impurities that may have passed through the filter paper.

3.4 SCHEMATIC DIAGRAM

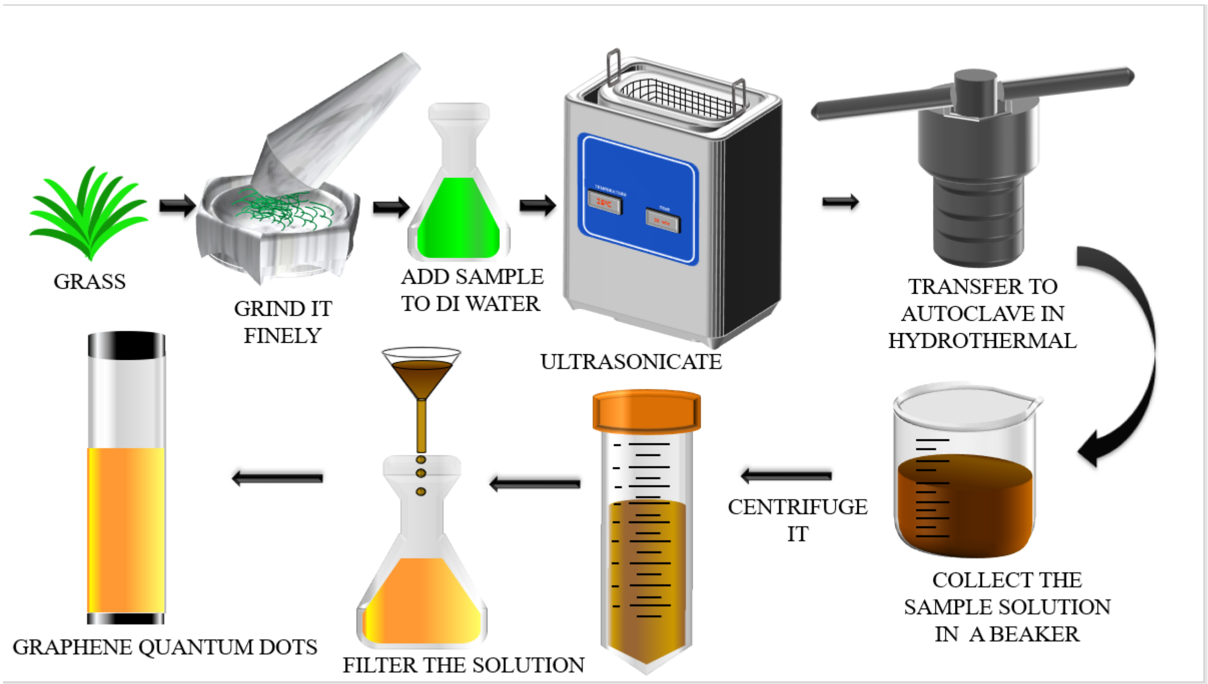

Fig. 1: Schematic representation of synthesis process of GQDs

CHAPTER – 4

CHARACTERIZATION TECHNIQUE

Absorption spectra were analysed using a dual beam UV-vis spectrometer (Model: UV-1900I, SHIMADZU) for UV-vis spectroscopy studies. A quartz cuvette has optical path of 10 mm was used for the samples. PL and PL-excitation observations were recorded using the Fluorolog-3 spectrofluorometer (Horiba Jobin Yvon), which was set up with a PMT, 450W Xenon, and flash lamps. XRD patterns were acquired using a Bruker D8 advance that has a Cu (Kα) radiation source with an accelerating voltage of 40kV and a current of 20mA [27]. TALOS thermos-scientific tools were used to capture high-resolution images of the GQDs using a technique called TEM at an accelerating voltage of 200 kV. Using Model No.- Nicolet iS50 FTIR spectrometer, was used to gather the spectra throughout the frequency range of 500-4000 cm^{-1}. The EDX was performed using Model-Gemini SEM 500.

CHAPTER - 5

RESULTS AND DISCUSSION

5.1 XRD ANALYSIS

XRD spectrum gave a very pronounced diffraction peak at 25.42° which arises from the well-known (002) plane, indicating the formation of the crystalline GQDs as shown in Fig. 2 [28].

The peak at about 21.23°, was assigned as silica, based on a natural concentration in the grass precursor. The peak at 25.42° is related to the (002) plane of graphitic carbon, hence proving that graphene-like structures are formed. The slight deviation from the usual graphite peak position points out that defects and functional groups exist, which are inherent in the quantum dot structure coming from a biomass precursor with two significant peaks [27,29].

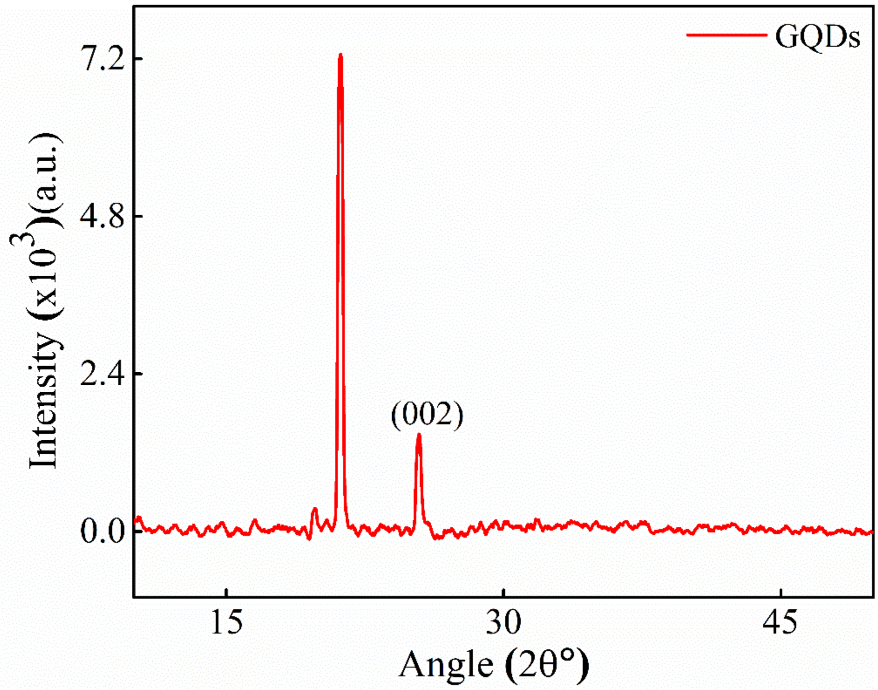

Fig. 2: XRD pattern of GQDs

5.2 EDX ANALYSIS

The similarity of the particles shows the estimated distribution of the chemicals in the GQDs in the EDX. The spectrum in Fig. 3 showed that GQDs had components of C (42%), N (22%) and O (21%).

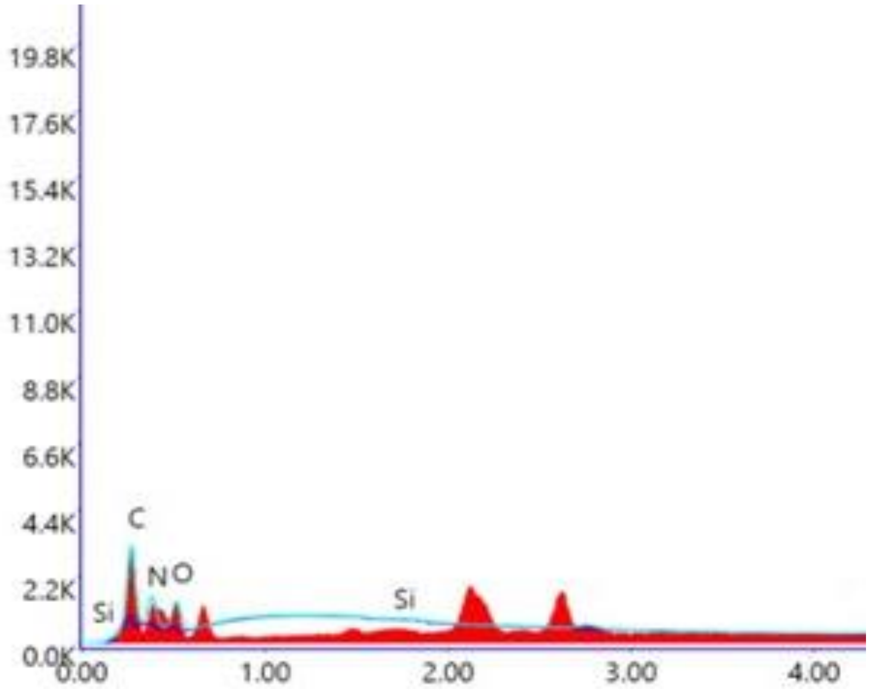

Fig. 3: EDX Spectrum of the GQDs

5.3 HRTEM ANALYSIS

HRTEM has been utilized in several studies to examine size and morphology of GQDs. It provides morphological information and is frequently used to determine size distribution of GQDs [28]. The synthesized GQDs is shown in Fig.4. The sample was prepared by dropping the sample solution onto a copper grid and letting it dry at room temperature. Fig. 5 depicts the size of the GQDs, which is 4.29 ±0.0.45 nm.

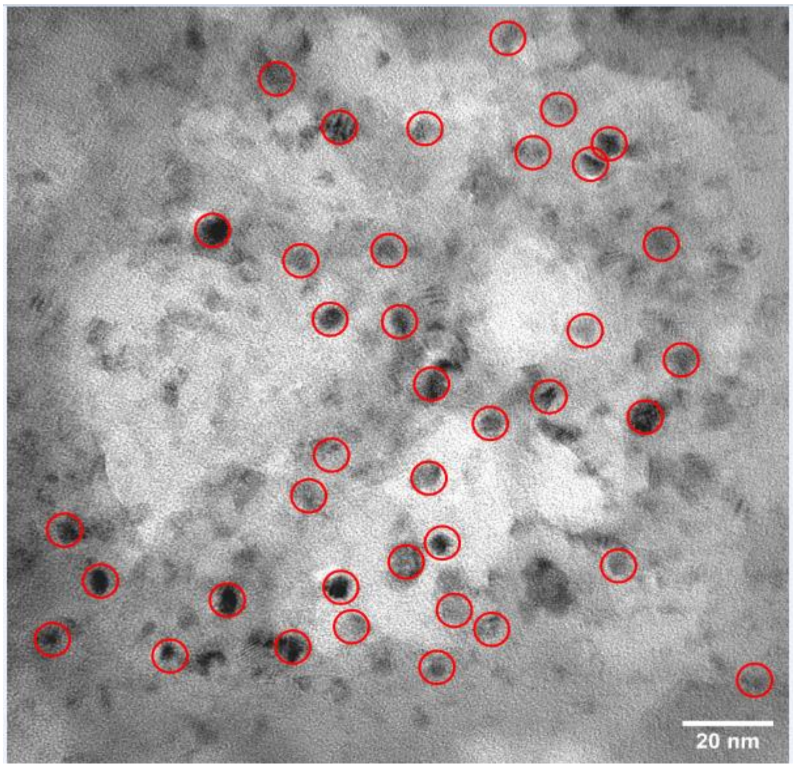

Fig. 4. HRTEM image of the GQDs

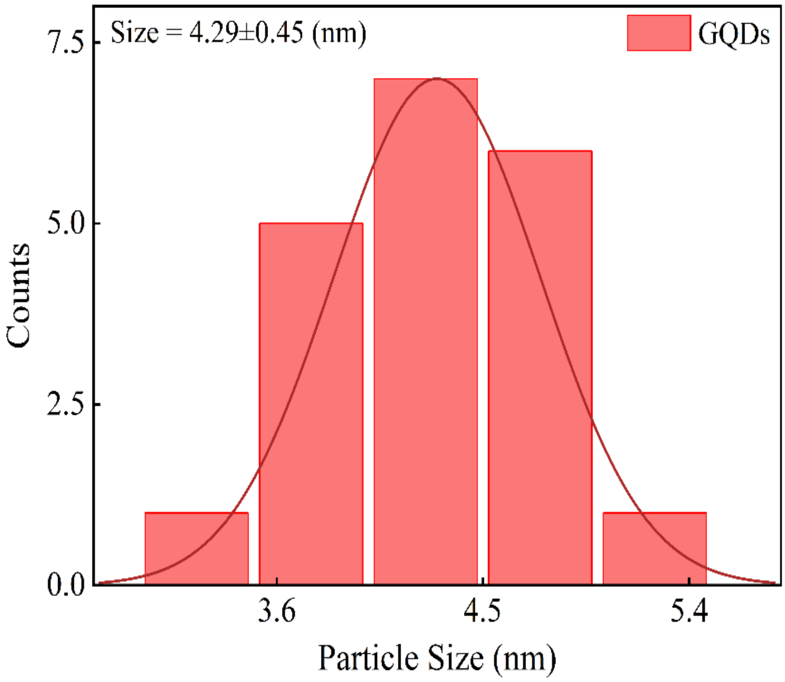

Fig. 5: Size distribution histogram of GQDs

5.4 ABSORBANCE ANALYSIS

Fig. 6 shows the absorbance spectrum of GQDs with its second derivative, which is derived from grass waste, exhibiting an absorbance peak at 272 nm, corresponds to π-π^* transition of sp^2 carbon domains, indication aromaticity in the GQDs structure. Additionally, a peak at 320 nm depicts the n-π^* transition of carbonyl groups in GQDs. This transition results from the interaction of non-bonding electron pairs in oxygen functional groups with π^* orbitals typically observed in GQDs [29,30]. The absorption peak aligns with previously documented GQDs, thereby validating the graphene-based quantum structure produced via the hydrothermal method [16]. Prior research has shown that absorption features within the 250 nm - 280 nm range are indicative of electronic transitions in the carbon backbone of GQDs [31,32]. The peaks suggest a hybrid structure comprising intact graphene domains and an abundance of oxygen-rich functional groups [29,33]. For better resolution enhancement and accurate identification of the precise locations of these overlapping absorption features, the second derivative absorption spectrum was plotted.

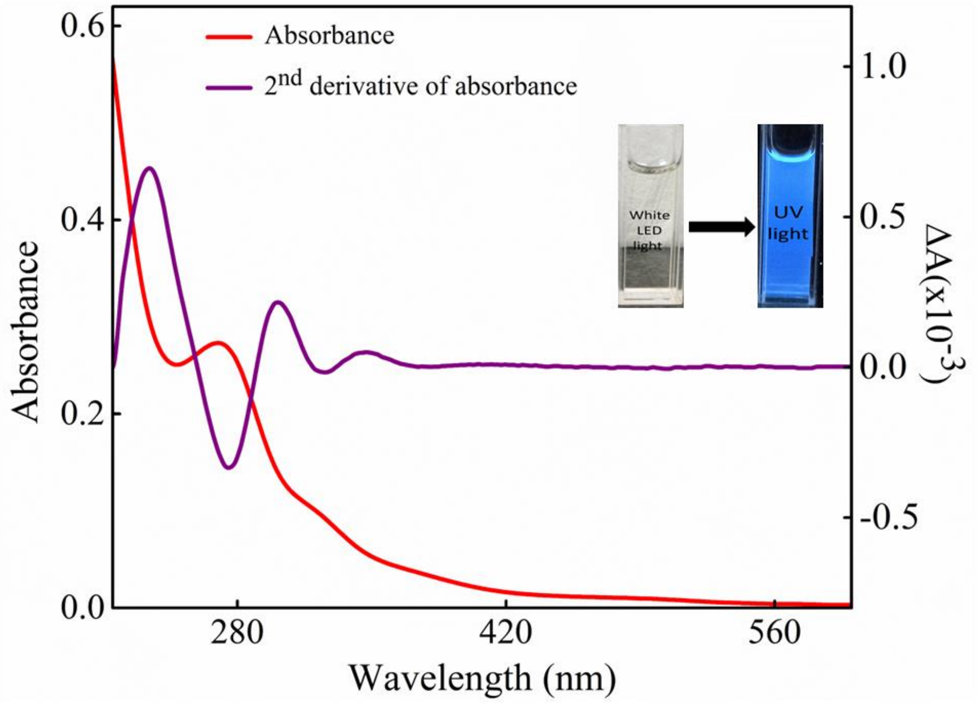

Fig. 6: Absorption Spectrum of the synthesized GQDs with its 2nd order derivative

The Tauc plot calculates the optical bandgap of the GQDs based on their absorption spectra. The plot of $(\alpha h\nu)^{1/\gamma}$ and photon energy ($h\nu$) is shown below, with the value of γ varying according to the kind of electronic transition. In particular, materials having a direct bandgap get the value of $\gamma = 1/2$, while materials with an indirect bandgap have the value $\gamma = 2$. Thus, as shown in Fig. 7, a direct bandgap of GQDs was calculated 3.8 eV [34,35].

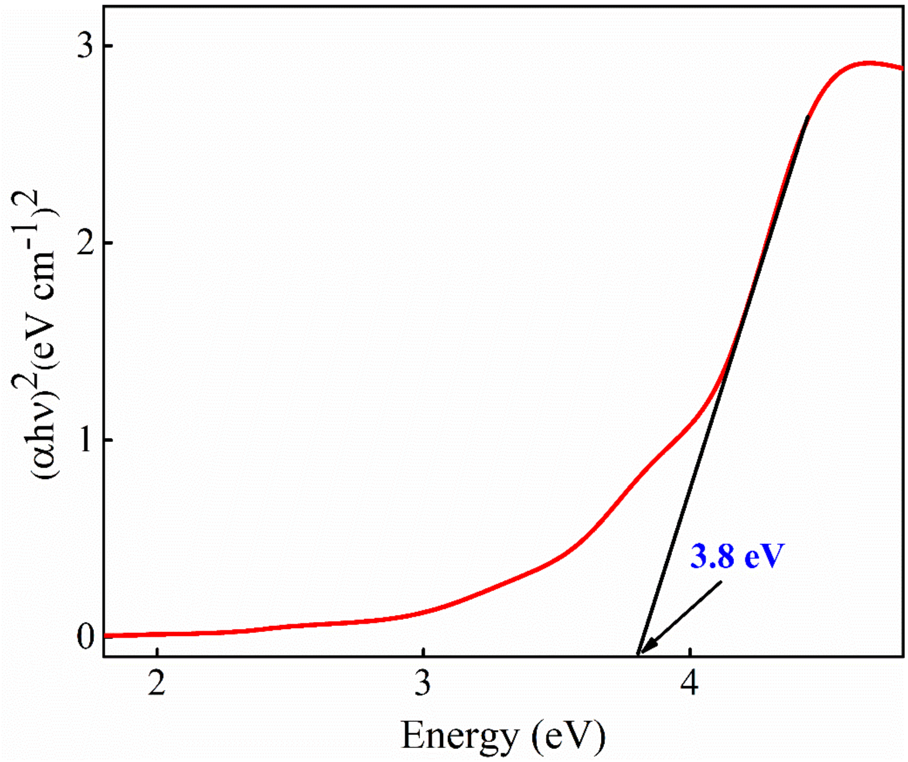

Fig. 7: Tauc Plot of the GQDs

5.5 PL ANALYSIS

Fig. 8 illustrates PL spectra of GQDs, exhibits a pronounced blue emission at 440nm upon excitation at 350 nm. Excitons radiatively recombine in small isolated sp^2 carbon domains within the GQDs structure, producing the emission wavelength [31,36]. The 440 nm measurement aligns with prior research on GQDs, indicating the development of quantum- confined graphene structures characterized by a notable size distribution [36,37].

The excitation wavelength of 350 nm falls within the range for GQDs (300 - 400 nm), resulting in blue emission, which indicates that grass derived GQDs have electronic states that effectively absorb in the region. The Stokes shift observed between the excitation wavelength (350 nm) and the emission wavelength (440 nm) suggests that energy relaxation processes take place within the electronic structure of GQDs prior to photon emission [36].

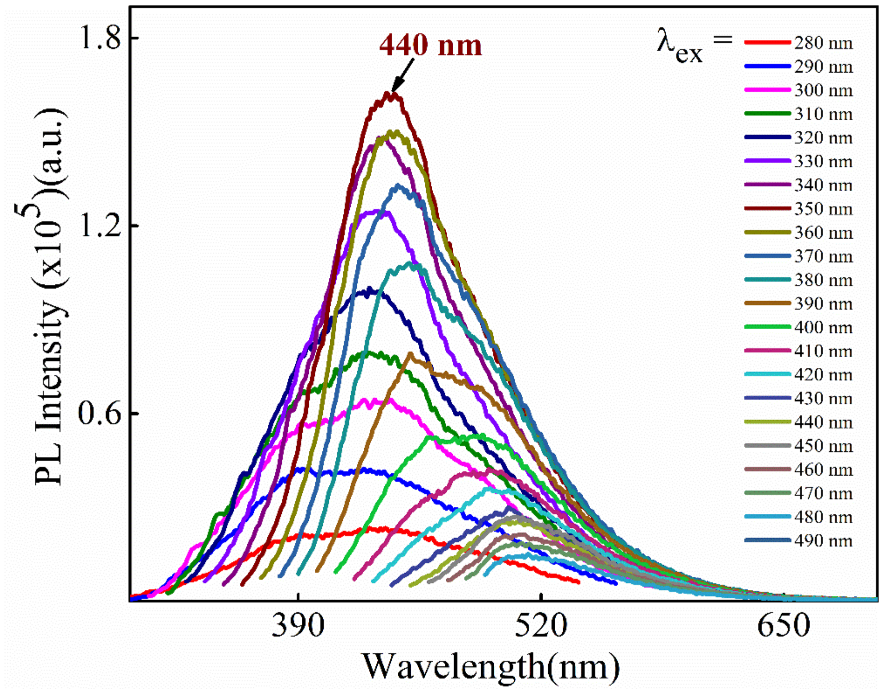

Fig. 8: PL Spectra of the GQDs

Fig. 9 illustrates the normalization of intensity associated with the redshift. Using the emission wavelength as excitation source we plotted excitation wavelength vs intensity plot.

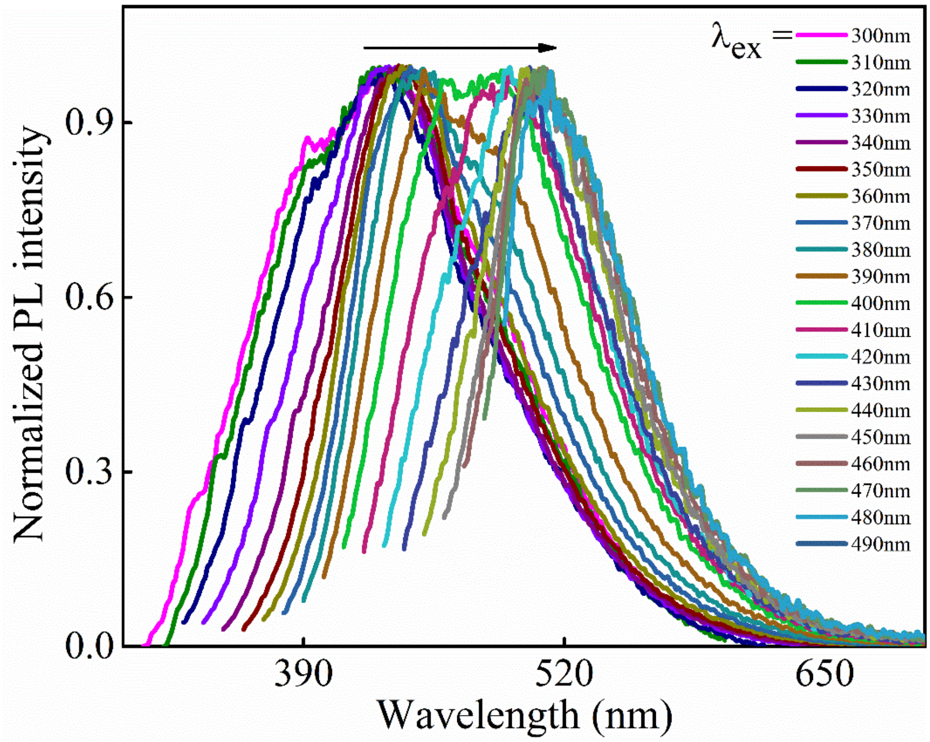

Fig. 9: Normalised PL Spectra of the GQDs

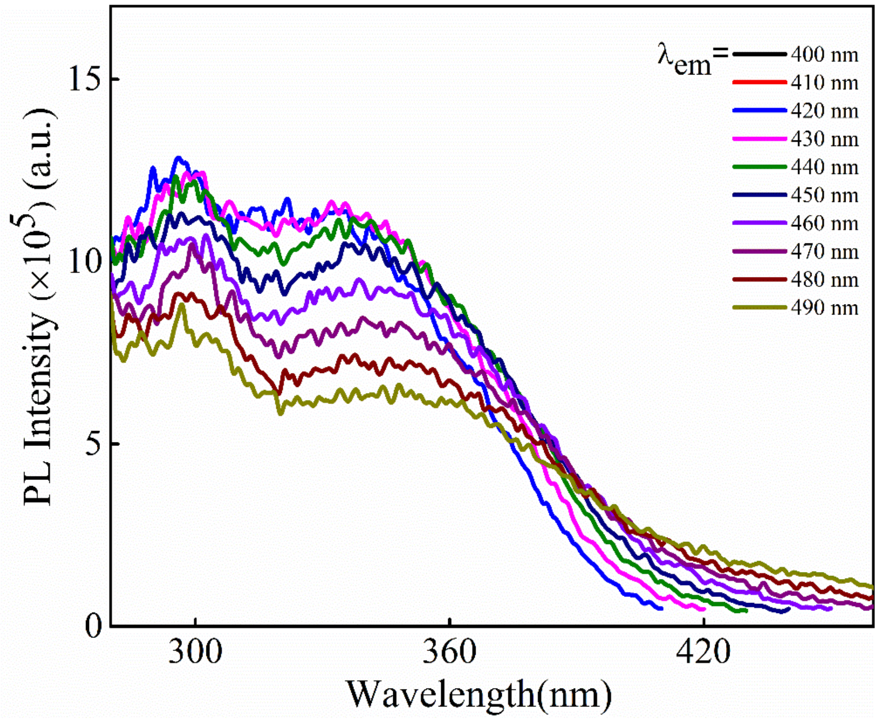

Fig. 10: Excitation Spectra of the GQDs

We use 420 nm emission wavelength as an excitation source, the normalised absorbance and normalised excitation plot are approximately overlapping each other as shown in fig. 11.

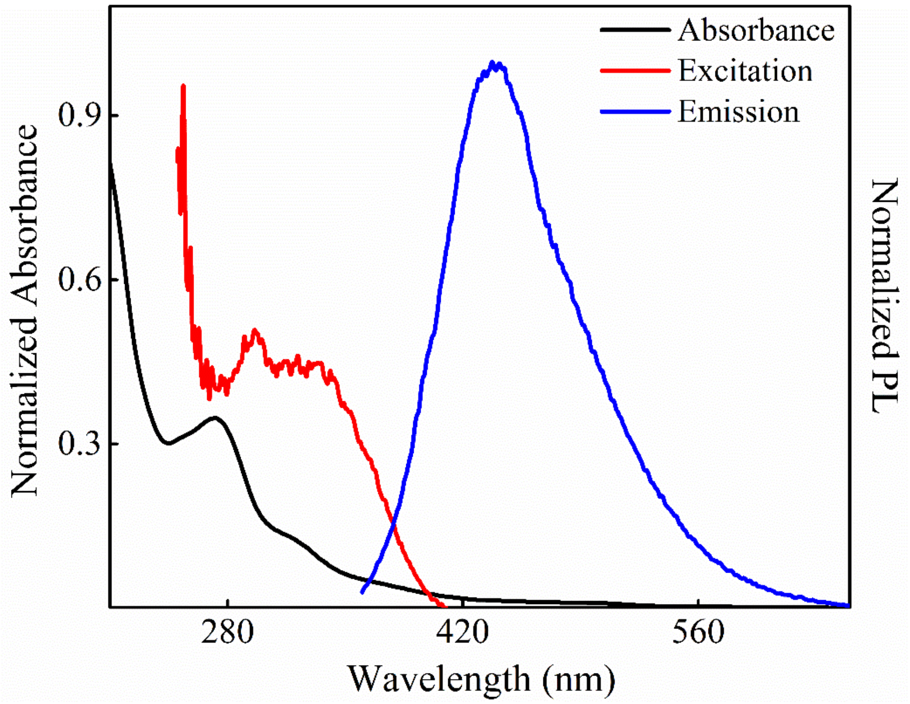

Fig. 11: Overlapped absorbance, excitation (at emission wavelength 420 nm) and PL Spectra (at excitation wavelength 350 nm) of the GQDs

The PL intensity of GQDs are nearly constant within 9 months of period which shows the stability.

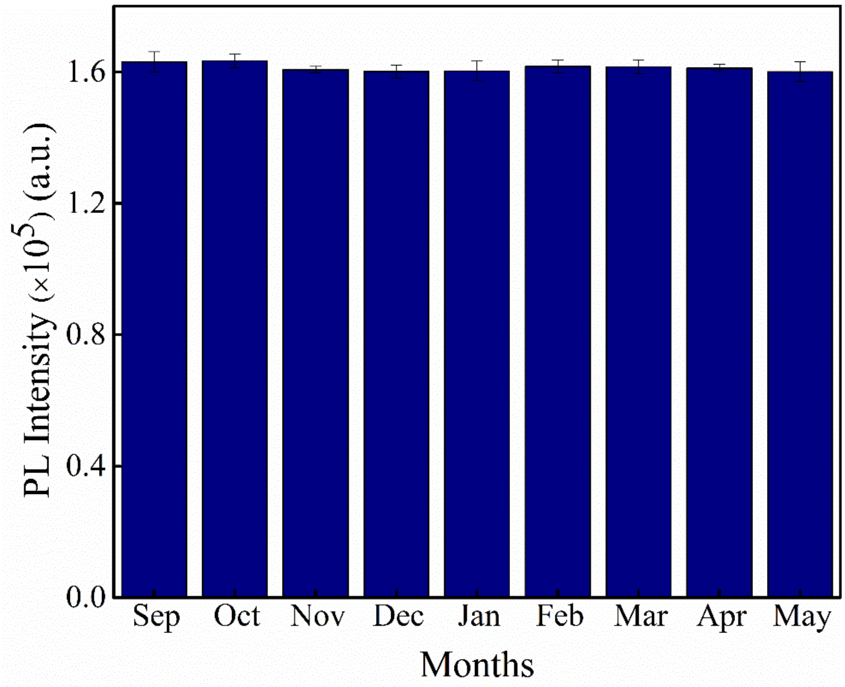

Fig. 12: PL spectra of GQDs at 350 nm monitored in the repeated interval up to 9 months, representing the stability of GQDs

5.6 FTIR ANALYSIS

Fig. 13 illustrates the FTIR spectrum, highlighting the functional groups that influence the physicochemical properties. The spectral peak observed at 3322 $\mathrm{cm^{-1}}$ corresponds to O-H stretching modes, indicating that hydroxyl functional groups are present on the GQD surface [38,39]. The presence of hydroxyl groups enhances water dispersibility and offers reactive sites for functionalisation. A notable peak at 2109 $\mathrm{cm^{-1}}$ is C≡N stretching vibrations, denotes nitrogen functionalities derived from the grass precursor during synthesis [40,41]. Nitrogen presence enhances the distinct optical properties of GQDs. The peak at 1635 $\mathrm{cm^{-1}}$ corresponds to C=C stretching vibrations within the graphitic domains of the GQDs, confirming the maintenance of the sp^2 carbon network despite the hydrothermal method. The spectral peak at 1014 $\mathrm{cm^{-1}}$ signifies C-O stretching vibrations, suggesting the presence of epoxy or other oxygen containing functional groups [42,43].

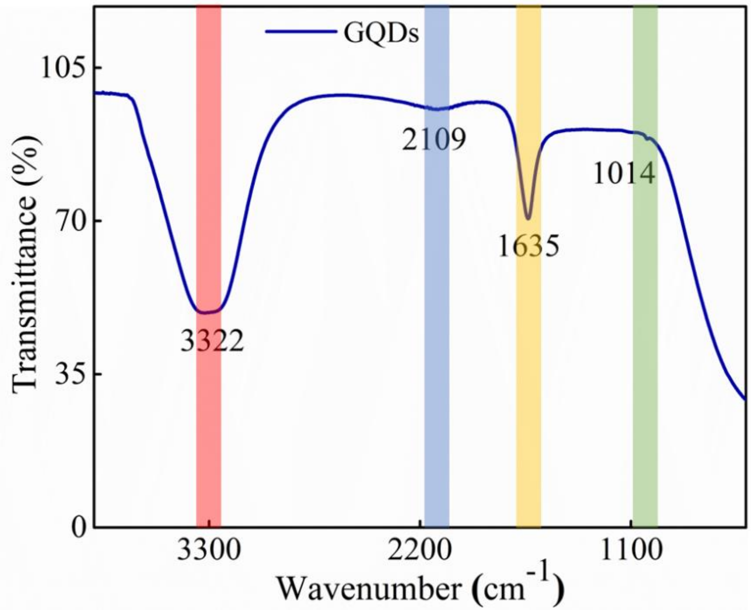

Fig. 13: FTIR Spectrum of the GQDs

CHAPTER -6

CONCLUSIONS

This work used a simple hydrothermal method to synthesize (GQDs) from grass waste. The GQDs structural, optical and morphological characteristics were investigated using various characterization methods. While EDX showed a high carbon content couple with nitrogen and oxygen, suggesting the existence of functional groups, XRD examination verified the presence of graphitic carbon and silica. The GQDs were virtually spherical, evenly distributed, and had an average size of around 4.04 nm, according to TEM images. Characteristic absorption peaks associated with π-π^* and n-π^* transitions were revealed by UV-VIS spectroscopy, indicating the existence of oxygen-containing groups and sp^2 carbon domains. The Tauc plot's predicted direct bandgap of 3.76 eV confirms that the synthesized dots are quantum-confined. Strong blue emission was seen at 440 nm by PL analysis, indicating distinct electronic transitions caused by the sp^2 clusters. The existence of hydroxyl, cyano and other oxygen-containing functional groups on GQDs surface was further verified by FTIR spectra. These groups help explain the particles' high water dispersibility and surface modification potential. Overall, the results show that GQDs synthesized from grass have intriguing optical and structural characteristics, making them appropriated for various uses in bioimaging, optoelectronics, and sensing.

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APPENDIX - I

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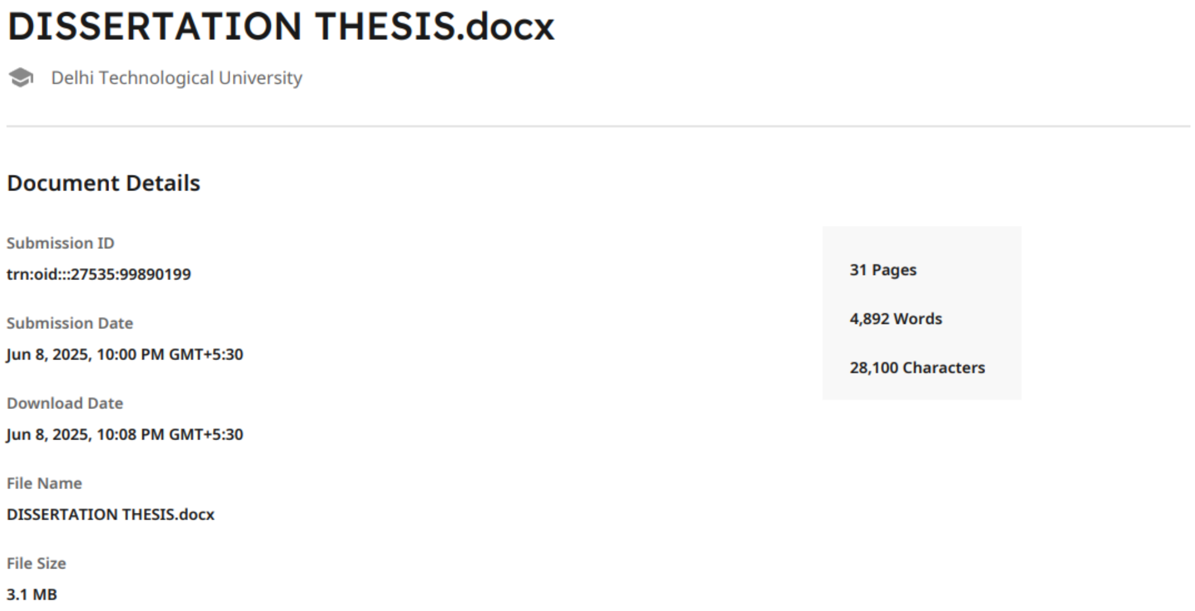

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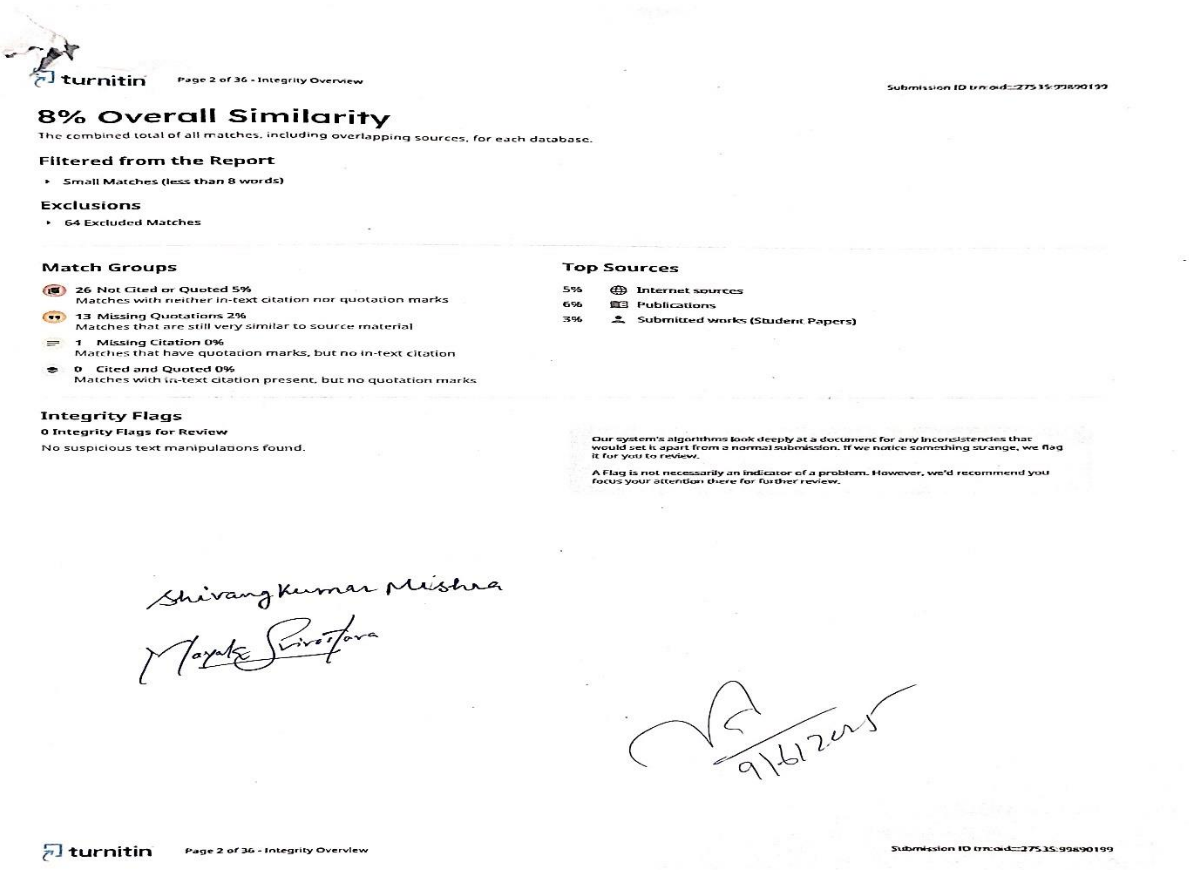

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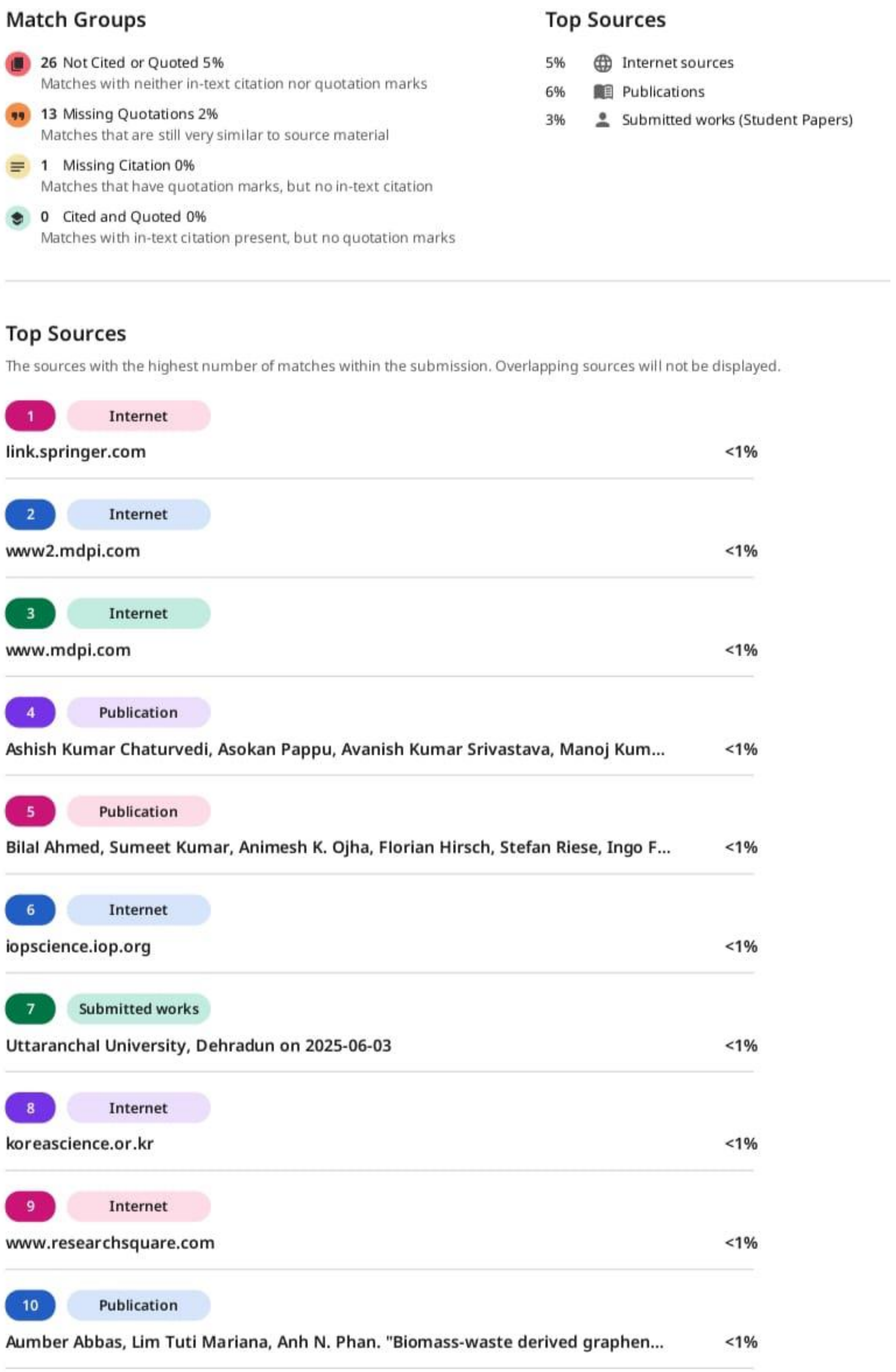

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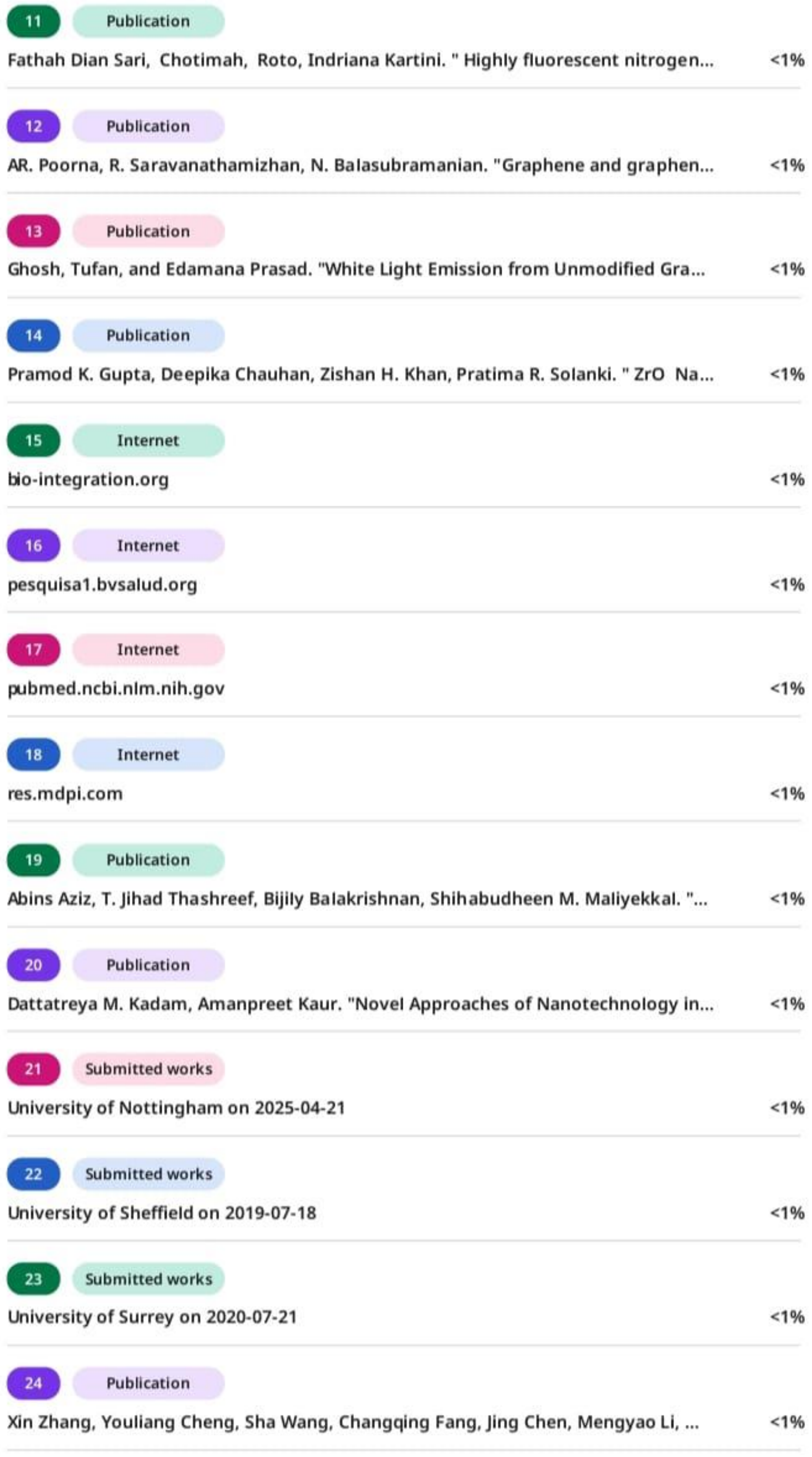

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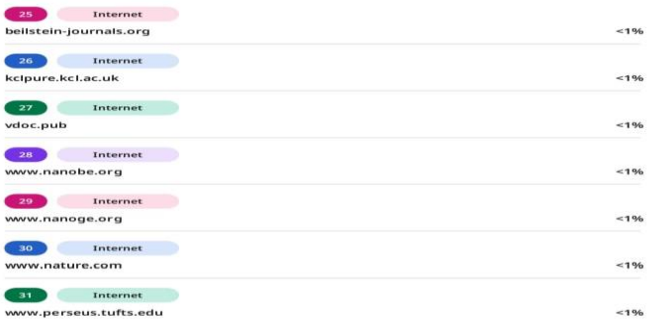

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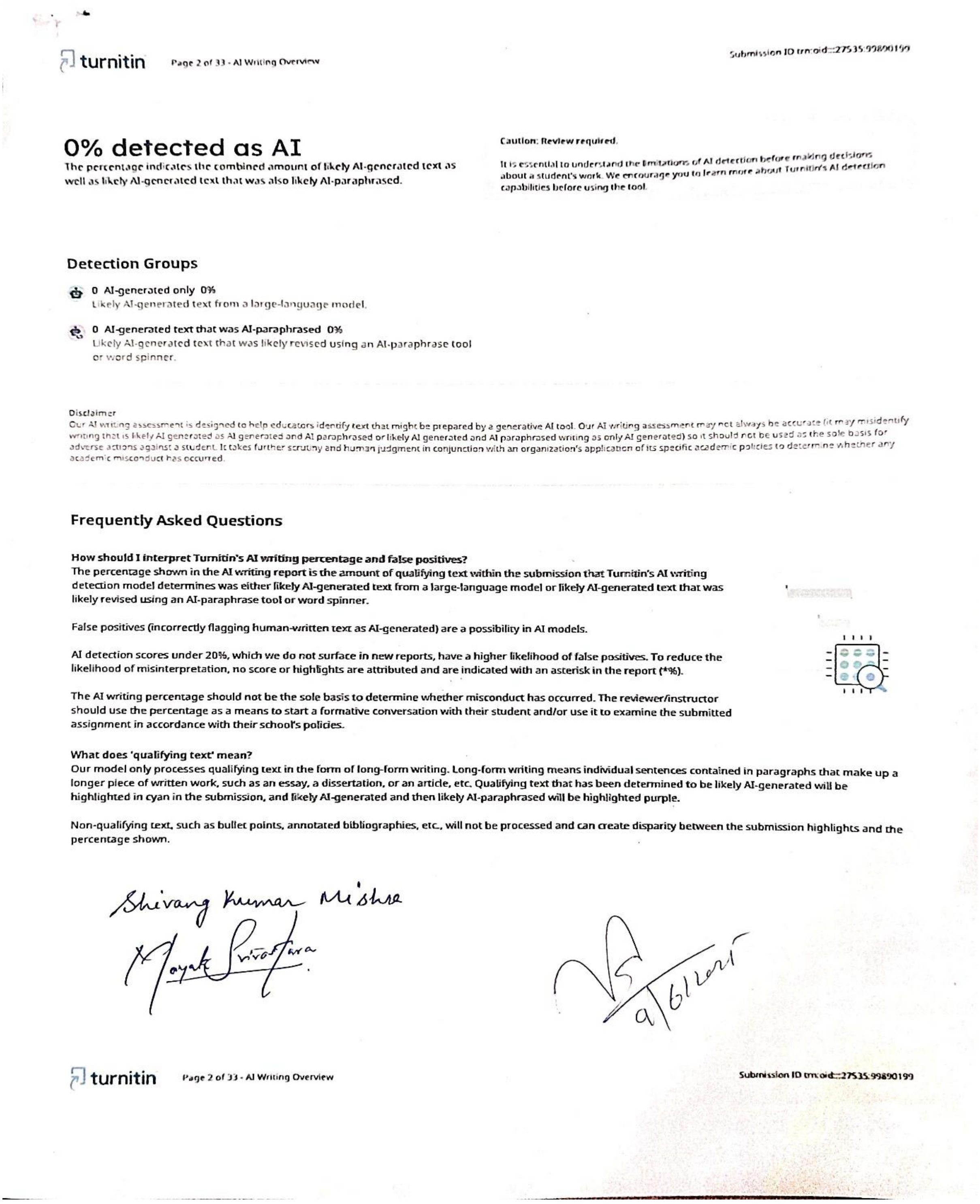

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Shivang Kumar Mishra
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APPENDIX - II

CONFERENCE RECORD

PROOF OF REGISTRATION

3rd International Conference on Advanced Functional Materials and Devices for sustainable development (AFMD-2025)

Department of Physics and Internal Quality Assurance Cell (IQAC) of Atma Ram Sanatan Dharma College,University of Delhi

Springer

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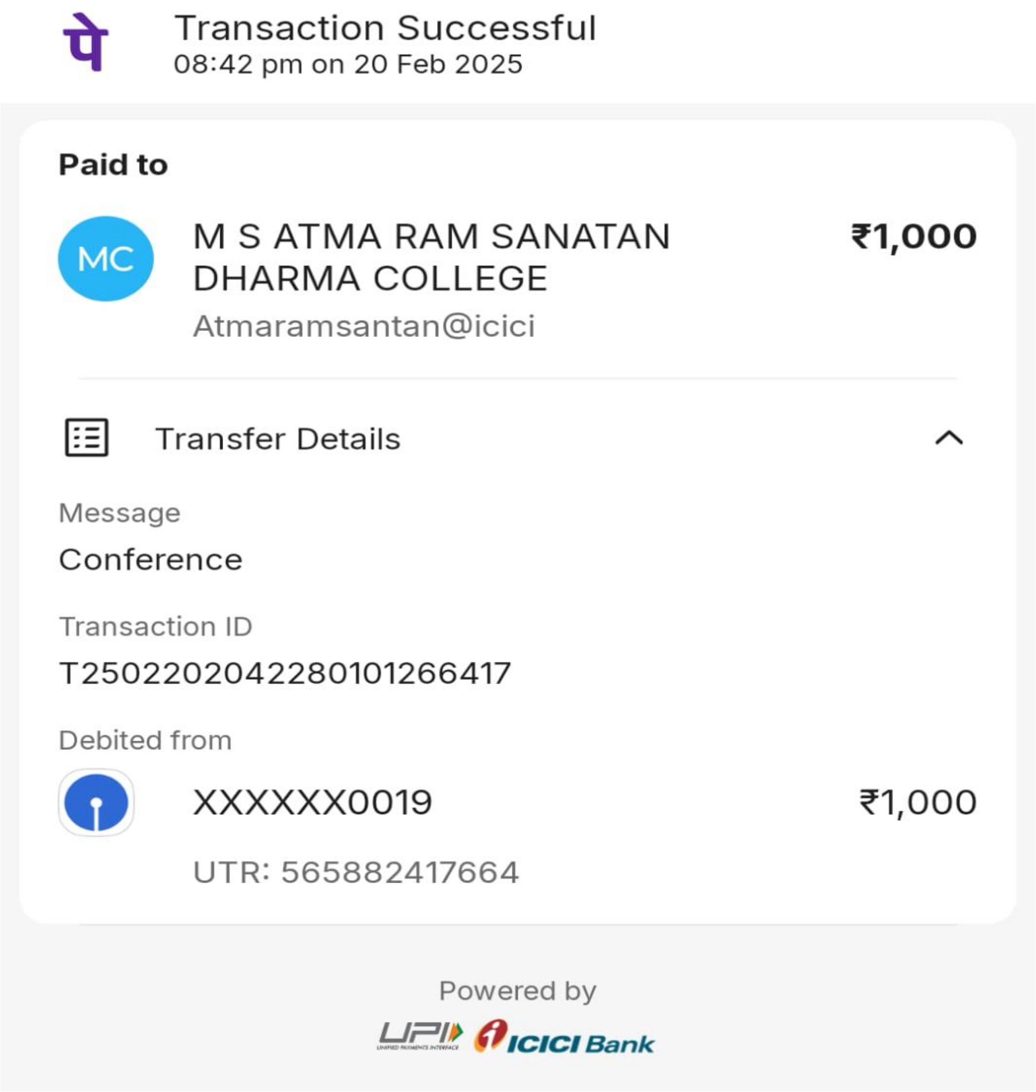

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Acceptance Letter for 3rd International Conference on Advanced Functional Materials & Devices for Sustainable Development (AFMD-2025) at ARSD College

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Sun, Feb 16, 2:38 AM

Acceptance Letter – AFMD-2025

Dear Prof./Dr./Mr./Ms. **SHIVANG KUMAR MISHRA**
DELHI TECHNOLOGICAL UNIVERSITY

Greetings from the organizing committee of the International Conference on "Advanced Functional Materials & Devices for Sustainable Development (AFMD-2025)." The conference will be held in hybrid mode* (online & offline) at Atma Ram Sanatan Dharma College, University of Delhi, from March 03-05, 2025. It is being organized by the Department of Physics and Internal Quality Assurance cell (IQAC) of ARSD College.

We are pleased to inform you that the technical committee has accepted your participation, **titled**:

PP-41:-Production of Graphene Quantum Dots Using Bermuda Grass
Type of Presentation: **Poster Presentation:-Offline Mode (ARSD College, Dhalakuan, New Delhi India)**

*Program will take place as per following manner:

- ***Day-1 Offline Mode at ARSD College***
- ***Day 2&3 Online Mode through Zoom of Google Meet***

Participation Letter for 3rd International Conference on Advanced Functional Materials & Devices for Sustainable Development (AFMD-2025) at ARSD College

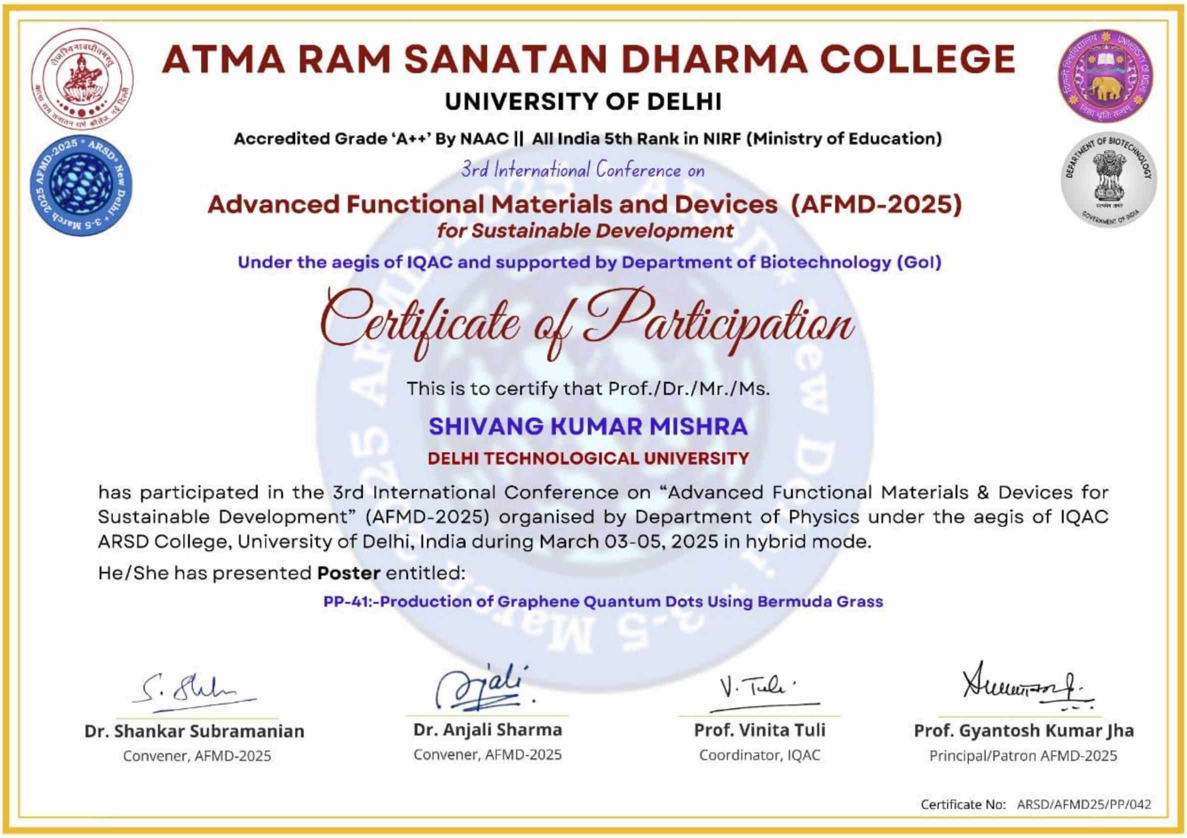
afmd2025... 19 Feb

Participation Letter – AFMD-2025

Dear Prof./Dr./Mr./Ms. **MAYANK SRIVASTAVA**
DELHI TECHNOLOGICAL UNIVERSITY

Greetings from the organizing committee of the International Conference on "Advanced Functional Materials & Devices for Sustainable Development (AFMD-2025)." The conference will be held in hybrid mode* (online & offline) at Atma Ram Sanatan Dharma College, University of Delhi, from March 03-05, 2025. It is being organized

PARTICIPATION CERTIFICATES



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3rd International Conference on

Advanced Functional Materials and Devices (AFMD-2025)
for Sustainable Development

Under the aegis of IQAC and supported by Department of Biotechnology (GoI)

Certificate of Participation

This is to certify that Prof./Dr./Mr./Ms.

SHIVANG KUMAR MISHRA

DELHI TECHNOLOGICAL UNIVERSITY

has participated in the 3rd International Conference on "Advanced Functional Materials & Devices for Sustainable Development" (AFMD-2025) organised by Department of Physics under the aegis of IQAC ARSD College, University of Delhi, India during March 03-05, 2025 in hybrid mode.

He/She has presented **Poster** entitled:

PP-41:-Production of Graphene Quantum Dots Using Bermuda Grass

Dr. Shankar Subramanian Convener, AFMD-2025	**Dr. Anjali Sharma** Convener, AFMD-2025	**Prof. Vinita Tuli** Coordinator, IQAC	**Prof. Gyantosh Kumar Jha** Principal/Patron AFMD-2025

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3rd International Conference on Advanced Functional Materials and Devices for sustainable development (AFMD-2025)

Department of Physics and Internal Quality Assurance Cell (IQAC) of Atma Ram Sanatan Dharma College,University of Delhi

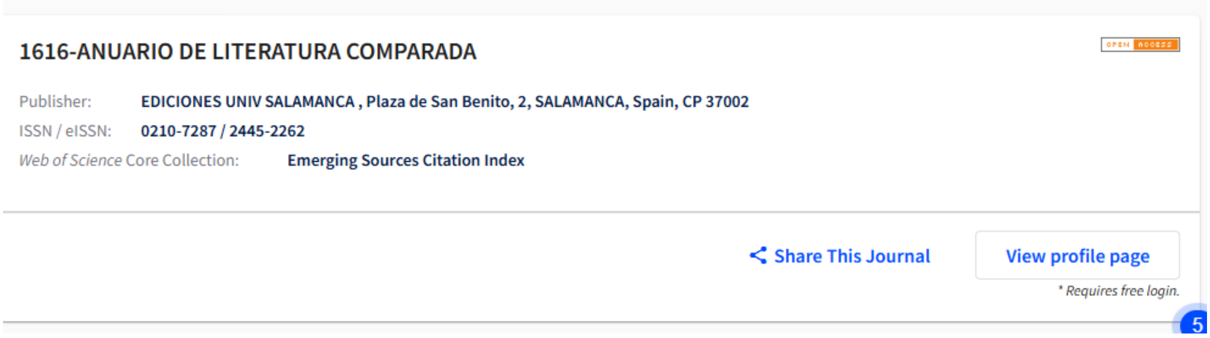
Publications

Abstract book will be published.

All selected papers for AFMD 2025 will undergo the stringent review process by the International Review Committee of the AFMD 2025. The peer reviewed and selected papers of AFMD 2025 will be published by Springer with three different publishing houses.

1. Journal of Electronic Materials (Spinger Nature Link) : Scopus Indexed **(JEM Impact Factor: 2.2)**

Note: Manuscripts will be evaluated according to the same high standards as would be applied to any article published in the journal. Authors are encouraged to read carefully and comply with the Instructions for Authors on the JEM website. All submitted papers will go through a plagiarism check to evaluate overlap with previously published work.



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