Facile Solvothermal Synthesis Of Visible Light Active CDS Nanoparticles For The Photodegradation Of Rose Bengal (RB) Dye

Section A-Research paper

FACILE SOLVOTHERMAL SYNTHESIS OF VISIBLE LIGHT ACTIVE CDS NANOPARTICLES FOR THE PHOTODEGRADATION OF ROSE BENGAL (RB) DYE

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ABSTRACT

In this study, CdS nanophotocatalyst was prepared by low temperature solvothermal method for photodegradation of Rose Bengal (RB) dye in aqueous solutions in the presence of visible light photon. Further, synthesized CdS nanoparticles were characterized by various techniques such as Fourier Transform Infra-Red (FT-IR) Spectroscopy, X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM), and UV-Vis spectroscopy. The obtained CdSNPsshown hexagonal wurtzite structure with decent crystallinity, good optical features and exhibited admirable photocatalytic efficiency (98.50%) towards the photodegradation of Rose Bengal (RB) dye.Kinetic parameters for the degradation of RBwith the prepared CdS nanoparticleswere also reported and data well fitted to thepseudo-first-order kinetic model.The stability of CdS photocatalyst was remained constant and efficiency not decreased much more over the use of fivecycles.

Keywords: -CdS, Nanophotocatalysts, Rose Bengal, Photodegradation, Kinetic parameters.

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1. INTRODUCTION

In recent years, extensively increased industrialization due to fast growing rate of population and urbanization from which highly toxic and hazardous pollutants such as pesticides, herbicides, nitrophenols, and organic dyes are discharged into water resources. The elimination of these water contaminants is a difficult task [1].Synthetic textile dyes with other industrial dye waste are considered one of the major water contaminantson the planet because of their offensive, poisonous, mutagenic and stable nature [2-4]. In the present scenario several techniques are utilizing to remove water contaminants, such as flocculation,coagulation, adsorption, and filtration, biological, but they are not much efficient, ecofriendly and economical [5,6] and the pollutantsare not completely degraded in nonhazardous products, but transform into different forms, and at the same time produce a large amount of secondary toxic products as well[7].

The photo-degradation has been considered one of the efficientmethod for water treatment in compare to the traditional physicochemical and biological purificationtechniques which are insufficient for effective removal of pollutants [2, 8-12]. In recent years, photo-degradation of pollutants using semiconductors has been proven an effective process for the degradation of water pollutants especially organic dyes such as Rhodamine B [13,14] Brilliant cresyl blue,[15,16] Malachite green [17]. Several semiconductor nanomaterials including CdS nanoparticles have been used for effective heterogeneous photocatalytic degradation of organic dye pollutants in water [12, 18-20].

Last two decades, Nanoscience and nanotechnology have been gained much importance across the globe and thenanomaterials are using in various field for multiple applications such as biomedicine, devices, energy, solar cells, biotechnology, water treatment, delivering vaccines and drugs [21], treating communicableailments, cancer detection, construction of scaffolds and in bioremediation.

Currently utilization of the II-VI group metal sulphide semiconductor nanoparticlesis gained much attention as an important material for the photocatalysis for the proficient degradation of toxic pollutants including organic dyes, antibiotics, pesticides etc. under visible light illumination and to resolve the numerous aqueous environmental pollution problems [22-25].Among various metal sulfides II-VI semiconductor nanoparticles, a great attention has been showered on the cadmium sulphide (CdS) nanoparticles because of the admirable characteristics such as availability of discrete energy levels, tunable band gap, size dependent optical properties, well-developed synthetic protocols, easy preparation technique with good chemical stabilityand exciting photosensitivity [26]. CdS is a visible-light-driven photocatalyst that has an energy band gap of 2.4 eV[27].It is extensivelyexplored in various fields such as in detection ofphotochemical catalysis, visible radiations, light emitting diodes, solar cells, gas sensors, optoelectronic devices, luminescence devices, and environmental sensors [28-31]. Moreover, narrow band gap and negative edge potential of the conduction band of CdShas turned it into excellent semiconductor material for degradationof organic pollutants and H_2generationusing solar light [32,33].Narrow band gap CdS allow to absorb solar-light photon effectively whereas higher surface area permits higerlight photon absorption on the surfaces of CdS.So far, CdS nanoparticles numerous morphologieshave been prepared using diversemethods such as chemical vapor deposition process [29], colloidal method [34], hydrothermal method [35], template method [36], sol gel templatemethod [37], thermal decomposition method [38], solvothermalmethod [39], reversed micelle method[40], thermal evaporation method [41] and co-precipitation method [1].
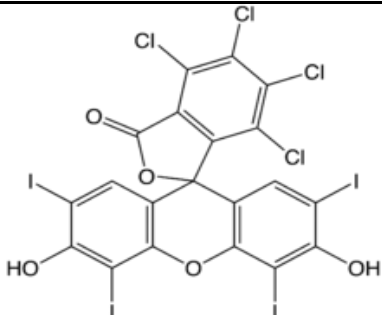
In the present work, CdS nanoparticles were synthesized by low temperature solvothermal method using 2-naphthylthiourea as source of (Sulphide) S^{2-} ions in basic medium. The synthesized CdS nanoparticles were exploited as nanophotocatalystfor the photodegradation of Rose Bengal (RB) under visible light illumination which demonstrated exiting performance and stability.

2. MATERIALS AND METHOD
2.1. Materials used

Cadmium acetate, 2-naphthylthiourea, sodium hydroxide, hydrochloric acid, rose bengal (RB) and ethyl alcohol were used in preparation and experimental work in current study.For photocatalytic degradation, rose bengal was selected as a typical water contaminant in the present work. Rose bengal isfrequently used in eye drops to stain injured conjunctival and corneal cells. The chemicalformula and some properties of the RB dye are illustrated in Table 1.

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Table 1 The chemical structure and physicochemical properties of RB dye.

Structure	λ_{max} (nm)	MW (g/mol)	Molecular formula	Solubility (g/L)
	500-650	973.67	$C_{20}H_4Cl_4I_4O_5$	100

2.2. Synthesis of CdS Nanoparticles

CdS nanoparticles were synthesized by simple and facile low temperature solvothermal method in alcoholic solution. In a typical process, 50 mL0.1 M cadmium acetate and50 mL of 0.1 M 2-naphthylthiourea (2-NTU) were prepared separately in ethyl alcohol and stirrer for 30 min. After that 0.1 M 2-naphthylthiourea solution was mixed with cadmium acetate solution drop wise at constant stirring and pH of mixture was raised to 9 by addition of sodium hydroxide (1M) solution, yellow precipitate is appeared in mother liquor. The mother liquor along with precipitate was stirred for 2 h at 60^0C and cool down to room temperature naturally. Then, the as-obtained suspension was centrifuged and washed several times with distilled water and finally with alcohol and dried at 70^0C for 5 hin a hot air oven. The schematic synthesis protocol for theCdS nanoparticles is shown in Figure1.
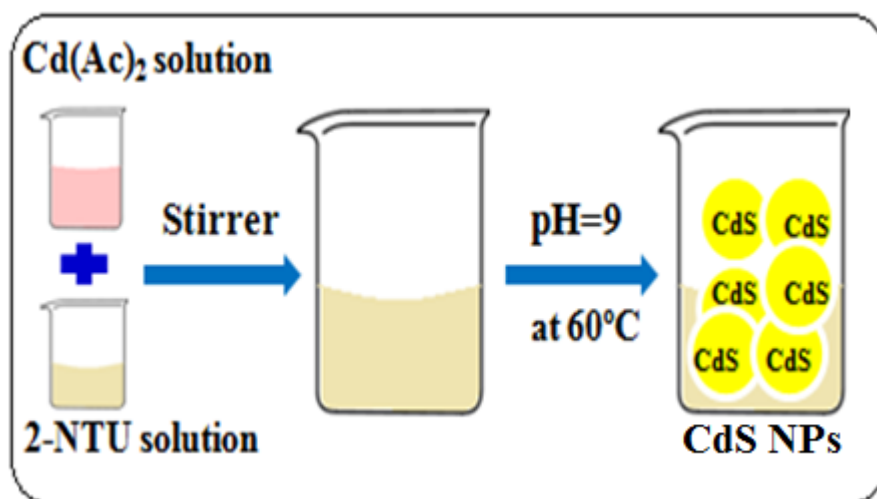
Figure 1 Flow chart of synthesis of CdS nanoparticles.

2.3. Characterization

The morphological and structural analysisof the synthesized sample was carried out by Fourier Transform Infrared Spectroscopy (FTIR) Spectroscopy, Scanning Electron Microscopy (SEM) and X-ray diffraction (XRD). The optical properties were determined by using UV-Visible Spectroscopy.

2.4. Photocatalytic experiment

The photocatalytic activity of the synthesized CdS nanoparticles was studied by performing photodegradation experiments for Rose Bengal (RB) dye under visiblelight irradiation. The optimization of reaction conditions was achieved by varying the catalyst dose amount, initial dye concentration, pH value of solution and reaction time. In a typical photocatalytic experiment, 50mL of the dye solution of desired concentration containing the appropriate quantity of the catalyst at optimized pH was magnetically stirred in dark for 30 min to attain the adsorption–desorption equilibrium between dye and catalyst surface. 5mL of the dye solution was withdrawn prior starting the irradiation. Aliquots (5 mL) were taken out from the dye solution at suitable time intervals during the light irradiation and centrifuged to remove catalyst, and the absorbance

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intensity of RB dye solution was measured on a UV-spectrophotometer. To evaluate the auto-degradation of dye, standard experiment without using nano-catalyst was also performedusing same reaction conditions. The percentagedegradation of RB was estimated using equation (1) as given follows:
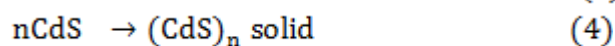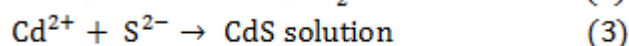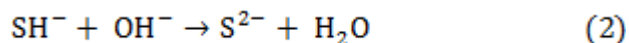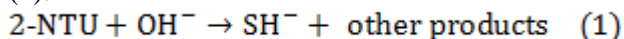
$$\text{Degrdation efficiency (\%)} = \frac{C_0 - C_t}{C_0} \times 100 = \frac{A_0 - A_t}{A_0} \times 100 \quad (1)$$

Where C_0 and C_t are the initial concentration and concentration at time (t), while A_0 the absorbance of dye solutions at t=0while$A_t$is absorbance of dye solutionsat t=t.

3. RESULTS AND DISCUSSION
3.1. Synthesis of CdS Nanoparticles
The CdS nanoparticles were synthesized using low temperature solvothermal method in basic medium, in which Cd^{2+} and S^{2-} were supplied from cadmium acetate and 2-naphthylthiourea (2-NTU), respectively. In basic medium 2-naphthylthiourea produced SH^- ions which subsequently released S^{2-} that interacted with Cd^{2+} ionsand form yellow coloredCdS solution. The CdS particles agglomerated to generate CdS nanoparticles. The process of formation of CdS nanoparticles is represented in given reactions (1)-(4).

$$2\text{-NTU} + OH^- \rightarrow SH^- + \text{ other products} \quad (1)$$

$$SH^- + OH^- \rightarrow S^{2-} + H_2O \quad (2)$$

$$Cd^{2+} + S^{2-} \rightarrow CdS \text{ solution} \quad (3)$$

$$nCdS \rightarrow (CdS)_n \text{ solid} \quad (4)$$

3.2. Characterization
XRD pattern for synthesized CdS nanoparticle is shown in Figure 2. The diffraction peaks shown in XRD pattern at 2θ values of 25.26°, 26.5°, 28.26°, 38.85°, 43.79°, 48.02°, 52.08° and 70.79° can beindexed to the (100), (002), (101), (102), (110), (103), (112) and (211) crystal planes of hexagonal CdS, which clearly indicates that the as-prepared CdScontain high crystallinity, phase purity and have no any other impurity peaks.

The average crystalline size of the prepared nanoparticles is estimated from the Scherrer equation (2):

$$D = \frac{K\lambda}{\beta cos\theta} \quad (2)$$

where$D$ is the crystalline size,K is constant (0.9), λ is the wavelengthof the incident X-ray (0.15417 nm), θ is the diffraction angle and β is full width at halfmaximum (FWHM) of the highest intense (002) peak of the hexagonal phase of CdS. The evaluated average crystallite size is 18nmfor CdS nanoparticles.
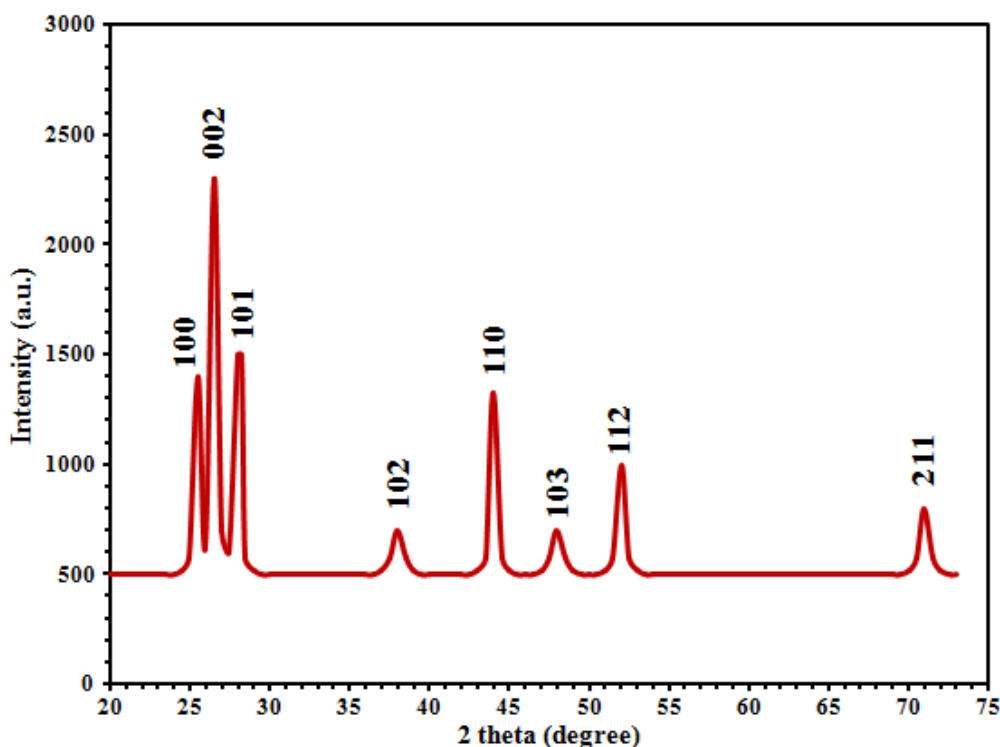

Figure 2 XRD pattern for CdS nanoparticles.

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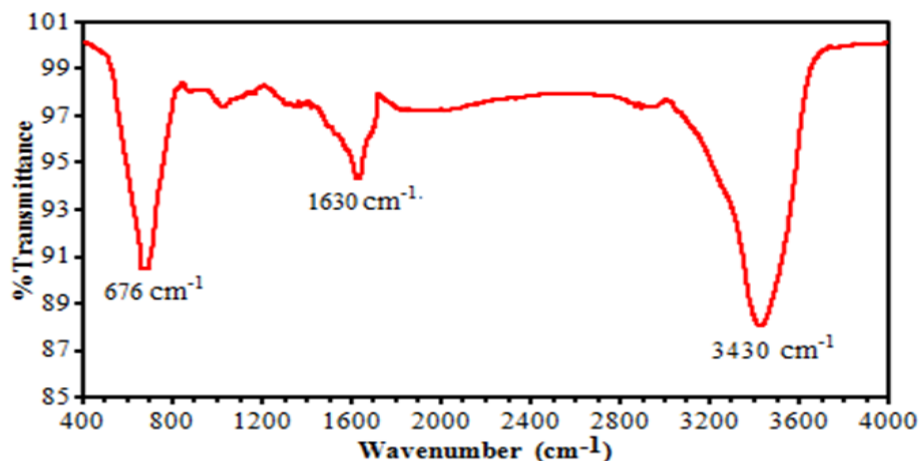
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Figure 3 FTIR spectra for CdS nanoparticles.

FTIR is utilized to study the bonding and functionalities in theprepared product. The FTIR spectrum of synthesized CdS nanoparticles is shown in Figure 3. The absorption peaks noticed in spectrum at 3430 cm^{-1} and 1630 cm^{-1} are attributed to the –OH bonding vibrations of H_2O molecules adsorbedon the surface of CdS NPs.

An intense peak at 400-700 cm^{-1} can be assigned to metal-sulphur (M-S) bond which confirms construction of CdS nanoparticles [42]. The peak observed at 676 cm^{-1} in FTIR spectrum is corresponding to the Cd-S bond [43, 44]. The SEM micrographs at various magnifications of synthesized sample are shown in Figure 4. The particles do not have definite shape, however seem like stone and possess rough surfaces. It might be due to more of a fine amorphous powder. The UV absorption spectrum of the synthesized CdS nanoparticles is revealed in Figure 5(a). The spectrum exhibited a distinct absorption peak at 576 nm, which shows blue-shift as compare to the absorption peak of bulk CdS indicating quantum size effect.

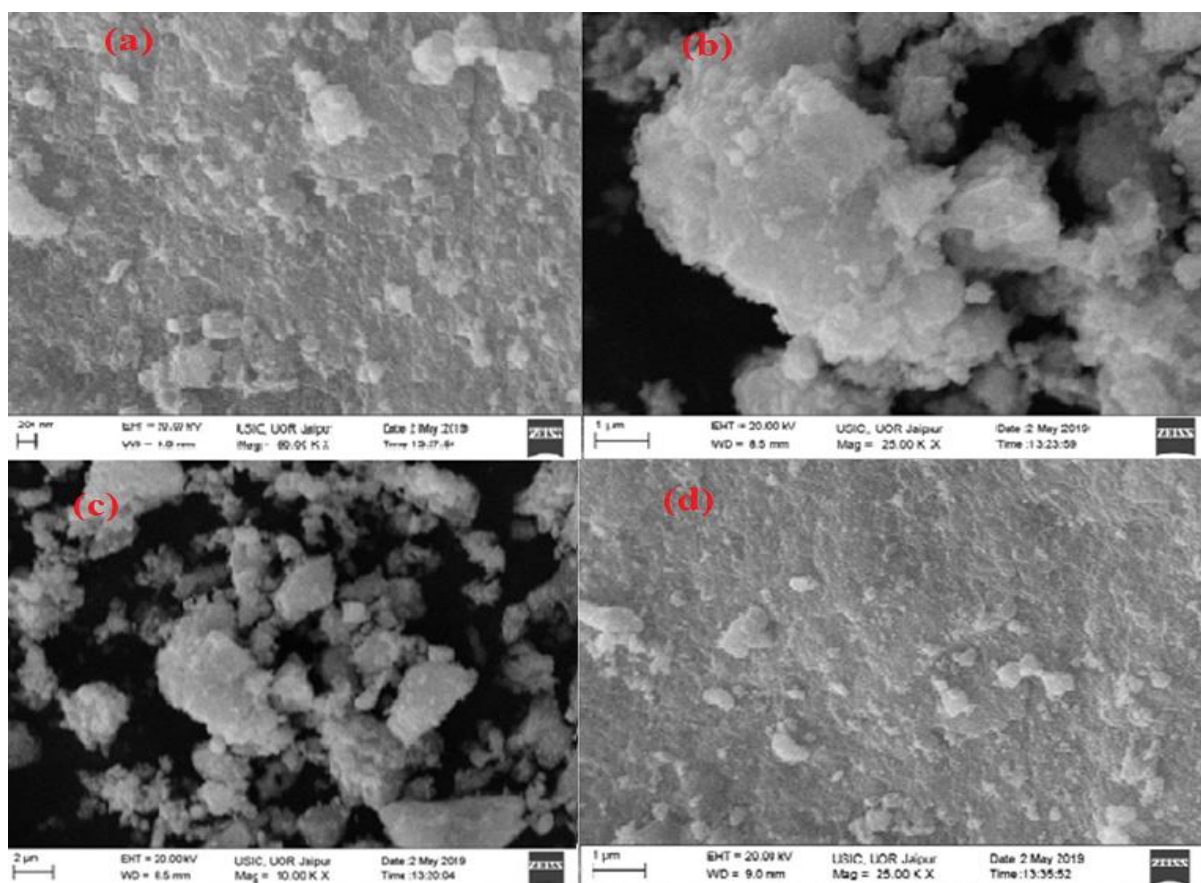**Figure 4** SEM images of the synthesized CdS nanoparticles.

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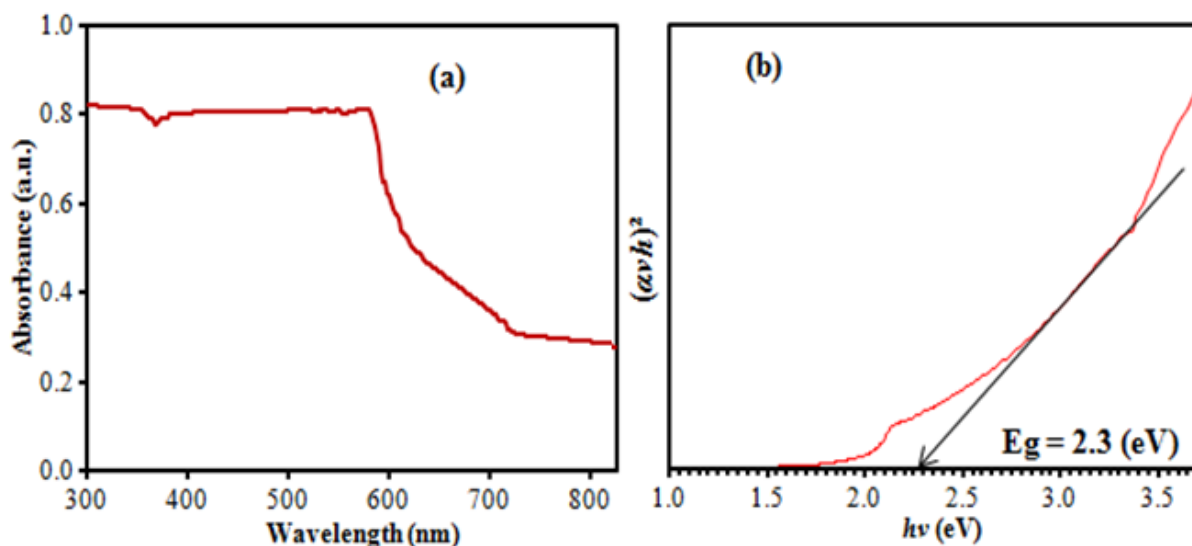
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Figure 5(a)UV absorption spectra, and (b) bandgap value for synthesized CdS nanoparticles.

3.3. Bandgap calculation

The bandgap energy (Eg) value can be evaluated from the UV–Visabsorption spectrum via a Tauc plot drawn between $(\alpha h\upsilon)^2$ and $(h\upsilon)$. The optical bandgap energy for the prepared CdS nanoparticles was calculated using the Debye-Scherer's relation as given by equation (3) [45].

$$(\alpha h\upsilon)^n = K\left(h\upsilon - E_g\right) \quad (3)$$

where K is a absorption coefficient, E_g is the bandgap of material, υ is the frequency of the incident radiation and *h* is the Planck's constant, and the exponent n is 2 for direct band allowed transitions. The optical band gap energy was estimated by extrapolating the straight-line portion of $(\alpha h\upsilon)^2$ *vs.hv* plotto *hv* axis at α=0 as shown in Figure 5(b). The obtained band gap for CdS nanoparticles is 2.3.eV.

3.4. Photocatalytic Degradation of RB Dye

The photodegradation performance of the synthesized CdS nanoparticles was investigated by performing photo-degradation experiments of Rose Bengal (RB) dye using visible light. The standard experiments were also performed by illuminating dye aqueous solution without using CdS NPs and using CdS NPs in the dark.

Investigation of the samples in both cases do not show any significant degradation of the dye. As shown in Figure 6(a) the absorption peak of RB dye at 553 nm is repeatedlyfalling with time due to photo-degradation of RB dye into non-hazardous products and the pink color of the dye solution completely changed to colorless after the 80 min of light irradiation and no absorption peak noticed, which indicated the complete photodegradation of RB dye by CdS nanoparticles.

The degradation efficiency of synthesized CdS catalyst for RB dye under visible light irradiation was determined to be 98.50% after 80 min of light irradiation. It is suggesting that CdS demonstrated good photocatalytic activity in this process. Figure 6(b) portrays the change in RB concentration (C_t/C_o) with time over CdS nanoparticles under illumination with visible light and for blank RB solution in the dark. It can be observed from Figure 6(b) that the RB solution in dark with a catalyst not degradedappreciably, it only adsorbed on the surface of catalyst. However, in absence of catalyst it is self-degraded slowly under visible light illumination. The enhanced photocatalytic performance of CdS nanoparticles may be due to the small size and large specific surface area.

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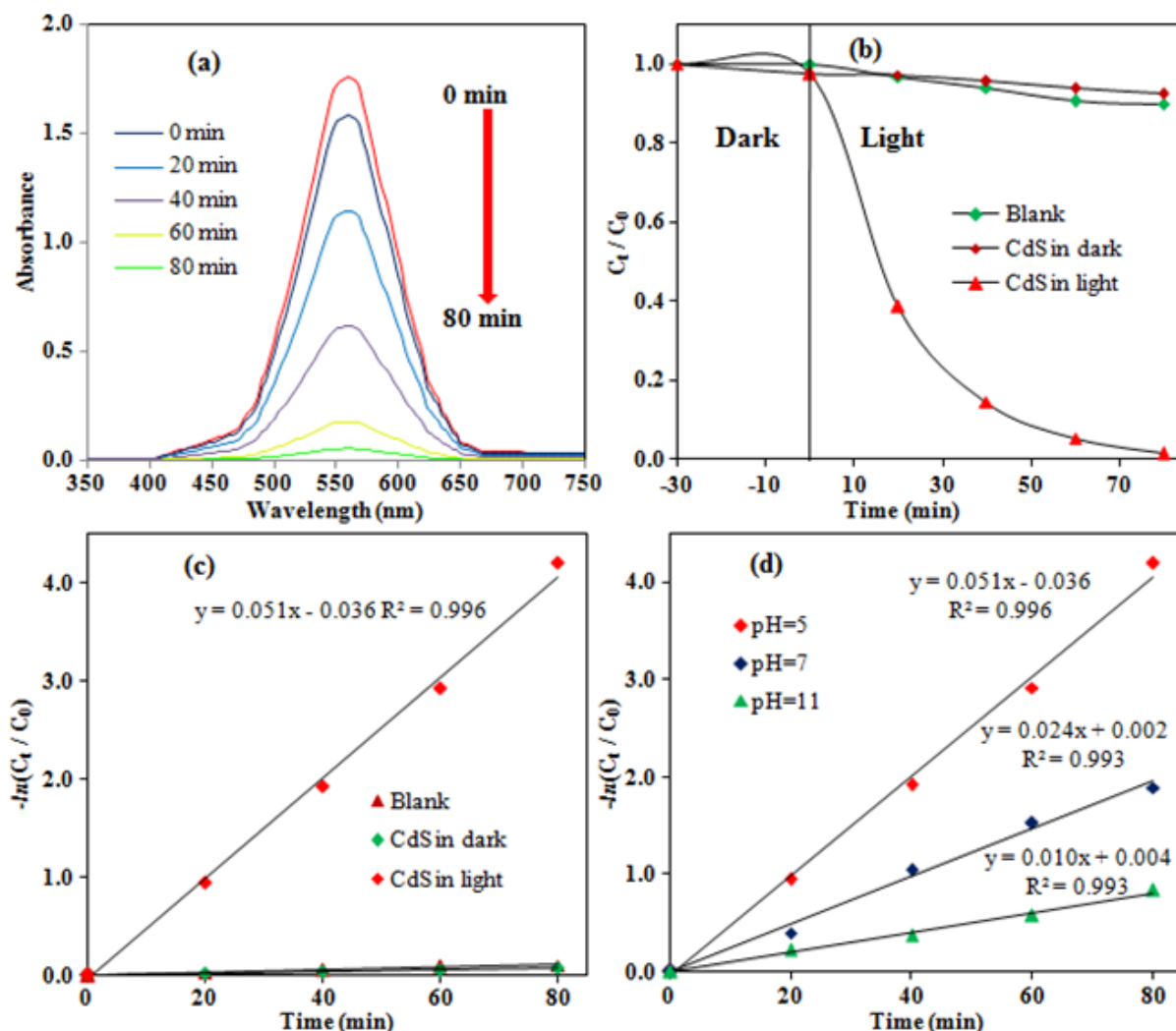
Figure 6 (a) Time-dependent UV-Vis spectrum of Rose Bengal (RB) dye solution, (b) The degradation of (RB) dye in the presence of CdS nanoparticles under visible light, (c) Pseudo first order kinetic plots for the photocatalytic degradation of RB dye at optimized reaction parameters,and (d) Pseudo first order kinetic plots showing the effect of pH of solution on the kinetics of RB photodegradation.

3.5. Kinetics of Photodegradation of RB Dye

The reaction kinetics of photodegradation of RBby with the synthesized CdS nanophotocatalyst under visible light illumination at optimized reaction parameters was investigated as displayed in Figure6(c).The degradation reactions of CdS nanoparticles with RB dye followedpseudo-first-order kinetics. The measured experimental data well fitted with the pseudo-first-order kinetic model as given below Eq (4):

$$ ln\left(\frac{A_o}{A_t}\right) = ln\left(\frac{C_o}{C_t}\right) = kt \quad (4) $$

where, k (min⁻¹) is thepseudo-first-order kinetic rate constant, A_o and A_t are the initial absorbance and absorbance at time t of the RB dye, and C_o and C_t are dye concentrations at t=0 and t=t time, respectively. The rate constants k for the pseudo first-order reaction isdetermined form the slope the graph drawn $-ln$ (C_t/C_o) as a function of the irradiation time (t). The plot for pseudo first-order photodegradation reaction of RB dye is shown in Figure 6(c)withcorresponding slopes of the fitting lines. The rate constant obtained for photodegradation reaction of RB in the presence of catalyst (0.0512 min⁻¹) is very much greater than the self-degradation reaction in absence of catalyst (0.00142 min⁻¹) under the light illumination. The determined values forthe rate constant (k) and correlation coefficient (R2) are tabulated in Table 2.

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Table 2 Reaction kinetics parameters for RB photodegradation by CdS photocatalyst

| Reaction condition	Rate constant (min^{-1})	Correlation coefficient (R2)
In light without catalyst	0.00142	0.977
In dark with catalyst	0.00084	0.916
In light with catalyst	0.0512	0.996

Table 3 Effect of pH on reaction kinetics parameters for RB photodegradation by CdS photocatalyst

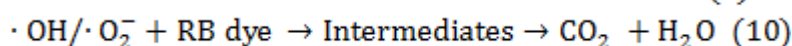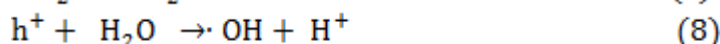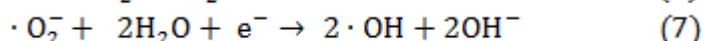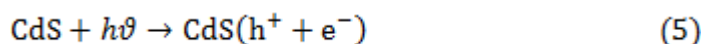| pH value	Rate constant (min^{-1})	Correlation coefficient (R2)
5	0.0512	0.996
7	0.0244	0.993
11	0.01007	0.993

In order to investigate the effect of solution pH on kinetics of RB photodegradation by the CdS photocatalyst, the photocatalysis reactions were carried out in the pH range of 5 to 11 keeping constant the dose amount of catalyst and dye concentration at room temperature. The influence of pH on kinetics of photodegradation reaction is illustrated in Figure 6(d) and the results are tabulated in Table 3. It can be observed from the results that rate constant values are decreasing with the increase of solution pH, the highest value of rate constant (0.0512 min^{-1}) was obtained for pH=5 while the lowest for pH=11(0.01007 min^{-1}). The higher value of rate constant at 5 pH was reported due to the enhanced adsorption of anionic RB dye on the surface of CdS photocatalyst due to positive charge on surface at this pH range [46]. On the contrary, in the basic medium (pH 11), surface of CdS photocatalyst carries negative charge that repels the anionic dye due to this rate constant value determined lower.

3.6. Photodegradation Mechanism

The activation of CdS nanocatalystby light photon (*hυ*) absorption produces electron and holes at the valance band (VB) of catalyst, which are powerful oxidizing and reducing agents, respectively.The photogenerated holes in stay at the VB while the electrons jump at the conduction band(CB) from where they transfer to the surface of catalyst, and trap by dissolved oxygen molecules produce hydroxyl free radicals (·OH), while the holes stay at VB also transfer to the surface of catalyst and inter with the water molecules to generate superoxide radical anions($\cdot O_2^-$). These produced highly active free radical species interact with dye molecules and degrade into intermediate products, which finally decompose intonon-hazardousinorganic CO_2 and H_2O molecules. The proposed possible photodegradation mechanism for the RB dye in presence of CdS catalyst is schematically represented in Figure 7 and explained by given in Eqs (5)-(10).
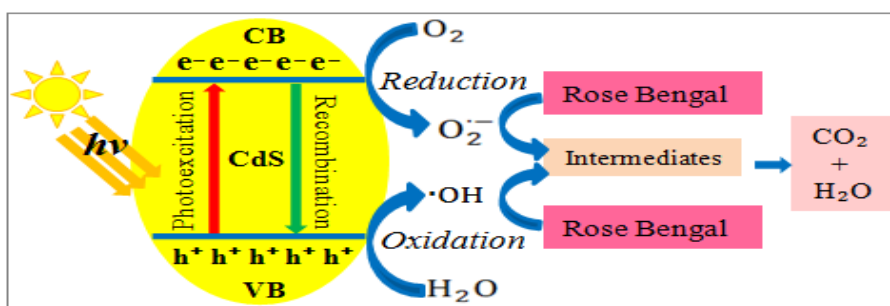
Figure 7 Schematic representation of the possiblemechanism involved in photocatalysis.

$$CdS + h\vartheta \rightarrow CdS(h^+ + e^-) \qquad (5)$$
$$e^- + O_2 \rightarrow \cdot O_2^- \qquad (6)$$
$$\cdot O_2^- + 2H_2O + e^- \rightarrow 2 \cdot OH + 2OH^- \qquad (7)$$
$$h^+ + H_2O \rightarrow \cdot OH + H^+ \qquad (8)$$
$$h^+ + OH^- \rightarrow \cdot OH \qquad (9)$$
$$\cdot OH/\cdot O_2^- + RB\ dye \rightarrow Intermediates \rightarrow CO_2 + H_2O \qquad (10)$$

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3.7. Reusability of Catalyst

The stability is a very important characteristic besides photocatalytic activity of any photocatalyst for the practical applications. The stability of the synthesized CdS photocatalyst was also investigated. The photocatalytic degradation experiment for RB dye was repeated applying similar procedures as mentioned in experimental section except catalyst for five cyclic runs. After each cycle of the photodegradation study the photocatalyst was recovered by centrifugation, washed with ethanol and water several times and dried in an oven, then reused for the next cycle. The photodegradation efficiency of CdS nanoparticles in five cyclicruns was determined to be 98.50%, 95.54%, 92.07%, 88.50% and 85.05% for first, second, third, fourth and fifth cycles, respectively as shown in Figure 8. It can be seen from the results that the photocatalytic activity of the CdS nanoparticles is not significantly decreased and activity remained almost constant overfive cycles of RB degradation.

The reduction in the competence of the CdS NPs might be due to the adsorption of dye molecules on the active locations of NPs [47]. Moreover, the assembling of the NPs when they are separated from solution possibly one more reason for the decline of the performance of the photocatalysts [48].
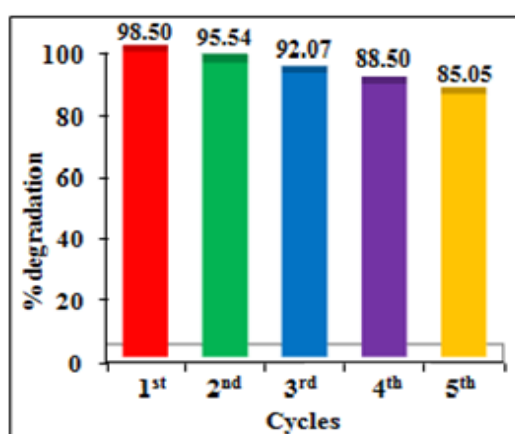
Figure 8 Bar chart showing reusability of the CdS photocatalyst for the photodegradation of RB for five cycles under white light irradiation.

The catalytic performance of the obtained CdS NPs was compared with various formerly developed catalysts which have been used for decolorization of different dyes and demonstrated in Table 4. From the comparative analysis it can be clearly indicated that CdS NPs prepared in the current study has shown good photo-degradation performance for the degradation of RB dye with visible light photons.

Table 4. Comparison study of catalytic performance of CdS NPs with other formerly developed catalysts

Catalysts	Light source	Time (min)	% Degradation	Organic Dye	References
ZnO/Ag$_2$O NPs	UV	40	96%	Methylene blue (MB)	[49]
Gd-doped MnFe$_2$O$_4$	Visible	70	96.35%	Methylene blue (MB)	[50]
p-Bi$_2$O$_3$/n-ZnO	Visible	60	93%	alizarin red (AR)	[51]
CdS	Visible	80	99%	Congo red (CR)	[52]
CdS	Visible	240	100%	Reactive red 141 (RR141)	[52]
CdS	Visible	240	95%	Reactive red 141 (RR141)	[53]
TiO$_2$	UV	90	90%	Reactive red 141 (RR141)	[54]
CdS	Visible	80	98.50%	Rose bengal (RB)	**This work**

4. CONCLUSIONS

Cadmium sulphide nanoparticles were synthesized via a simple and facilelow temperature solvothermal method, using 2-naphthylthiourea as sulphide ion sources. The structural, morphological and optical characteristics of the synthesized CdS nanoparticles were characterized by Fourier Transform Infra-Red (FT-IR) Spectroscopy, X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM), and UV-

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vis spectroscopy. The prepared CdSphotocatalyst demonstrated hexagonal structure with high crystallinity, excellent optical properties and exhibited admirable photocatalytic efficiency toward photodegradation of RB. Kinetic parameters for the degradation of RBin presence of CdS nanoparticle were reported.The experimental data well fitted with the pseudo-first-order kinetic model which confirmed that the photodegradation of RB dye followed pseudo first order kinetics. The stability of CdS photocatalyst was remained constant and efficiency not decreased much more over the use of five cyclic run of reuse. The dye degradation under optimum conditions on 80 minof visible lightirradiation was determined to be 98.50% and the photodegradation efficiency of CdS nanoparticles after fourth run of reuse was determined 85.05% which demonstrated good stability and reusability of prepared CdS nanoparticles.

Acknowledgements

Conflicts of Interest

Author proclaims no conflicts of interest.

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Graphical Abstract
➢ CdS NPs prepared using facile solvothermal method.
➢ Prepared CdS NPs used for photodegradation of RB dye under visible light radiation.
➢ CdS NPs exhibited excellent photocatalytic activity and degraded 98.50% of RB dye in 80 min of light illumination.
➢ The prepared NPs demonstrated good stability and reusability, after fourth run of reuse 85% of activity retained.

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