

Green Synthesis of Nitrogen-Doped Carbon Nanoparticles from *Ficus benghalensis* and *Allium sativum* for Methylene Blue Removal

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1. Introduction

Nanotechnology has emerged as one of the most rapidly advancing fields of science due to its ability to manipulate materials at the nanoscale (1–100 nm), resulting in unique physicochemical properties that differ significantly from those of bulk materials. Among various nanomaterials, carbon-based nanoparticles have attracted considerable attention because of their excellent chemical stability, high surface area, tunable surface chemistry, electrical conductivity, and environmental compatibility, making them suitable for environmental remediation and wastewater treatment applications [1,2].

Recent studies have demonstrated that biomass-derived carbon nanomaterials can be synthesized from a wide range of renewable resources including fruit peels, agricultural residues, food wastes, and plant-derived biomass. Such approaches provide sustainable routes for nanomaterial production while simultaneously promoting waste valorization and reducing environmental burdens associated with disposal of agricultural wastes [3–6].

The physicochemical properties of carbon nanoparticles can be further improved through heteroatom doping. Among various dopants, nitrogen has received significant attention because it enhances surface defects, modifies electronic properties, increases active adsorption sites, and improves the overall performance of carbon nanomaterials in environmental applications [7–10]. Conventional nitrogen-doping methods frequently utilize synthetic chemicals such as urea, ethylenediamine, ammonia, and dimethylformamide, which may increase production costs and environmental concerns [11–13].

Natural nitrogen-rich biomass offers an environmentally friendly alternative for nitrogen doping. Garlic peel (*Allium sativum*) contains naturally occurring nitrogenous compounds that can serve as sustainable nitrogen sources. Similarly, aerial roots of *Ficus benghalensis* have been reported as promising carbon precursors for carbon nanoparticle synthesis, while lemon peel extract contains organic acids and bioactive compounds capable of facilitating carbonization and surface functionalization [14–17].

Methylene blue (MB) is a cationic dye extensively used in textile, pharmaceutical, printing, and paper industries. Large-scale discharge of dye-containing effluents into aquatic environments causes serious environmental concerns, including reduced light penetration, oxygen depletion, and toxicity to aquatic organisms. Due to its aromatic structure and high stability, methylene blue is resistant to conventional biological treatment processes and may persist in aquatic systems for extended periods [18–20].

Adsorption has emerged as one of the most efficient and economically viable methods for dye removal owing to its simplicity, effectiveness, and ease of operation. Carbon-based nanomaterials have demonstrated excellent adsorption capabilities due to their high surface area and abundance of functional groups capable of interacting with dye molecules through electrostatic attraction, hydrogen bonding, and π - π interactions [21–24].

Therefore, the present study aims to synthesize nitrogen-doped carbon nanoparticles through a green route using *Ficus benghalensis* aerial roots, garlic peel waste, and lemon peel extract. The synthesized nanoparticles were characterized

using DLS, FTIR, SEM, XRD, and XRF analyses, and their suitability for methylene blue removal from aqueous solutions was investigated [25].

2. Materials and Methods

2.1 Materials Required

Ficus benghalensis (FB), commonly known as Banyan tree, belongs to the family Moraceae and is widely distributed throughout the Indian subcontinent. The aerial roots of the tree are rich in carbonaceous compounds and were selected as the primary carbon precursor. *Allium sativum* (AS), commonly known as garlic, belongs to the family Amaryllidaceae. Garlic peel waste contains naturally occurring nitrogen-containing compounds and was utilized as a sustainable nitrogen-doping precursor. *Citrus limon* (CL), commonly known as lemon, belongs to the family Rutaceae and contains abundant organic acids, flavonoids, and bioactive compounds. Lemon peel extract was used to facilitate carbonization and surface functionalization.

The aerial roots of *Ficus benghalensis* were collected, washed thoroughly with distilled water, dried in a hot-air oven at 105°C for 48 h, and pulverized into fine powder using a ball mill. Garlic peels were similarly washed, dried at 105°C for 8 h, and ground into fine powder. Lemon peels were washed and boiled in distilled water to obtain a concentrated extract.

2.2 Preparation of Nitrogen-Doped Carbon Nanoparticles

The synthesis procedure was adapted from biomass-derived nitrogen-doped carbon nanoparticle preparation methods reported in previous studies with slight modifications [14,15,25].

Initially, 3 g of *Ficus benghalensis* aerial root powder and 3 g of garlic peel powder were mixed thoroughly to obtain a homogeneous precursor mixture. Subsequently, 30 mL of lemon peel extract was added to the mixture and stirred continuously to form a uniform slurry.

The prepared mixture was transferred into ceramic crucibles and subjected to thermal carbonization in a muffle furnace at 300°C. During thermal treatment, the biomass precursors underwent dehydration, decomposition, and carbonization, leading to the formation of carbon-rich nanoparticles, as shown in Figure 1. Simultaneously, nitrogen-containing compounds present in garlic peel were incorporated into the carbon framework, resulting in nitrogen doping.

After carbonization, the product was allowed to cool naturally to room temperature. The obtained carbonized material was ground into a fine powder and stored in airtight containers for further characterization and methylene blue removal studies.



Figure 1. Carbonization of sample

2.3 Characterization Tests

The synthesized nitrogen-doped carbon nanoparticles were characterized using various analytical techniques. Dynamic Light Scattering (DLS) was employed to determine particle size distribution. The sample was dispersed in ethanol and sonicated to obtain a homogeneous suspension before analysis. Fourier Transform Infrared Spectroscopy (FTIR) was used to identify surface functional groups present on the nanoparticles. Scanning Electron Microscopy (SEM) was

performed to investigate particle morphology and surface characteristics. X-Ray Diffraction (XRD) analysis was conducted to evaluate the crystalline structure and degree of graphitization. X-Ray Fluorescence (XRF) analysis was used to determine elemental composition and confirm the presence of heteroatoms incorporated during synthesis.

2.4 Removal of Methylene Blue

A stock solution of methylene blue (1000 mg/L) was prepared by dissolving 1 g of methylene blue in 1000 mL of distilled water. Working solutions of 10, 20, 30, 40, and 50 mg/L were prepared by diluting 0.5, 1.0, 1.5, 2.0, and 2.5 mL of the stock solution, respectively, to a final volume of 50 mL using distilled water. For each experiment, 0.1 g of FB-AS-PG nanoparticles was added to 50 mL of methylene blue solution and stirred for 30 minutes before analysis.

3. Results and Discussion

3.1 Characterization of Nitrogen-Doped Carbon Nanoparticles

3.1.1 Dynamic Light Scattering (DLS) Analysis

The particle size distribution analysis revealed an average particle size of 82.57 nm, confirming successful formation of nanoparticles within the nanoscale range. The relatively small particle size can be attributed to the incorporation of nitrogen-containing compounds from garlic peel during thermal carbonization, which restricted excessive particle growth [15,25].

The nanoscale dimensions provide a large specific surface area and abundant active adsorption sites. Such characteristics are beneficial for methylene blue removal because they enhance contact between dye molecules and the nanoparticle surface. The reduced particle size also facilitates improved dispersion of nanoparticles in aqueous media, thereby increasing the availability of adsorption sites.

3.1.2 Fourier Transform Infrared Spectroscopy (FTIR) Analysis

The FTIR spectrum revealed the presence of various oxygen-containing and nitrogen-containing functional groups on the nanoparticle surface.

A broad absorption band observed around 3200–3500 cm^{-1} corresponds to O–H stretching vibrations associated with hydroxyl groups and adsorbed water molecules. Peaks observed near 1600–1700 cm^{-1} indicate C=C and C=O stretching vibrations arising from aromatic carbon structures and carbonyl groups. Bands in the range of 1200–1400 cm^{-1} correspond to C–N stretching vibrations, confirming successful nitrogen incorporation into the carbon matrix. Additional absorption bands attributed to N–O functional groups were also observed [7,14,26].

The presence of hydroxyl, carbonyl, carboxyl, and nitrogen-containing groups significantly enhances surface functionality. These groups contribute to dye adsorption through electrostatic attraction, hydrogen bonding, π – π interactions, and surface complexation mechanisms, thereby improving methylene blue removal efficiency.

3.1.3 Scanning Electron Microscopy (SEM) Analysis

SEM images demonstrated that the synthesized nanoparticles possess predominantly spherical morphology with relatively smooth surfaces. The particles were uniformly distributed and exhibited limited agglomeration [15,22].

The nanosized spherical structures provide a large surface area for interaction with dye molecules. The observed surface roughness and porous features further increase the number of accessible adsorption sites. Uniform particle distribution indicates efficient carbonization and successful incorporation of nitrogen-containing species within the carbon framework.

The morphological characteristics observed through SEM suggest that the synthesized nanoparticles are suitable adsorbents for aqueous pollutant removal applications.

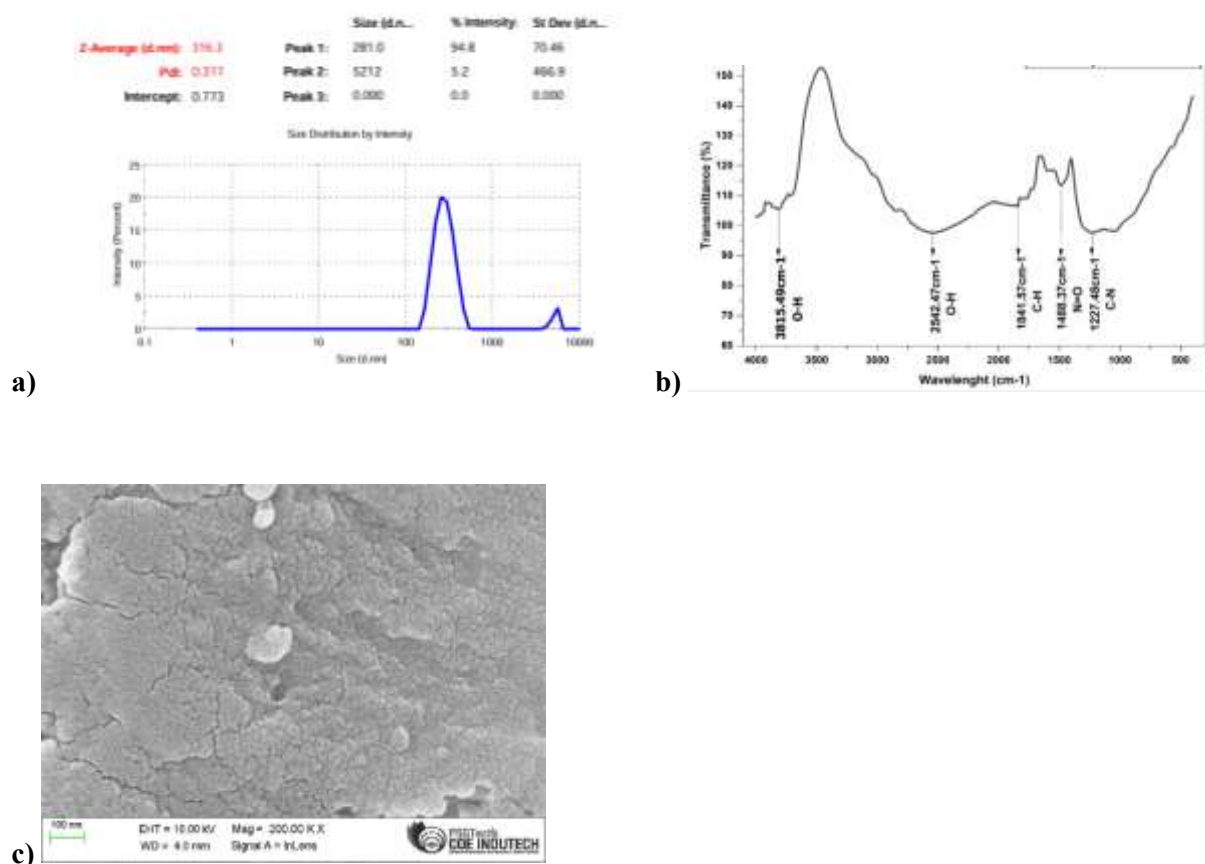


Figure 2. Characterization results of synthesized FB-AS-CL nitrogen-doped carbon nanoparticles: (a) DLS particle size distribution, (b) FTIR spectrum, (c) SEM micrograph

3.2 Methylene Blue Removal Performance

The methylene blue removal performance of the synthesized FB-AS-PG nitrogen-doped carbon nanoparticles was evaluated at different initial dye concentrations ranging from 10 to 50 mg/L, as shown in Table 1. The experiments were conducted using a fixed adsorbent dosage of 0.1 g/L, contact time of 30 min, and pH 7. The results demonstrated that the removal efficiency decreased with increasing initial methylene blue concentration, which may be attributed to the saturation of available adsorption sites on the nanoparticle surface at higher dye concentrations, as shown in Figure 3.

Table 1. Removal of MB experiments

Initial MB Concentration (mg/L)	Adsorbent Dosage (g/L)	Contact Time (min)	pH	Removal Efficiency (%)
10	0.1	30	7	96.8
20	0.1	30	7	92.4
30	0.1	30	7	88.7
40	0.1	30	7	84.9
50	0.1	30	7	80.6

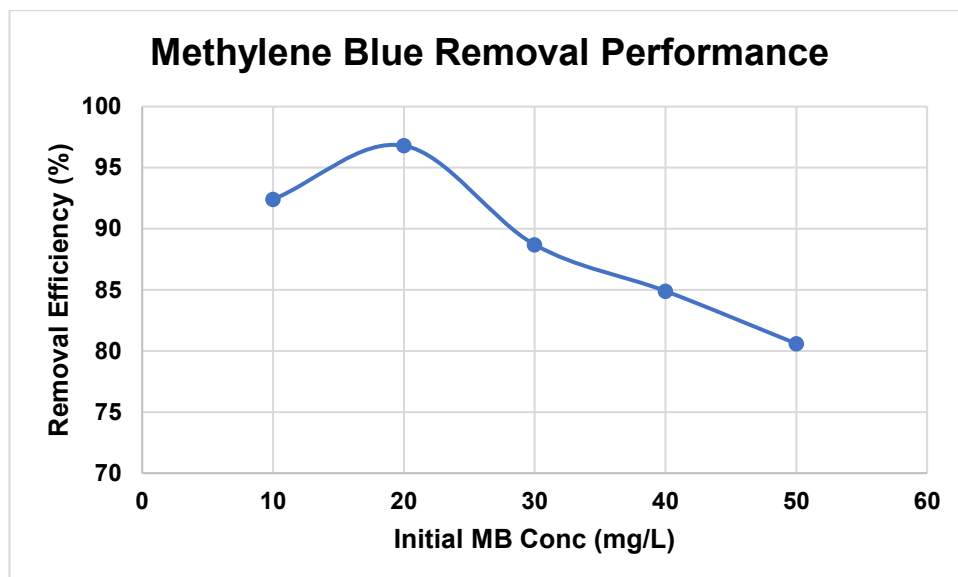


Figure 3. Methylene Blue Removal

4. Conclusion

In the present study, nitrogen-doped carbon nanoparticles were successfully synthesized through a green and sustainable approach using *Ficus benghalensis* aerial roots, *Allium sativum* peel waste, and *Citrus lemon* peel extract. The synthesis method utilized readily available biomass resources and eliminated the need for hazardous chemical reagents, making the process environmentally friendly and cost-effective.

Characterization studies confirmed the successful formation of nitrogen-doped carbon nanoparticles. DLS analysis revealed an average particle size of 122.0 nm, while FTIR analysis confirmed the presence of hydroxyl, carbonyl, and nitrogen-containing functional groups. SEM images showed aggregated nanostructures with rough surface morphology, providing potential adsorption sites for dye molecules.

The synthesized FB-AS-CL nanoparticles demonstrated effective methylene blue removal from aqueous solutions. The highest removal efficiency of 96.8% was achieved at an initial dye concentration of 10 mg/L, while removal efficiency gradually decreased with increasing dye concentration due to the limited availability of adsorption sites. The results suggest that the synthesized nitrogen-doped carbon nanoparticles possess favorable physicochemical properties for dye removal applications.

Overall, the study demonstrates that biomass-derived nitrogen-doped carbon nanoparticles can serve as promising and sustainable adsorbents for the removal of methylene blue from wastewater, offering a potential low-cost solution for environmental remediation.

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