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RESEARCH ARTICLE

Photocatalytical Degradation of Methylene Blue using Laboratory Prepared Cadmium Sulphide

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Abstract

The cadmium sulphide (CdS) has been successfully synthesized in laboratory by chemical precipitation method. Cadmium chloride and sodium sulfide were used as precursor for the synthesis of CdS photocatalyst. The synthesized material was characterized by Fourier Transform Infrared Spectroscopy, X-ray diffraction, Scanning Electron Microscopy. The XRD peaks are obvious at 25.05, 26.9, 28.29, 43.9, 47.9, 52.07, 2θ degrees which indicates the crystalline phase of as prepared CdS material and average crystalline size was found to be 5.25 nm. Synthesized CdS nanoparticles were used to degrade the methylene blue (MB) dye under the irradiation of UV light. Different parameters like pH, concentration of MB dye, dose of CdS photocatalyst, UV irradiation time were evaluated. The preeminent pH of MB solution was found to be 9, the optimum catalyst dose was found to be 100 mg. Maximum 96% MB degradation was established when 1% of 1mL H_2O_2 was added along with 100 mg photocatalyst with continuous 210 minutes irradiation of UV light. The H_2O_2 provides sufficient •OH radicals and upsurges the efficiency of CdS photocatalyst. The results revealed that laboratory prepared CdS nanoparticles could be a good material to use as a photocatalyst for the degradation of cationic dye like MB.

Keywords: Photocatalyst; Degradation; Methylene Blue dye; Cadmium sulphide

Introduction

In recent years, extensive study and research has been concentrated on semiconductor materials like ZnO, TiO_2, Cu_2O, Bi_2WO_6, WO_3, Bi_2O_3, ZnS, ZrO_2, $CdWO_4$, NiO, Ga_2O_3, $BiMoO_6$, $ZnWO_4$ [1-4] for the purpose of photocatalyst in different fields like dye degradation in water, sterilization of bacteria, molds, pests, algae [5], removal of air pollutants like nitrous oxide, formaldehyde, carbon monoxide, sulfur oxide [6-7]. Some other applications of photocatalyst are conversion of energy on solar cell [8], self-cleaning materials on the surface of glass and shows anti-fogging activity on the side mirror of car [9]. In this study, CdS has been prepared, characterized and was used for the degradation of methylene blue (MB) dye at different reaction conditions.

The CdS is one of the most significant semiconductor and has attracted a lot of attention from researchers and scientists for possible uses in pollution remediation, optoelectronics, nanodevices, and biological labeling in the future, because of its tunable electronic band gap, which depends on the size and shape of the nanocrystals [10]. The photocatalytic activity of CdS is notable because of its photosensitization, which enables the injection of photogenerated electrons from the light-activated CdS's conduction band into the non-activated nanomaterials. Additionally, the holes on the CdS particle persisted, which significantly increases the photocatalytic activity of other nanostructures [10].

Materials and Methods

The X-ray diffraction patterns were recorded

using Bruker D2 Phaser. The FTIR spectra were recorded by using PerkinElmer Spectrum IR version 10.6.2. Visible Spectrophotometer 2306, AB 1908003, Electronics India has been used for the determination of required spectrophotometric data. The zeta potential was measured by using Horiba Scientific SZ-100V2, Japan. The SEM images of prepared CdS was recorded by Zeiss in China. LR grade cadmium chloride ($CdCl_2$) was obtained from S.D. Fine-CHEM Limited, Mumbai. Similarly, LR sodium sulphide (Na_2S) has been purchased from Merck Limited, Mumbai. The hydrogen peroxide was obtained from Nike Fine Chemicals, India whereas methylene blue was procured from Merck Life Science Private Limited, India.

Preparation of Cadmium Sulphide

The cadmium sulphide was prepared by the chemical precipitation method. The 150 mL of 0.5 M cadmium chloride was taken in a beaker and 150 mL of 0.5M sodium sulfide was added drop wisely with constant stirring in the magnetic stirrer for 4 hrs. The yellow precipitate was formed which was filtered and washed 5 times with distilled water and 5 times with ethanol. Then it was dried in an oven and further sintered for 2 hrs at 110˚C. After that it was ground to fine powder by using mortar [11].

Photocatalytic activity of synthesized CdS photocatalyst

The 50 mL of 10 mg/L MB solution was taken in a beaker and pH was maintained by using 0.1 M NaOH and 0.1 M HCl solution. The 10 mg of synthesized CdS photocatalyst was added to the beaker containing methylene blue solution. It was stirred for 10 minutes by using magnetic stirrer and kept in dark for 15 minutes to achieve the absorption desorption equilibrium. Then the solution was kept under the UV chamber. The UV lamp of 16 watt was used to study the photocatalytic activity of synthesized CdS photocatalyst for the degradation of MB dye. After certain interval of time, the solutions were centrifuged at 14000 rpm for 5 minutes and then absorbance of solutions measured by using spectrophotometer to calculate the degradation efficiency of CdS photocatalyst. Ultimately, the degradation percent was measured by using following formula,

$$\text{Degradation}\ (\%) = \frac{Co - C}{Co} \times 100 \ldots\ldots\ldots (1)$$

Where, Co = Initial Concentration, C = Final Concentration

Results and Discussion

Characterization

X-ray Diffraction (XRD): XRD patterns of CdS is shown in the **Fig. 1.** Well defined and sharp diffraction peaks are clearly seen.

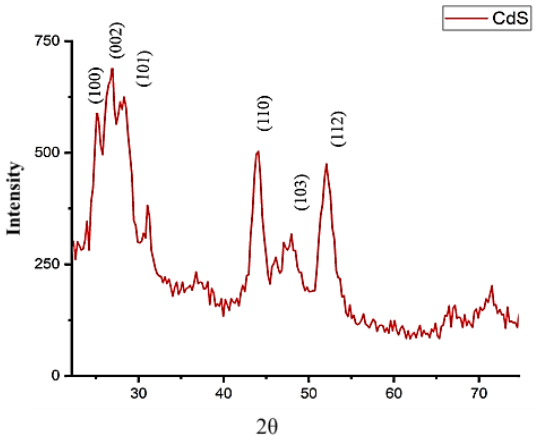

Fig. 1. XRD pattern of CdS nanoparticles.

In **Fig. 1,** peaks at 25.09(100), 26.9(002), 28.29(101), 36.75(102), 43.9(110), 47.9(103), 52.07(112) $2\theta^0$ are well distinct which corresponds to the hexagonal wurtzite structure of CdS according to JCPDS card number 10-0454. The sharp peaks are also an indication of crystalline nature of the material. These values are also in accordance with literature [11]. The crystallite size of CdS was calculated by using Debye Scherrer equation (2):

$$D = K\lambda / \beta \cos\theta \ldots\ldots\ldots\ldots\ldots (2)$$

Where, K (0.9) is the shape factor for spherical particles, λ (0.15406 nm) is the wavelength of incident radiation, β is the full width at half-maximum height, θ is Bragg's angle and D is the crystallite size. The average crystallite size was found to be 5.25 nm. Hence, synthesized CdS was found to be nano in size. Band gap energy of synthesized CdS was obtained by UV spectra (not shown) and was found to be 2.8 eV.

Fourier Transform Infrared Spectroscopy (FTIR)

The FTIR spectra of CdS nanoparticles was obtained at wave number of 4000-500 cm^{-1} (**Fig. 2).** The bands were clearly seen. The large broad band around 3359.87 cm^{-1} was observed which is assigned to the O-H stretching bond. It may be due to moisture absorbed on the surface of CdS material [12]. Similarly, a strong band at 1620.61 cm^{-1} was seen which is assigned for the bending vibration of H_2O molecules [13].

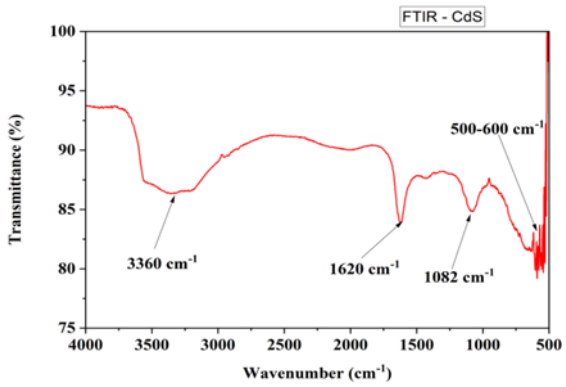

Fig. 2. FTIR spectrum of CdS nanoparticles.

In the same way, broad band around 1082 cm^{-1} was observed which represents the presence of S-O bond. Likewise, the strong band at around 600-500 cm^{-1} is attributed to the Cd-S stretching vibration which confirms the formation of CdS [14]. Similar bands are also reported in the literature [15].

Scanning Electron Microscopy (SEM)

The SEM was used to analyze the surface morphology of the synthesized CdS nanoparticles. In **Fig. 3,** one can see white smooth solid particles.

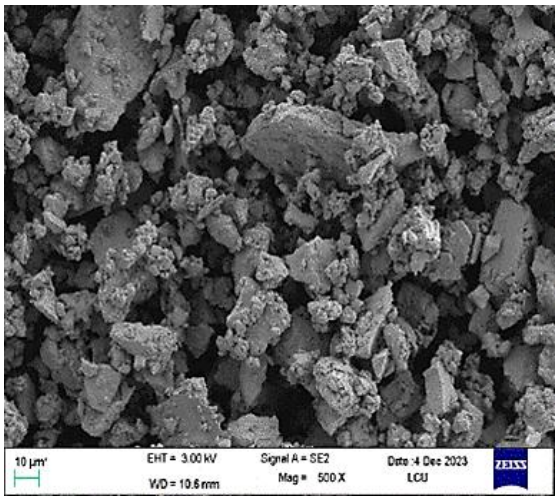

Fig. 3. SEM image of CdS.

Some of them are found to be smooth and irregular shape, whereas some are found to be in agglomerated form. Such type of material is also reported in the literature [16]. Further, **Fig. 3** also proves that as prepared CdS particles are nano in size.

Zeta potential Analysis

The zeta potential of colloidal dispersion of CdS nanoparticles was measured and result is presented in **Fig. 4.**

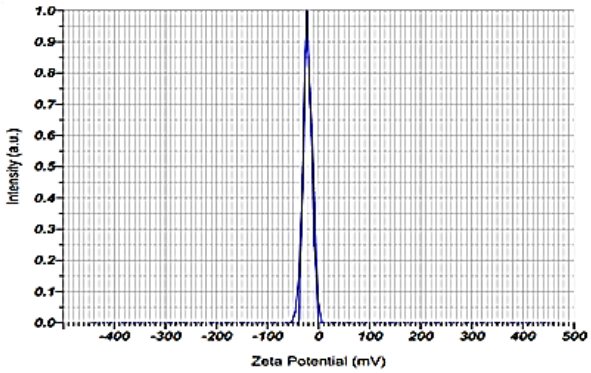

Fig. 4. Zeta Potential of colloidal dispersion of CdS nanoparticles.

As can be seen in **Fig. 4**, mean zeta potential of CdS dispersion was found to be –21.9 mV. Similar value of zeta potential for bare CdS was reported in literature [17]. This small negative zeta potential value is an indication of comparatively less stability of CdS in colloidal

dispersions as compared to the capped CdS as mentioned in literature [18]. Though, the magnitude of the zeta potential was found to be negative indicating negative charge on the CdS nanoparticle, however it was smaller than boundary value of ± 30mV, denoted the potential stability of CdS nanoparticles in the colloidal system. Here, it was predictable to a chance of agglomeration of CdS nanoparticles as the Vander Waals attractive forces may act upon them. If all the particles in suspension have a large negative or large positive zeta potential, then they will tend to repel each other and there will be no tendency for the particles to come together. Hence, it was concluded that there is a need of capping agent in CdS nanoparticles which helps to prevent from agglomeration during photocatalytical activities. Capping agent may also help to repel each other in colloidal dispersion. The zeta potential can also be influenced by the dielectric constant of a particle and the ionic strength of the surrounding media. Therefore, when measuring the zeta potential of nanoparticles, the choice of the dispersing media and pH of the media should be carefully considered whenever possible.

Applications

Photocatalytic degradation of MB

Photocatalytic activity of CdS nanoparticles were then investigated by measuring the degradation of MB dye under the irradiation of UV light. Photocatalytic degradation of methylene blue was carried out by varying the various parameters such as pH, time of UV irradiation, concentration of methylene blue, dose of photocatalyst.

Effect of pH on Photocatalytic degradation of methylene blue

During the starting phase of experiment, 50 mL of (10 mg/L) MB solution was taken. The pH was varied from 3-9 in different batch solution of (10 mg/L) MB and 10 mg of prepared CdS was used as photocatalyst. All the solutions were irradiated by UV light for 2 hrs. Then the degradation percent was calculated by using equation (1) and results are presented in graph (**Fig. 5).**

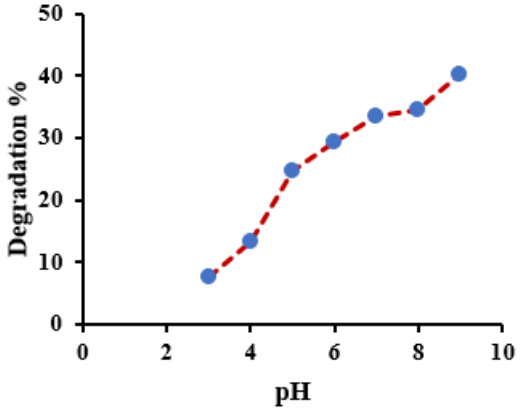

Fig. 5. Effect of different pH on photocatalytic degradation of MB dye.

Fig. 5 shows the degradation efficiency of CdS nanoparticle under UV light at different pH. As can be seen in the curve, degradation % was found to be 7% at pH 3 which was increasing slightly at pH 4. When pH varied up to 9, degradation % was found to be increasing continuously. At pH 9 it was found to be 40%. After pH 9, degradation percent was found to be in increasing trend. However, after pH 9 data are fluctuating it may be due to high concentration of OH^-. The high concentration of OH^- may attract cationic dye rather than degradation. So, basic pH 9 was picked for further work which was also reported in literature [19].

Effect of CdS catalyst dose on Photocatalytic degradation of MB

For this experiment, pH 9 was maintained, concentration of MB 10 mg/L and volume of MB 50 mL was taken for each test. UV irradiation time was set to 2 hrs. Catalyst dose was varied from 10 mg to 200 mg.

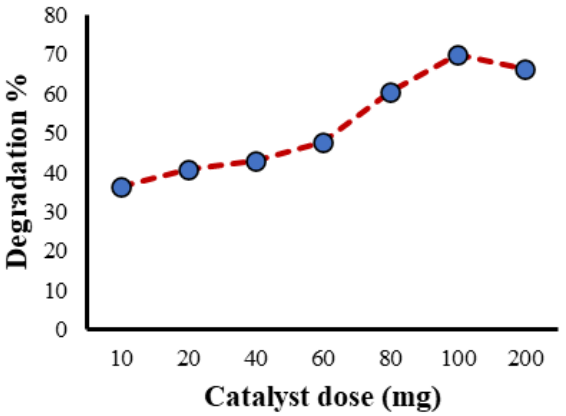

Fig. 6. Photocatalytic degradation of MB at different CdS dose.

Fig. 6 shows the degradation percent at different catalyst dose. When the catalyst dose was 10 mg, degradation of MB was found to be 36%. Degradation percent was found to be increased to 40% when 20 mg of catalyst was used. When catalyst dose was further increased up to 100 mg, degradation percent increased continuously and found to be 70% degradation. The increase in degradation percent may be due to the increase in number of electron-hole pair formation. The 100 mg of catalyst dose showed the highest degradation of dye. After that when catalyst dose was increased up to 200 mg, degradation percent seems to be in decreasing trend. It may be due to turbidity in the solution system. Such turbidity reduces the light penetration in the bottom of the solution which leads to the low rate of the reaction. Beside this, there might also be possibility of an agglomeration of the catalyst nanoparticles at high dose which reduces the effectiveness of the catalyst. So, 100 mg catalyst is said to optimum dose and was chosen for further experiment.

Effect of concentration of MB on photocatalytic degradation of MB

The pH 9 and catalyst dose 100 mg were chosen for this experiment. To study the effect of MB dye concentration, series of MB solutions 10 mg/L, 15 mg/L, 20 mg/L and 25 mg/L were used. The degradation percent were studied after 2 hrs of UV irradiation (**Fig. 7).**

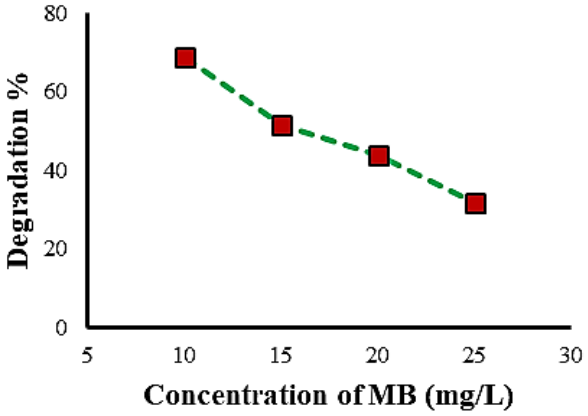

Fig. 7. Photocatalytic degradation of MB at different concentration of MB.

As can be seen in **Fig. 7,** the maximum degradation was perceived in 10 mg/L MB solution and minimum degradation was found in 25 mg/L MB solution.

In 10 mg/L MB solution, 69% degradation was observed. In 15 mg/L 52% degradation was found. Similarly, 44% and 32% degradation were obtained in 20 mg/L and 25 mg/L respectively. The results revealed that degradation percent decreases with increases in concentration of MB solution. It may be due to the aggregation of dye molecules on catalyst surface which can quench the excited molecules. Also, may be due to fully occupied active site of catalyst by high concentration of dye molecules. Though in 5 mg MB solution nearly 70 % degradation was observed, here 10 mg/L MB solution was taken for further experiment.

Here, with 100 mg of catalyst only 69% degradation was found within 2 hrs. of UV irradiation. For the further improvement, UV irradiation time was varied keeping other parameters constant.

Effect of UV irradiation time on photocatalytic degradation of MB

For this experiment, pH 9 and catalyst dose

100 mg and MB concentration 10 mg/L were taken. Irradiation time was varied from 30 to 210 minutes. The results are presented in **Fig. 8.** As can be seen in curve **(Fig. 8),** at 30 min of UV irradiation, degradation percent of MB dye solution was found to be 50%. When UV irradiation time was increased continuously up to 210 min, the degradation percent increased continuously. The 58%, 68%, 73%, 77%, 80% and 82% degradation were found at 60, 90, 120, 150, 180 and 210 minutes of UV irradiation time respectively. From this experiment, a significant, 82% degradation was perceived within 210 minutes of the UV irradiation.

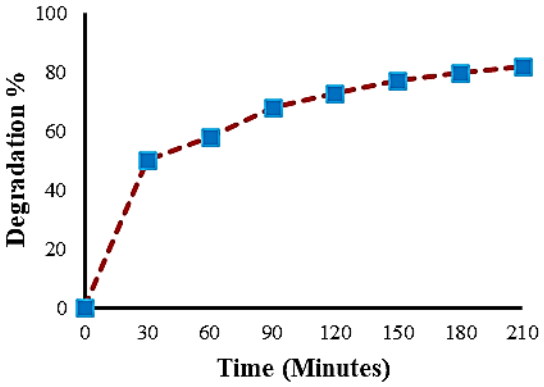

Fig. 8. Photocatalytic degradation of MB with different time.

Though a significant, 82% degradation was found within 210 min of UV irradiation time, we tried to increase the degradation percent. So, to achieve more degradation within same time of UV irradiation time, next experiment was carried out by adding oxidant H_2O_2 in reaction system.

Effect of oxidant (H_2O_2) on photocatalytic degradation of MB

For this experiment, pH 9, catalyst dose 100 mg and concentration of methylene blue 10 mg/L were used. Here 1 mL of 1% H_2O_2 was used. Then degradation percent was noted at 60, 90, 120, 150, 180 and 210 minutes of UV irradiation. The results achieved are presented in **Fig. 9.**

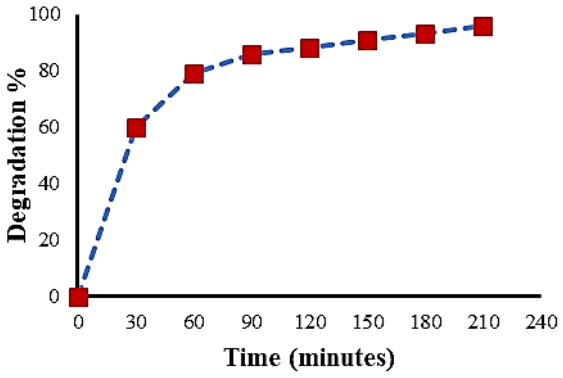

Fig. 9. Photocatalytic degradation of MB in presence of oxidant H_2O_2.

As can be seen in **Fig. 9** degradation percent was found to be increased significantly. The 60% degradation was found within 30 minutes of UV irradiation. The 79%, 86%, 88%, 91%, 93% and 96% degradation were found within 60, 90, 120, 150, 180 and 210 minutes of UV irradiation time respectively. From this experiment, maximum 96% degradation was displayed within the 210 minutes of UV irradiation. Such increase in degradation percent may be due to presence of H_2O_2. The H_2O_2 may help to generate large number of hydroxyl radicals such as $OH^{\bullet}$, $^{\bullet}OOH$, OH^{-} in presence of UV light. As a result, degradation and decolorization of MB increases.

Photocatalytic degradation of MB at different conditions

Comparative study of Photocatalytic degradation of 10 mg/L MB at pH 9, with 100 mg catalyst dose at different interval of UV irradiation time was carried for various conditions. Following four experiments were carried for comparative study.

(1) Degradation of MB solution under UV

(2) Degradation of MB solution with catalyst under dark

(3) Degradation of MB solution with only catalyst under UV

(4) Degradation of MB solution with catalyst and 1% of 1 mL H_2O_2 under UV light

The results are presented in **Fig. 10**.

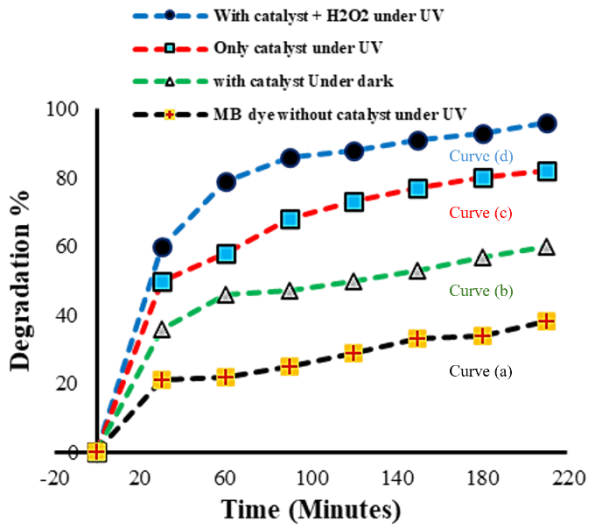

Fig. 10. Photocatalytic degradation of MB at different conditions.

Fig. 10 shows degradation curves at four different conditions. In **Fig. 10,** curve (**a**) shows the degradation of MB solution without catalyst kept under UV irradiation. The low degradation percent was observed. At 210 min of UV irradiation, only 38% degradation was reached. The curve in **Fig. 10** (**b**) shows the degradation of MB solution with catalyst under dark (no UV light was passed). The slow increase in degradation percent was observed and it reached to 60% after keeping 210 min under dark. Similarly, curve in **Fig. 10** (**c**) shows the degradation of MB solution with catalyst under UV. In this condition significant increase in degradation can be seen. The 82% degradation was observed within the 210 min of UV irradiation. Likewise, curve in **Fig. 10 (d)** represents the degradation of MB solution with catalyst and 1% of 1 mL H_2O_2 under UV light. In this curve, one can see high degradation, 96% within 210 min of UV irradiation was achieved.

Reaction rate and order of reaction

The further investigation on reaction rate was carried out using Langmuir Hinshelwood kinetic model $\ln (C_0/C) = kt$ ..... (3)

where, K is rate constant (min^{-1}), C_0 is initial concentration of MB dye (mg/L) and C is the actual concentration of MB after irradiation of UV light at time t. The calibration curve was obtained at 660 nm of wavelength for the determination of MB which is shown in **Fig. 11**.

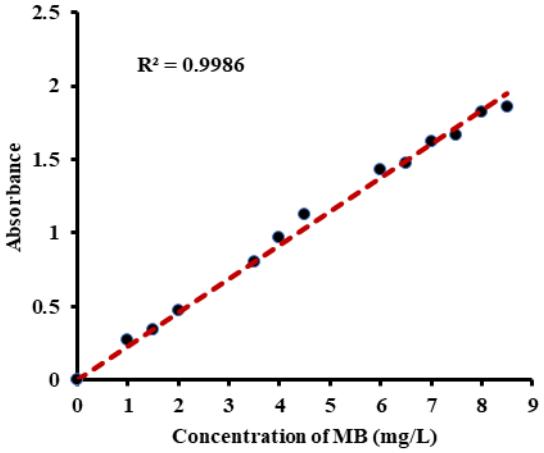

Fig. 11. Calibration curve for the determination of MB.

Here, following two conditions has been applied: (a) 50 mL of 10 mg/L MB solution at pH 9 without catalyst under UV light (b) 50 mL of 10 mg/L MB solution at pH 9 with 100 mg catalyst and with H_2O_2 under UV light. The results are presented in a graph (**Fig. 12**) where $\ln C_O/C$ was plotted against irradiation time in minute.

Fig. 12 shows the pseudo first-order kinetics for MB degradation at two different conditions. Curve **(a)** in **Fig. 12** shows the kinetics of MB without catalyst under UV light. The slope of the curve exposed the rate constant (k) which was found to be 0.0025 min^{-1} (R^2 = 0.9624). It is an indication of slow rate of degradation of MB. The curve **(b)** in **Fig. 12** shows the kinetics of MB in presence of H_2O_2 along with catalyst. The slope of the curve showed high-rate constant (k) 0.016 min^{-1} (R^2 = 0.9257) in comparison to curve **(a)** in **Fig. 12**. This greater value indicates the high rate of

degradation of MB dye in presence of oxidant H_2O_2 along with catalyst.

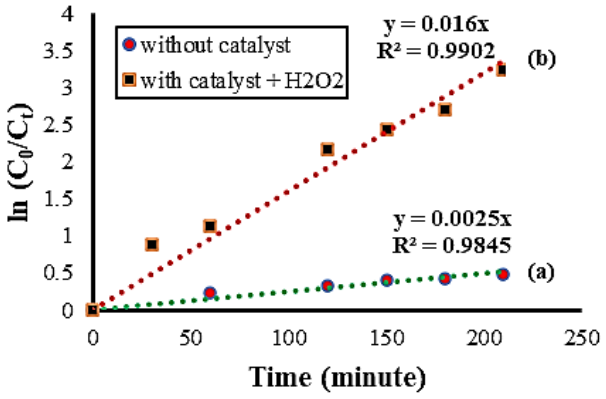

Fig. 12. ln C_O/C as a function of irradiation time.

Conclusions

In this research work, cadmium sulfide nanoparticles have been synthesized by using chemical precipitation method. Then it was characterized by XRD, FTIR and SEM analysis. Band gap energy of synthesized CdS was found to be 2.8 eV. The average crystalline size was found to be 5.25 nm from XRD. It clearly indicated that as prepared CdS is nano in size. In FTIR spectra, the Cd-S stretching vibration band was found at 600-500 cm^{-1} which confirming the formation of CdS. The SEM image showed the smooth and irregular shaped CdS nanoparticles. Some are found in agglomerated form which may be due to nano size of CdS. The 82 % MB dye degradation was found in 10 mg/L MB solution within 210 min of UV irradiation time. Maximum degradation 96% was achieved when 1% of 1 mL oxidant H_2O_2 was added along with 100 mg of catalyst which was significantly higher than previous works. Hence, it can be concluded that the CdS can be effectively used as photocatalyst to degrade cationic dyes like MB.

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Author's Contribution Statement

Kalpana Dhungana: Conceptualization, Writing: original draft, Methodology, **Armila Rajbhandari (Nyachhyon)**: Writing: review and editing, Investigation, Formal analysis, Data analysis, Visualization, Validation, Resources, Supervision.

Conflict of Interest

The authors do not have any conflict of interest throughout this research work.

Data Availability Statement

The data supporting this study's findings are available from the corresponding authors upon reasonable request.

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