Photocatalytic Activity of CdS Nanosheets Prepared by Chemical Bath Deposition Method

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Abstract. CdS nanosheets are synthesized via the chemical bath deposition method (CBD) using three different Cd/S molar ratios for the photocatalytic degradation of methylene blue (MB), as an example of an organic pollutant. The resulting nanosheets were characterized by XRD, FESEM, and UV-VIS spectroscopy. The XRD data analysis for prepared powder resulted in mixed phases of zinc-blende and wurtzite in all different Cd/S molar ratios with average crystalline size increased from (50.9 nm to 59.7 nm). The as-prepared CdS shows an absorbance edge increased from (340 nm, to 490). The value of the bandgap decreased from (2.6 eV to 2.3 eV) with an increasing molar ratio. Increasing molar ratio also leads to high and stable photocatalytic with high degradation efficiency (92.7-98.4 %) after 150 min of sunlight irradiation.

Graphical Abstract

Keywords: CdS; Chemical bath deposition (CBD); Nanosheet; Photocatalytic activity; Methylene blue.
1. Introduction

In recent years semiconductors used to be as photocatalysis for the degradation of organic pollutants under visible light irradiation [1]. CdS have recently attracted much attention because of its wide probable applications in environmental protection, solar cell, sensor technology, and other correlated fields [2]. CdS is an n-type semiconductor having a narrow bandgap [3] therefore, CdS can absorb visible or ultraviolet light with a wavelength less than 514 nm, which makes CdS more efficient in photocatalysis for visible light [4]. CdS exist in three different crystal structures, namely wurtzite, zinc blend and high-pressure rock-salt phase [5,6]. Among the first two crystal forms, the wurtzite type is usually considered to be the most photocatalytically active, it is certainly the most widely used and researched stage because it has higher stability and simplicity of production [6].

In principle, morphological influence, crystalline size, and Structure the characteristics of semiconductors [7]. These variables largely depend on the preparation method and experimental conditions [8]. Various sizes and shapes of nanoparticles, including spherical nanoparticles, have been successfully synthesized in recent years. [7, 9]. Flower-like, fishbone-like, branch-like [1], Nanosheet [10]and nanorods [11]. Many methods have been employed for the synthesis of CdS nanostructures such as co-precipitation [12], solvothermal [13], hydrothermal [14, 15] sonochemical [16], sol-gel method [17], method including the microwave-assisted polyol synthesis [18], and chemical bath deposition [19, 20, 21]. In addition, since size and morphology play an important role in the physical and optical properties of various forms of CdS, it is reported that the size and shape of the growing crystal can be adjusted by understanding its crystal symmetry and growth manner. [9, 18, 22].

Among these methods, chemical bath deposition (CBD) simple lab equipment, ambient has much attention has been paid due to the benefits of low cost, excellent efficiency and high production potential on a large scale [19,22]. Since the past two decades, the progress of photocatalysts has not only become a challenge, but also an interesting research subject. Removal of organic dyes in a short time as a result of a photocatalytic reaction have been advanced in recent years.

Heterogeneous semiconductors photocatalysis occurs in photocatalysts surface under the illumination of external photons. CdS photocatalysts are simply excited to form photogenerated electrons and holes, and have been widely used in the photodegradation of organic pollutants in sewage under visible light. In this work, CdS nanosheets are synthesized via chemical bath deposition method, then the effects of Cd: S molar ratio on structure morphological and optical properties were studied. The photocatalytic performance of CdS nanosheets toward photodegradation of methylene blue dye under natural solar light irradiation. The current research could lead to a realistic and cost-effective wastewater treatment process.
2. Experimental

2.1 Materials

The chemicals used in this study were high-purity of Cadmium chloride Monohydrate (CdCl₂·H₂O, 99%; CDH), thiourea extra pure (CH₄N₂S, 99%; Scharlau), tri-Sodium citrate dihydrate (C₆H₅Na₃O₇·2H₂O, 99%), Ammonia solution (NH₃, > 99%, 32% w/w; Scharlau), and MB pure (Acros Organics).

2.2 Preparation of CdS nanosheets

CdS nanosheets fabricated by low-cost chemical bath deposition (CBD) technique. Double-distilled water was used as the solvent, 100 ml aqueous solution of cadmium chloride CdCl₂·H₂O around (0.25, 0.5, and 1) g added with its (0.25) g tri-Sodium citrate as capping agent and so we have solution A. 10 ml aqueous solution of thiourea in a different ratio (0.07, 0.14, 0.28) g to get solution B, this solution was added to solution (A) drop wise under constant magnetic stirring for 30 min followed by heat treatment at 50 °C. After complete mixing, the pH was adjusted to 10 by adding NH₃ drop by drop. The solution was placed in a chemical bath whose temperature was kept around 70-80 °C during the growth and the deposition process.

Primarily the solution was transparent, after some time it altered into yellow colour and the deposition time was 2h. The precipitate was separated from the solution via centrifugation then the separated powder was washed several times with ethanol and distilled water and dried at 60 °C. CdS powder results in three different concentrations named C1, C2, and C3 respectively.

This powder was used to record X-ray diffraction (XRD) analysis, which were used to distinguish the crystalline phase and crystallite size of CdS powder, and conducted on an X-ray diffractometer using CuKa (PhillipsXpert, Holland). The morphological studies of the nanosheets were done by using Field Emission Scanning Electron Microscopy (FESEM) (Tescan Mira3, France). Optical absorbance spectra of all samples were recorded using UV–visible spectrophotometer (Optima Sp-3000 plus UV-Vis-NIR (Split- beam Optics, Dual detectors) spectrophotometer in the spectral range 200-900 nm. The obtained CdS powder then employed for photocatalytic experiments.

2.3 Photocatalysis experiments

Study the photocatalytic activity of CdS powder by degrading MB standard solution in a photochemical vessel with a concentration of 10 mg/L in distilled water used as an organic pollutant model to measure the photocatalytic activity of CdS nanosheets. About 50 mg of CdS nanopowder was added into a glass beaker containing 50 ml of MB aqueous solution. The solution is added into the photocatalytic vessel and stirred for 1h in the dark to confirm that the adsorption and desorption equilibrium of MB is established on CdS surface. Subsequently, under constant magnetic stirring, the suspension was irradiated with sunlight to maintain its stability. Every 30 minutes, 4 ml of the suspension was extracted and then centrifuged for 15 minutes to separating the powder from the solution. Ultraviolet-visible analysis is used to measure the absorbance changes of supernatants with
wavelengths between 400 nm and 800 nm over time. After removing the color of the MB dye solution, the absorbance value was measured at $\lambda = 660$ nm. The same experimental process was applied for all three concentrations.

3. Results and discussion

The structures analysis and properties of CdS powder were confirmed by XRD and shown in figure 1. Cubic (C) and wurtzite hexagonal (H) phases were obtained in all three different concentrations C1, C2, and C3.

The peaks shown in the XRD patterns correspond to the (100), (002), (101), (102), (110), (103), (200), (112), (004) and (202) of CdS hexagonal phase (JCPDS no. 06-0314) while (111), (220), (311), (222), and (420) crystal planes of cubic CdS phase (JCPDS no. 01-089-0440). Crystallite sizes were calculated using Scherrer formula [23]:

$$D = \frac{K\lambda}{\beta \cos \theta} \quad \text{............. (1)}$$

Where D is the crystallite size, $\lambda$ is the wavelength of radiation equal to 1.5408 Å, $\beta$ is the FWHM (full width at half maximum) and $\theta$ is the Bragg angle and k is a constant equal to 0.9, assuming that the particles are spherical.

The diffraction peaks were used to measure the crystallite sizes of (002) and (111) corresponding to hexagonal and cubic crystalline planes respectively. The crystallite sizes of the samples were calculated to be 20.7, 41.4, and 55.1 nm for the concentrations C1, C2 and C3 respectively with average crystal size 50.9, 51.0 and 59.7 nm respectively.

The lattice constants of CdS Nanosheet hexagonal $a = b = 0.413$ nm, $c = 0.670$ nm were calculated using the following equation:

$$\frac{1}{d^2} = \frac{4}{3} \left( \frac{h^2 + h^2 + k^2}{a^2} \right) + \frac{l^2}{c^2} \quad \text{........... (2)}$$

While the lattice constants of cubic CdS $a = b = c = 0.585$ nm calculated using the following equation:

$$\frac{1}{d^2} = \frac{h^2 + k^2 + l^2}{a^2} \quad \text{.............(3)}$$

Where d is the interplanar distance, and $h, k,$ and $l$ are Miller indices. Moreover, $a$ and $c$ are lattice constants. These are in good agreement with JCPDS card. However, the increase in concentrations apparent clear changes and an increase in the intensity of the major peaks with the appearance of some peaks in the third concentration.
Field Emission Scanning Electron Microscopy was used to determine the surface morphology properties of CdS Nanosheets. The morphologies of the as-grown CdS Nanosheets indicate that the structures were responsive to changes in cadmium and sulfur concentrations, as shown in Fig. 2. Nanosheets were convergent with each other in the first concentration (C1) and appeared to be dianthus flowers like with a diameter of 26.18 nm. While in (C2) concentration, the density of particle distribution on the surface increases and it makes a homogeneous structure with a 20.8 nm diameter. With increasing the concentration, the structure becomes sheets more smooth and homogeneous that resemble bone marrow tissue with a 21.6 nm diameter in the third concentration (C3).
Fig. 2 FESEM images of CdS nanosheets at various concentrations C1, C2 and C3.
UV–vis spectroscopy was used to obtain the optical properties and energy bandgap. Fig.3 shows the optical absorption spectra of CdS nanosheets. In comparison with C3, an apparent blue shift can be found in C1 and C2, because of quantum confinement effects [14]. However when the size of nanomaterial decreases the absorption peak shifts to higher energies[3] which make the value of the bandgap increased as shows in C1 and C2. The as-prepared CdS shows an absorbance edge near 340 nm, 480nm and 490 for C1, C2 and C3 respectively. This indicates CdS samples have a wide absorption range in the visible light spectrum.

![Fig. 3 optical absorption spectra of CdS nanosheets at various concentrations (C1, C2 and C3).](image)

Fig. 4 shows the direct energy band gap of prepared CdS, with the values (2.6 eV, 2.4 eV, and 2.3 eV) C1,C2 and C3 respectively this decrease in energy gap values with increasing concentrations matches The change in the energy gap corresponds with the previous reports [25].

![Fig. 4 energy band gap of CdS nanosheets at various concentrations (C1, C2 and C3).](image)
The photocatalytic behavior of C1, C2, and C3 samples in visible sunlight was tested by degrading MB dye aqueous solution. The change in intensity of the MB’s absorption peak was used to measure dye degradation. The absorption spectra of the C1, C2, and C3 sample solutions taken at different times (shown in Fig.5) show that at the beginning (t=0) the spectrum exhibit the intense absorption peak of MB at 660nm.

![Fig. 5 UV–vis absorption spectra of photocatalytic degradation of MB by (C1, C2, and C3) CdS photocatalysts at various time intervals.](image)
Mechanistically photocatalytic reaction of CdS is initiated when CdS irradiation by sunlight. Emission of electrons from the valence band to the conduction band produces electron-hole pairs (Eq.4)[26,27]. OH radicals are formed when the holes in CdS react with water molecules or hydroxide ions (Eq.5). Oxygen is usually provided as an electron acceptor to extend the recombination time of electron-hole pairs through photocatalytic oxidation (Eq.6). The OH radical is an effective oxidizing agent (Eq.7), and it attacks organic pollutants present at or close to the surface of CdS (Eq.8 and Eq.9), as seen in (Fig.6).

\[
\text{CdS} + \text{hv (visible)} \rightarrow \text{CdS (eCB -+ hVB+)} \ldots (4)
\]

\[
\text{CdS (hVB+)} + \text{H2O} \rightarrow \text{CdS} + \text{H+} + \text{OH} \ldots \ldots (5)
\]

\[
\text{CdS(eCB-)}+\text{O2} \rightarrow \text{CdS+O2} \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots (6)
\]

\[
\text{H+O2} \rightarrow \text{HO2} \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots (7)
\]

\[
\text{MB+OH*} \rightarrow \text{MB*+H2O} \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots (8)
\]

\[
\text{MB*+ OH} \rightarrow \text{Degradation products} \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots (9)
\]

![Fig.6 Schematic diagram of CdS photocatalyst photodegradation mechanism of MB dye under sunlight.](image)

Prepared powder, C1, C2 and C3 have a high degradation efficiency (92.7%, 96.8% and 98.4% respectively) after 150 min of sunlight irradiation as shown in Fig.7. Increases in concentration lead to an increase in photocatalytic efficiency as a greater contact area between the photocatalyst and dye molecule is formed, resulting in the maximum adsorbent effect [28] for MB dye molecules, resulting in a higher rate of degradation of MB dye molecules when exposed to sunlight. Table .1 displays the outcome result of this work.
Table 1: Outcome result of CdS nanosheets at various concentrations (C1, C2 and C3).

| Samples | Molar ratio of Cd/S source (mole) | Average crystallite sizes (nm) | Absorbance edge (nm) | Energy Gap (eV) | Degradation efficiency |
|---------|-----------------------------------|-------------------------------|----------------------|-----------------|------------------------|
| C1      | Cd : 0.01 S : 0.09                | 50.9                          | 340                  | 2.6             | 92.7 %                 |
| C2      | Cd : 0.02 S : 0.18                | 51                            | 480                  | 2.4             | 96.8 %                 |
| C3      | Cd : 0.04 S : 0.27                | 59.7                          | 490                  | 2.3             | 98.4 %                 |

Fig. 7 Degradation efficiency of MB using CdS nanosheets at various concentrations (C1, C2 and C3).

At absorption peak (660 nm) the rate of time-dependent photodegradation $C/C_0$ of MB is shown in Fig. 8 for CdS Nanosheet.
Fig. 8 Time dependent photodegradation of MB under sunlight irradiation of CdS nanosheets at various concentrations (C1, C2 and C3).

The first order pseudo rate constant $k$ can be calculated using the slope of the line from Fig. 9. The fairness of the fit is indicated by the fact that the linear regression or correlation coefficient ($R^2$) values reaches to 0.984, 0.905 and 0.872 for C1, C2 and C3 which is much closed to one. This result implies that the photocatalytic degradation kinetics can be successfully described by the pseudo first-order model. Table 2 compares current research and other studies on CdS nanostructure photocatalytic application.

![Graph showing first order kinetics of the degradation of (MB) dye with irradiation time of CdS nanosheets at various concentrations (C1, C2 and C3).]

**Fig. 9** first order kinetics of the degradation of (MB) dye with irradiation time of CdS nanosheets at various concentrations (C1, C2 and C3).

**Table 2:** Comparison of degradation by using various CdS nanostructures as photocatalysts.

| Reported        | Catalyst | Dye     | Concentration | Catalyst loading | Light source       | Time (min) | Degradation (%) |
|-----------------|----------|---------|---------------|------------------|--------------------|------------|-----------------|
| Teeradech et al. (2017) | CdS red azo (RR141) | 10 mg L$^{-1}$ | 50 mg/200 mL | 1. visible-light | 240 | 94.6% | [8] |
| Xiande et al. (2019) | CdS MB | 12 mg L$^{-1}$ | 0.0050 g/mL | Visible-light | 240 | 90.0% | [1] |

Note: The degradation efficiency is shown as a 24-hour result for comparison.
4. Conclusions

CdS Nanosheet was successfully synthesized using chemical bath deposition method with CdCl₂ and thiourea as the precursor. This is a low-cost and easy-fabrication method. XRD measurements showed that the crystallite size increases when the concentrations are increased. The degradation efficiency increased with increasing concentration and reaches to 98.4 % after 150 min under sunlight irradiation. The degradation efficiency of MB by synthesized samples increases as the concentrations increases of the CdS nanosheets.

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| Yang Tan et al. (2020) | CdS | RbB | 7 mg L⁻¹ | 50 mg/40 mL | UV-Visible light | 120 min | 93% |
|----------------------|-----|-----|-------|----------------|----------------|---------|-----|
| In this work | CdS | MB | 10 mg L⁻¹ | 50 mg/50 mL | Natural solar light | 150 min | 98.4% |

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