



## Synthesis of Cadmium Sulfide Nanoparticles Via Green Route for Photocatalytic Degradation of Methylene Blue Dye

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### ABSTRACT

This study presents a sustainable approach for the generation of cadmium sulfide nanoparticles (CdS NPs) employing *Hibiscus rosa-sinensis* leaf extract and evaluates their performance in the light-driven breakdown of methylene blue (MB) dye. The method of synthesis utilized the framework of green chemistry, ensuring minimal chemical usage and waste production. Multiple analytical techniques such as Fourier transform IR (FTIR) spectroscopy, X-ray diffraction (XRD) analysis, and scanning electron microscopy (SEM), were utilized in order to verify the formation and structural characteristics of the synthesized NPs. Photocatalytic efficacy of CdS NPs was assessed by analyzing MB dye degradation in the presence of natural sunlight. The synthesized NPs demonstrated significant photocatalytic activity, achieving effective degradation of the dye within a short time frame. This highlights their potential application in industrial wastewater (Textile, Paper, Leather, Plastic etc.) treatment.

**Keywords:** Cadmium sulfide nanoparticles, *Hibiscus rosa-sinensis*, Eco-friendly synthesis, Photocatalytic activity, Methylene blue.

### INTRODUCTION

Recent advancements in nanotechnology have accelerated to meet societal demands, particularly in material control through diverse synthesis methods such as hydrothermal, sol-gel, ultrasonication, and microwave irradiation<sup>1-5</sup>. These methods produce nanomaterials with distinct chemical, physical, and optical characteristics. Among these, NPs have gained interest for use

in photocatalytic degradation due to their size, shape, and surface area<sup>6-9</sup>. NPs, with sizes varying from 1 nm to 100 nm, can be synthesized through a variety of techniques. However, many traditional approaches such as hydrothermal and solvothermal techniques involve toxic chemicals, high costs, generation of toxic waste, and significant energy use, emphasizing the need for eco-friendly synthesis methods using plants extracts and microorganisms<sup>10-12</sup>.



Among green synthesis approaches, the utilization of plant extracts has attracted significant attention. Plants, including their leaves, fruits, roots, and stems, act as natural reducing and stabilizing agents due to their rich content of active compounds. Biosynthesis using the plant *Hibiscus rosa-sinensis* has gained attention due to its active compounds like tannins, alkaloids, phenols, and flavonoids facilitating the NP synthesis in reducing and stabilizing the metal salts<sup>13,14</sup>.

Among photocatalysts, CdS NPs belong to the group of chalcogenides; a II-IV group semiconductor NPs which are promising because of their narrow band gap, excellent photo response under visible light, and ability to breakdown organic pollutants like synthetic dyes<sup>15-18</sup>. With growing industrialization, pollutants like MB-a widely used but toxic synthetic dye pose significant environmental and health risks. MB's resistance to natural degradation disrupts aquatic ecosystems, necessitating eco-friendly removal strategies<sup>10,19,20</sup>.

Photocatalysis, which uses light energy to generate electron-hole pairs and drive redox reactions, is an effective method for organic pollutants degradation. This process produces highly oxidized hydroxyl radicals ( $\cdot\text{OH}$ ) that decompose contaminants. Unlike traditional methods such as adsorption, ozonation, and membrane filtration, photocatalysis is cost-effective, highly efficient, low cost, and environmentally compatible, making it a preferred choice for removing synthetic dyes<sup>6,21-24</sup>.

The present work explores the green production of CdS NPs utilising extract from *Hibiscus rosa-sinensis* leaves as a natural reducing agent. Structural and morphological aspects of the prepared NPs were analyzed, and their photocatalytic efficacy was assessed by degrading MB dye under sunlight. This study emphasizes a sustainable strategy for NP synthesis and wastewater treatment.

## MATERIALS AND METHODS

### Synthesis of CdS nanoparticles

*Hibiscus rosa-sinensis* leaves were washed with de-ionized water. Then they were dried and crushed to make fine powder with the aid of a blender. A mixture of 150 mL of de-ionized water, 10 mL of methanol and 10 g of the powdered leaves

were heated for 30 min at 100°C. The solution was left to cool until it reached room temperature and then subjected to filtration with Whatman No. 1 filter paper<sup>25</sup>. A 10 mL of aqueous *Hibiscus rosa-sinensis* leaf extract was introduced to 150 mL of cadmium sulfate solution (0.1 M) and agitated at 60°C for 2 hours. Hydrogen sulfide gas was then introduced into the mixture until a bright yellow precipitate of CdS NP was formed. Then the solution was allowed to cool and settle down. Finally, the mixture was filtered and the precipitate was given wash first with acetone followed by deionised water to remove the contaminants if present and dried completely for 24 h at 50°C.

### Characterization of CdS nanoparticles

The FTIR spectrum of CdS NPs was measured over the span of 4000 to 600  $\text{cm}^{-1}$  with the help of a PerkinElmer spectrophotometer. The XRD pattern was analysed using Rigaku Powder X-ray diffractometer using Cu-K $\alpha$  radiation ( $\lambda$ :1.5406 Å) scanning over  $2\theta$  interval of 20° to 65°. The surface features and elemental mapping of CdS NPs were investigated by using a Carl Zeiss SEM coupled with EDS.

## RESULTS AND DISCUSSION

### FTIR analysis

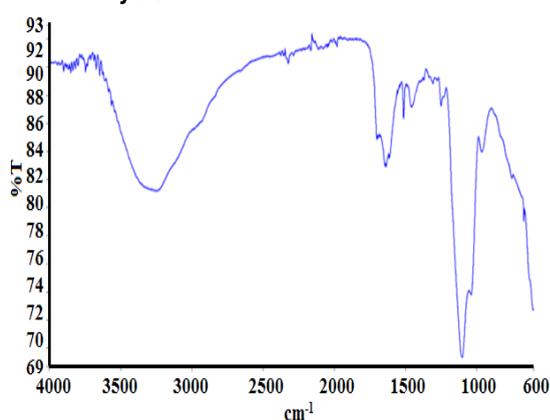


Fig. 1. FTIR spectrum of *Hibiscus rosa-sinensis* mediated CdS NPs

The IR spectrum of CdS NPs synthesized using *Hibiscus rosa-sinensis* extracts is depicted in Fig. 1. A broad and distinct peak at 3350  $\text{cm}^{-1}$  is assigned to the O-H stretching modes of hydroxyl ( $-\text{OH}$ ) units. A weak band occurring at 2982  $\text{cm}^{-1}$  is attributed to the C-H bond stretching of alkanes and alkyl groups. A signal at 1613  $\text{cm}^{-1}$  is linked to the

C=O bond stretching vibrations, while the signals at  $940\text{ cm}^{-1}$  and  $1108\text{ cm}^{-1}$  are associated with the stretching of aromatic C-H and C-O, respectively. These absorption bands are predominantly by the functional moieties contained in the extract of leaf. Additionally, an absorbance band at  $650\text{ cm}^{-1}$  marks the synthesis of CdS NPs.

### XRD Pattern Analysis

The diffraction pattern of CdS NPs exhibited sharp diffraction peaks (Fig. 2), signifying a well-defined crystalline structure. The diffraction signals appearing at  $2\theta$  angles of  $26.56^\circ$ ,  $43.92^\circ$ , and  $51.88^\circ$  confirmed the cubic phase of CdS NPs<sup>26,27</sup>. Additionally, the absence of any diffraction peaks corresponding to potential impurity phases indicated the successful synthesis of phase-pure cubic CdS NPs.

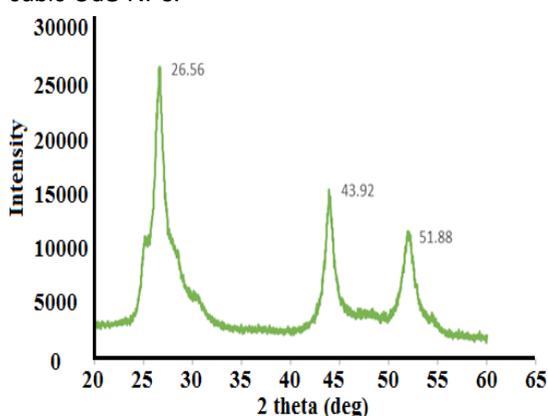


Fig. 2. XRD pattern of *Hibiscus rosa-sinensis* mediated CdS NPs

### Scanning electron microscopy and elemental study

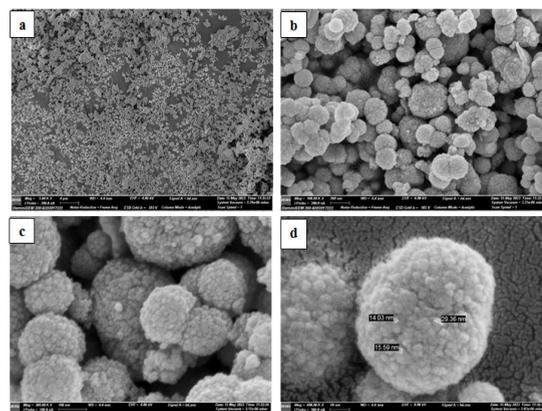


Fig. 3. SEM micrographs of the eco-friendly synthesized CdS NPs at magnifications: (a) x 5, (b) x 200, (c) x 300 and (d) x 400

The structural features of the synthesized CdS NPs was carried out using SEM, as depicted in

Fig. 3. The micrographs reveal that the NPs formed are close to being spherical and exhibit significant agglomeration. Their sizes vary, maintaining a predominantly spherical structure with some degree of clustering.

Further, the elemental composition of green synthesized CdS NPs was determined by Energy Dispersive X-Ray Spectroscopy (EDS). EDS spectrum in Fig. 4 clearly showed the characteristic signals for cadmium (Cd) and sulfur (S). This analysis revealed that the sample consists predominantly of Cd and S, with atomic percentages of 54.93% and 45.07%, respectively. The EDS spectrum further confirmed the existence of peaks for Cd and S, indicating the generation of absolute CdS without any additional elemental impurities.

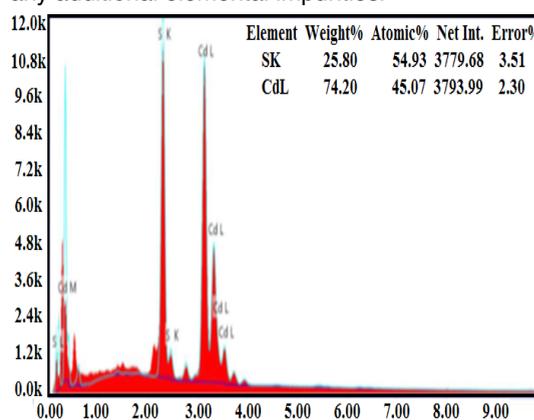


Fig. 4. EDS pattern of CdS NPs synthesized using *Hibiscus rosa-sinensis* leaf extract

### Photocatalytic activity of CdS nanoparticles

The green synthesized CdS NPs were utilized as photocatalysts for MB dye degradation. A comparative study was conducted to assess the efficiency of CdS NPs as photocatalysts in dye degradation. In a typical experiment, first set (Set 1) involved adding 5 mg of CdS NPs to 100 mL of an aqueous MB solution (100 ppm). In the second set (Set 2), the MB solution was prepared with the same concentration and volume as that of the Set 1 but without the addition of CdS NPs. Both the sets were agitated under dark condition for 30 min to attain chemisorption equilibrium. Subsequently, the solutions were subjected to sunlight for 2 h while stirring to initiate dye photo degradation. At 30 min intervals during the process, 5 mL of the solution was withdrawn from each set into centrifuge tubes. After centrifugation, the

solutions were analysed with a Dual Beam Cary 60 UV-Visible spectrophotometer to monitor dye absorbance over time and assess the degradation progress.

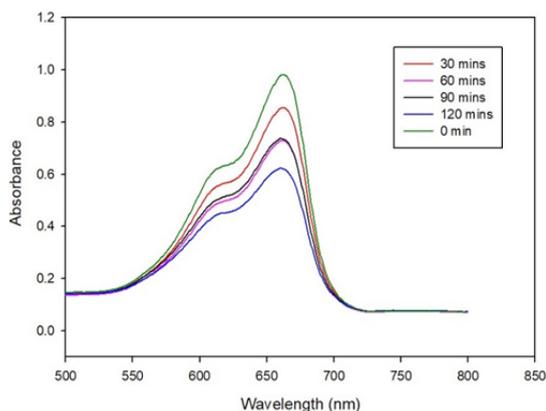


Fig. 5. Absorbance spectrum of set 1 (with CdS NPs) for MB degradation

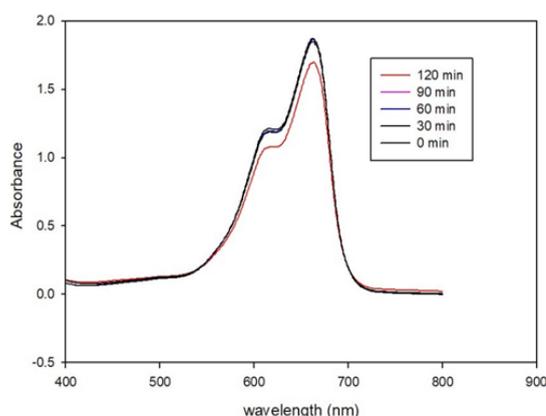


Fig. 6. Absorbance spectrum of set 2 (without CdS NPs) for MB degradation

For Set 1, the absorbance of MB progressively decreased over time due to its degradation, as shown in Fig. 5. The degradation percentage reached 30.55% within 120 minute. On the other hand, the Set 2 exhibited minimal variation in absorbance, indicating negligible degradation Fig. 6. Fig. 7 illustrates the percentage degradation over time intervals for both the sets as determined through UV-Visible analysis. The degradation percentage was determined using the following equation:

$$\% \text{ of degradation} = \frac{C_0 - C_t}{C_0} \times 100$$

Here,  $C_0$  ( $\text{mg L}^{-1}$ ): initial concentration, and  $C_t$  ( $\text{mg L}^{-1}$ ): concentration of MB dye at time  $t$  after degradation.

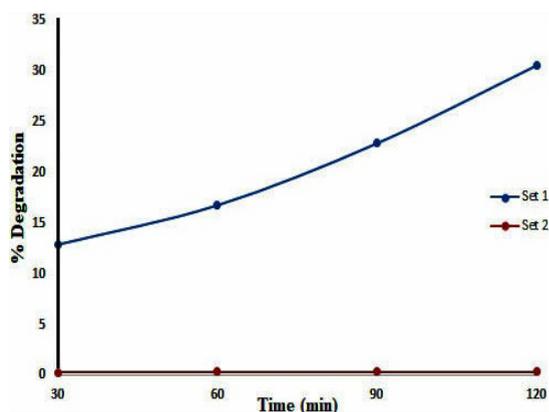


Fig. 7. Degradation percentage of MB at various intervals of time for Set 1 and 2

### CONCLUSION

The current study demonstrates the successful green synthesis of CdS NPs utilising *Hibiscus rosa-sinensis* leaf extract. The synthesized CdS NPs exhibited significant photocatalytic activity, achieving up to 30.55% degradation of 100 ppm concentration of MB dye within 120 min in the presence natural sunlight. This highlights their potential as an efficient and bio-based photocatalyst for the elimination of synthetic dyes present in wastewater. The structural, morphological, and compositional analyses confirmed the formation of high-purity, phase-specific CdS NPs, which can serve as a sustainable alternative for environmental remediation. While cadmium is known to be hazardous to the environment, utilizing its NPs in very small amounts for significant environmental pollution remediation is a justifiable approach, balancing efficiency with controlled usage.

### Future perspectives

This work opens up the way for further exploration into optimizing synthesis conditions and investigating key factors such as NP concentration, temperature, reaction time, solution pH, and dye concentration to improve CdS NPs photocatalytic performance. Additionally, studies on the long-term stability and reusability of these NPs are essential to ensure their practical and cost-effective application. Expanding the scope to include the degradation of various hazardous organic and inorganic pollutants in wastewater can further enhance their utility. By addressing these critical aspects, green synthesized CdS NPs can contribute to water pollution management and sustainable environmental solutions.

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**Conflict of interest**

The authors do not have any conflict of interest.

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