Received: 22 August 2025 | Revised: 02 November 2025 | Accepted: 17 November 2025

https://doi.org/10.56946/jce.v4i2.823

Journal of Chemistry and Environment

Research Article

The Structural, Optical and Thermal Characterization of g-C_3N_4 for Photocatalysis

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Abstract

Graphitic carbon nitride (g-C_3N_4)-based nanomaterials gained a lot of interest due to their special qualities, which include their chemical stability and ease of modification. Still, g-C_3N_4's ability to work as a photocatalyst is limited. The combination of adsorption and photocatalysis has become popular in recent years because adsorption is necessary for photocatalytic surface reactions. g-C_3N_4 was made by heating melamine and then cooling it down. Its structural, optical, and thermal properties were then studied. The XRD diffraction spectra show that g-C_3N_4 has a layered structure. The calculated *d*-spacing is 0.324 nm and the estimated crystallite size is 90.9 nm. The *UV–Vis* studies showed that the material strongly absorbs visible light and has a band gap of about 2.61 eV. TG and DSC results showed that the material was very stable at high temperatures and found a thermal transition point about 350 °C. These results indicate that g-C_3N_4 is a promising material for photocatalytic and environmental applications.

Keywords: Reactive red, methylene blue, biosynthesis, copper oxide nanoparticles, dye degradation, nanotechnology

1. Introduction

Pollution has gotten worse as industrial activity increases globally due to the release of toxic gasses and wastes. Even at varying concentrations, these pollutants are harmful to air and water systems and pose a serious risk to human health. Heavy metals and harmful microorganisms, which are commonly found in contaminated water, as well as persistent organic pollutants like dyes, antibiotics, and medications, are major concerns. Sometimes these stubborn pollutants are difficult to remove by conventional wastewater purification methods, and as a result, they wind up in aquatic environments where they can cause significant environmental harm [1]. This issue shows how important it is to find cheap and effective ways to clean wastewater so that harmful toxins don't get into the ecosystem [2]. Non-metallic semiconductors, especially graphitic carbon nitride (g-C_3N_4), are among the new solutions that have shown a lot of promise for photocatalytic applications, just like titanium dioxide (TiO_2), which is utilized a lot [3]. Graphitic carbon nitride has gotten a lot of attention since it is plentiful, chemically stable, easy to make, and has cheap precursors [4]. With a bandgap of about 2.7 eV, it absorbs more visible light than TiO_2, making it perfect for photocatalytic water treatment. Also, g-C_3N_4 can break down contaminants well, which makes it an important part of the photodegradation of organic compounds in wastewater [3, 5]. Additionally, it looks at applications in pollutant degradation, CO_2 reduction, and hydrogen evolution, with a focus on designing g-C_3N_4 composites to improve charge separation [6]. This polymeric, metal-free, n-type semiconductor has a π-conjugated structure, a low bandgap, and a high ability to adsorb on surfaces. This makes it a good choice for attaching both heavy metal ions and organic molecules [7]. It also works well with visible light and is quite stable chemically. However, its use in water treatment is limited by a number of factors, including as low electrical conductivity, a small surface area, quick recombination of photo-generated charge carriers, and limited absorption of visible light [8]. Different ways to make g-C_3N_4 have been looked into. For example, R. Manimozhi et al. made g-C_3N_4 by heating 5 grams of melamine in an alumina crucible in a nitrogen-filled furnace at 520 °C for four hours, with a ramp rate of 20 °C per minute. After that, the material was milled into a fine powder [9]. Lan et al. made g-C_3N_4 in the same way by heating urea to 520 °C for two hours in a muffle furnace, then letting it cool slowly and grinding it into a pale-yellow powder for later use [6]. The photodegradation results demonstrate that Alg/ZnO-g-C_3N_4 eliminates methylene blue (MB) with 73.46% degradation efficiency under UV-visible light and 78.18% under sunshine in 60 minutes [10]. The g-C_3N_4/ZnO (20/80) material, with a surface area of 25 m^2/g, displayed improved photocatalytic efficacy, degrading MB by 85% in 100 minutes under visible light and approximately double the effectiveness of commercial P25-TiO_2 (31%) [11]. g-C_3N_4 is typically added to heterostructure composites to make them better at breaking down dyes and other organic materials in dirty water. This makes them better at photocatalysis.

2. Preparation of g-C_3N_4

In the thermal condensation method to make graphitic carbon nitride (g-C_3N_4), tiny organic compounds that are high in nitrogen, like urea, melamine, thiourea, cyanimide, and dicyanamide, are usually utilized as precursors. The precise reaction conditions used during synthesis have a big effect on the structural units that are generated.

One simple way to make something used melamine as the starting material. About 9.5 grams of melamine were put in a covered ceramic crucible and heated in a muffle furnace for roughly two hours at room temperature. The crucible was left to cool down to room temperature after being heated. Then, the yellow substance was collected and pounded into a rough powder with a mortar and pestle.

3. Characterization of g-C_3N_4

A variety of analytical methods were used to investigate the structural, optical, and thermal stability of g-C_3N_4. For the examination of the structural properties of the prepared sample, X-ray diffraction techniques are performed using Cu$K\alpha$ (λ=0.15406 nm). UV-visible spectroscopy is used to record absorbance spectra in the 200 to 800 nm wavelength range. A simultaneous thermogravimetric and differential scanning calorimetric (TG-DSC) system (STA 7300) is used for determining thermal characteristics. The system worked in a nitrogen atmosphere with a flow rate of 60 ml/s.

4. Results and discussion

4.1 Structural study of g-C_3N_4

Figure 1 shows the diffraction pattern of the g-C_3N_4 sample that was made. It was recorded over a 2θ range of 10 ° to 80 °. The result shows two primary peaks, which are usually used to show that this material has been successfully produced. The most noticeable peak is at 27.47 °, which is the (002) plane. This shows how the stacked sheets in the material stack on top of each other. This feature looks like the way graphite stacks, which means that g-C_3N_4 has an ordered, layered structure [11]. There is a smaller and wider peak near 13.1 °, which is due to the (100) plane. This peak gives information about the in-plane structure of the carbon nitride framework, specifically how the heptazine (tri-*s*-triazine) units that make up the polymer network are arranged [12]. These two peaks together show that the material that was made has a normal graphitic structure with both interlayers stacking and in-plane ordering. The noticeable peak's sharpness and intensity also show a pretty strong level of structural order (i.e. the layers are stacked quite regularly, which means that precursors like melamine were successfully polymerized by heat).

The crystallite size of the g-C_3N_4 crystals can de estimated, using the Scherrer formula, $D = (0.9\lambda)/(\beta\cos\theta)$. In this formula, λ is the wavelength of the X-ray (0.15406 nm), β is the FWHM (in radian), and θ is the Bragg angle (in radian). The estimated crystallite size of g-C_3N_4 is around 90.9 nm, with the matching conspicuous (002) peak in the diffraction pattern and an interlayer spacing (*d*-spacing) of about 0.324 nm, characterized by π–π stacking interactions among the g-C_3N_4 sheets [15]. This can find with JCPDS 87-1526.

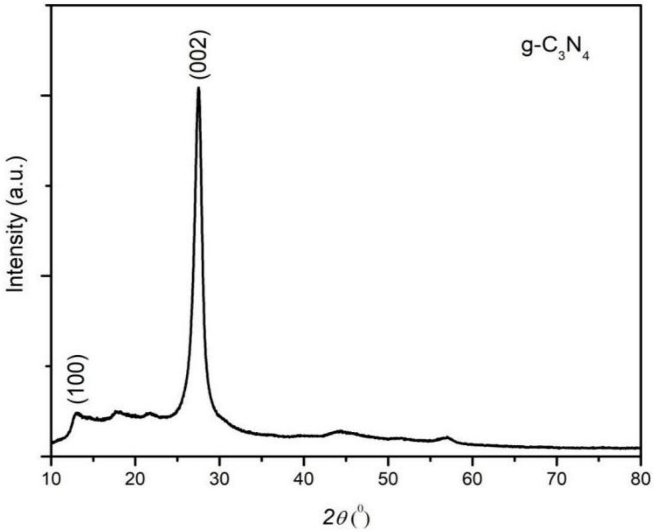

Figure 1. Diffraction pattern of g-C_3N_4.

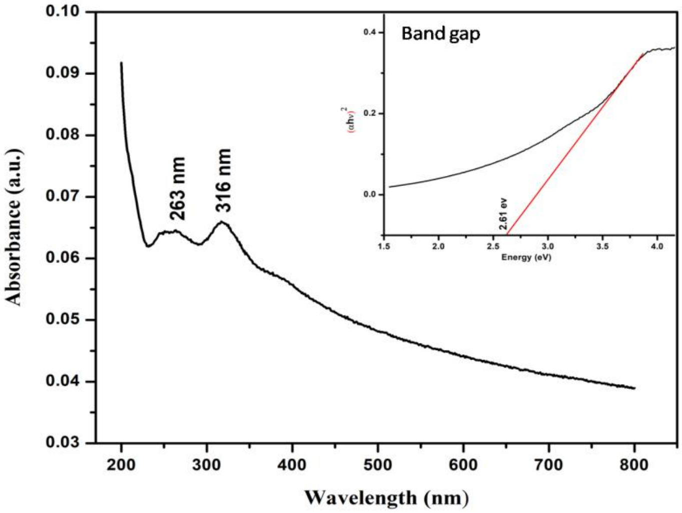

Figure 2. Absorbance and (inset figure) Band gap using Tauc plot of g-C_3N_4.

4.2 Optical study: Absorbance and bandgap of g-C_3N_4 :

Figure 2 shows the absorption spectra of graphitic carbon nitride (g-C_3N_4). The absorbance is shown against the wavelength from 200 to 800 nm. The spectrum shows two clear absorption peaks at about 263 nm and 316 nm. These peaks are due to $\pi \rightarrow \pi^*$ electronic transitions, which are typical of aromatic C–N bonding structures seen in the material that was made. This transition also includes the sp^2-hybridized carbon and nitrogen atoms in the aromatic tri-s-triazine units of g-C_3N_4. The conjugated system of C=N bonds in the heptazine rings makes this high-energy transition possible by moving the π electrons from the HOMO to the LUMO. Here the Tauc plot method is used (which is displayed in the inset of Figure 2) to find the optical band gap of g-C_3N_4. The calculated band gap is about 2.61 eV, which is quite close to the theoretical value [16]. This shows that the material has the expected semiconducting capabilities.

4.3 Thermogravimetric & DSC Analysis:

Figure 3 shows the thermogravimetric analysis (TGA) curve of g-C_3N_4. It shows how the weight of the substance changes with temperature. When the temperature rises from room temperature to 500 °C, the weight slowly drops by around 2.7 wt%. This small weight loss shows that the material is less thermally stable, which means that it doesn't break down much at this temperature range.

The graphs on the other side of Figure 3 show the Differential Scanning Calorimetry (DSC) curve, which shows how heat flows with temperature. The heat flow rises steadily until it reaches a peak of about 350 °C. This is probably because of thermal events like phase transitions or just the start the process of breakdown in the material.

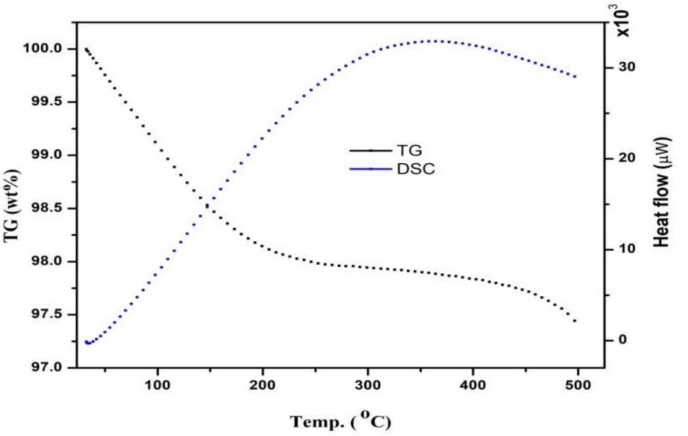

Figure 3. Thermogravimetric and DSC plot of g-C_3N_4.

4.4 FTIR measurements of g-C_3N_4:

Figure 4 shows the FTIR spectrum of g-C_3N_4. The results show that it has a variety of distinctive vibrational properties that are due to its unique chemical structure. The broad absorption band seen between 3000 and 3500 cm^{-1} is due to stretching vibrations of N-H bonds. This is usually linked to $-NH_2$ or –NH groups and moisture that has been absorbed, perhaps from the way the molecules were made.

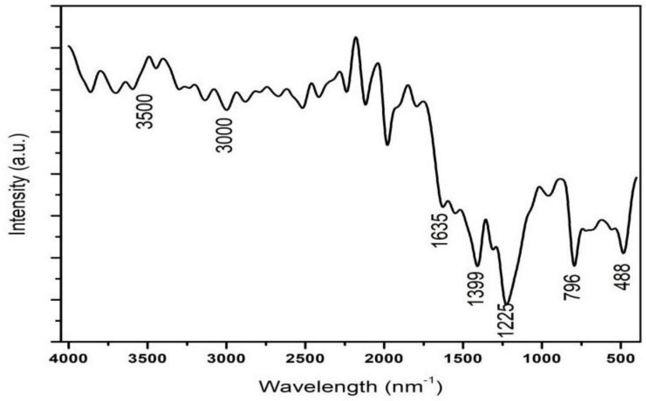

Figure 4. FTIR spectra of g-C_3N_4.

The presence of peaks in the range of 1205 to 1630 cm^{-1} corresponds to the stretching vibrations of aromatic C–N and C=N bonds inside the tri-s-triazine rings. The conjugated, nitrogen-rich heterocyclic framework, which is mostly made up of heptazine units, gives rise to these properties [17]. A sharp and distinct peak about 796 cm^{-1} is the most significant characteristic to take into account. This peak is the out-of-plane bending vibration of the triazine rings, which is a well-known feature of the g-C_3N_4 structure. Researchers usually refer to this peak the "breathing mode" of the heptazine units, which shows that the graphitic structure is there [18]. Also, lower absorptions below 600 cm^{-1} are usually linked to skeletal bending vibrations in the CN network. These spectral characteristics are in strong alignment with prior research and corroborate the structural integrity and chemical identity of g-C_3N_4 as evidenced in the FTIR spectrum.

4.5 Dye degradation activity under sun irradiance:

Figure 5(a) shows how methylene blue (MB) breaks down when exposed to natural sunshine and the manufactured photocatalyst. The distinctive absorption peak of MB is located at roughly 662 nm. The spectra taken at different times of irradiation reveal a definite drop in the absorption intensity over time, which shows that the dye is slowly breaking down. As the exposure duration gets longer, the absorption peak goes down significantly, which shows that MB molecules are breaking down into less harmful intermediates and final mineralized products. After 4 hours of exposure to sunshine, almost 72% of MB is broken down, and the solution transforms from dark blue to almost colorless, showing that photocatalytic activity is functioning.

Figure 5 (b) shows the percentage of degradation that corresponds to the amount of time the material is exposed to radiation. The curve shows that the dye concentration drops quickly and steadily, which shows that the degradation process works better in sunshine than in the dark or in control settings.

The current methodology demonstrates competitive or improved performance relative to previously reported photocatalysts. Under visible light, bulk g-C_3N_4 usually has a low to moderate ability to break down methylene blue (MB).

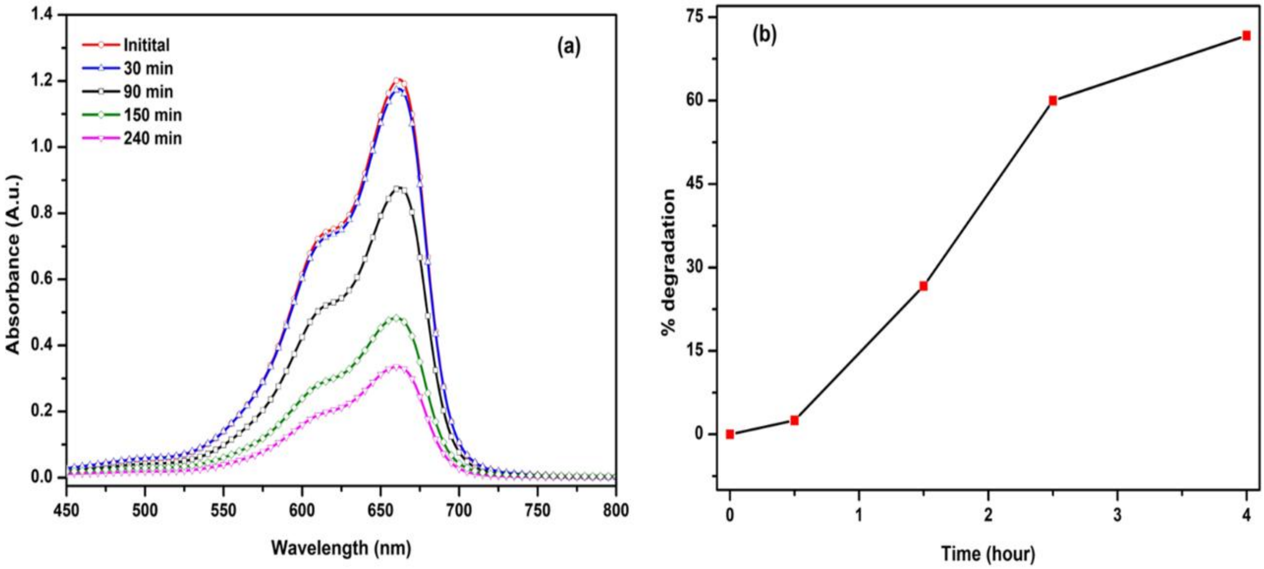

Figure 5. (a) Change in absorbance of MB dye with time (b) % degradation efficiency *w.r.t.* time.

After a few hours, experimental results usually show a breakdown rate of 40-55% or less, depending on how big the surface area is and how crystalline it is [19]. TiO_2-based catalysts exhibit approximately 60% degradation under analogous conditions. The increased activity in this work may be traced back to the nanostructured g-C_3N_4, which improved charge separation and made light absorption more efficient [20]. This study concluded that porous g-C_3N_4 is an exceptionally effective and promising substance for environmental remediation, especially for the extraction of organic contaminants such as methylene blue from wastewater by using visible light. But its performance is restricted by the fact that charge carriers can recombine and the redox potentials are not exceptionally high. The Z-scheme architecture [17], which is based on the two-step charge transfer that happens in natural photosynthesis, can improve charge separation, keep high oxidative and reductive potentials, and increase the amount of visible light that can be absorbed.

5. Conclusion

The successful synthesis of g-C_3N_4 using a straightforward thermal treatment of melamine underscores the material's simplicity in preparation and scalability. The XRD data show that the synthesized material is indeed graphitic carbon nitride. The optical absorption properties, such as a band gap of about 2.61 eV, show that the material is very responsive to visible light, which is important for photocatalytic uses. Thermal analysis showed that the material was stable even at high temperatures and didn't break down too much, making it even better for procedures that involve heat. These results show that g-C_3N_4 is a strong, cheap, and effective material with a lot of promise for usage in technologies linked to energy, wastewater treatment, and breaking down pollutants. Future efforts can concentrate on enhancing its photocatalytic efficacy via structural alterations and the creation of heterostructure composites. The results show that the material has a well-ordered, layered structure, which makes it good for uses like photocatalysis or energy conversion.

Authors Contribution

Conceptualization, R.S., A.S., P.K.J., and C.L.; methodology, R.S., M.Y., N.G., S.G.; software, M.Y., C.L., A.S.; formal analysis, R.S., M.Y. and C.L.; writing—original draft preparation, R.S., M.Y., C.L. and A.S.; writing—review and editing, C.L. and P.K.J. All authors have read and agreed to the published version of the manuscript.

Conflicts of Interest

There are no conflicts of interest reported by the writers.

Funding: No funding support is available for this study.

Acknowledgment

The authors express gratitude to the Centre for Non-Conventional Energy Resources and the Department of Physics at the University of Rajasthan, Jaipur for providing synthesis and TG-DTA-DSC facilities. Authors also want to thank S.S. Jain Subodh PG College, Jaipur for helping with the XRD and FTIR measurements.

Data Availability statement

The authors confirm that all data supporting the findings of this study are available within the manuscript. The XRD pattern of g-C_3 N_4 is a reliable fingerprint of graphitic structure, with well-documented peaks corresponding to interlayer and in-plane atomic arrangements. These features are consistently reported in the literature, confirming both the crystalline framework and the reproducibility of the material across synthesis methods [This can find with JCPDS 87-1526].

Ethical Approval

No Applicable

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How to cite this article:
Sharma, R., Yadav, M., Gupta, N., Goyal, S., Jain, P.K., Singh, A., & Lal, C. (2025). The Structural, Optical and Thermal Characterization of g-C3N4 for Photocatalysis. Journal of Chemistry and Environment, 4(2), p. 56–62.