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Synthesis, Characterization of Cadmium Sulphide nanoparticles and its Application as Photocatalytic degradation of Congored

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Abstract: A wide range of methods have been implemented for the removal of synthetic dyes from wastewater to decrease their impact on the environment. Photocatalytic degradation is proved to be the most popular and effective technique for the removal of dyes from aqueous solution. Therefore, we synthesized Cadmium Sulphide nanoparticles using thiourea as a source for Sulphide ion in the presence of Cadmium nitrate. The prepared nanoparticles were characterized by FTIR spectroscopy, x-ray diffraction and SEM. The Photocatalytic activity of CdS nanoparticles was tested for degradation of Congo red dye.

Keywords: Cadmium Sulphide, Congo red, XRD, SEM and FTIR.

1 Introduction

There are various physical, chemical and biological methods for synthesizing CdS nanoparticles [1-5].Metal sulfides in the form of nanoparticles exhibit Photocatalytic activity similar to the nanoparticles of metal oxides and other inorganic compounds [6,7]. Nanoparticles of transition metal compounds are proved to possess enhanced physical properties, such as optical, magnetic, semiconducting, electrical and catalytic properties compared to the bulk materials [8,9]. Therefore, synthesis, size characterization and the Photocatalytic activity of popular and well-used material CdS is chosen for the present investigation. These materials in the nano size can act as substitute to noble and costlier metals in terms of imparting abundance, cost effectiveness, and stability. Photocatalytic is a greener pathway that can be adopted for the oxidative degradation of organic matter without involving corrosive solvents. The present work is devoted to utilize the Photocatalytic properties of CdS NPs for the oxidative degradation of aqueous source pollutants and intensely colored dye such as Congo red Upon irradiation with Visible light, the dye solutions having nanoparticle catalysts produce a hole [H+] and an electron (e-) pair, which can recombine or dissociate at the surface of catalyst particle [10-12]. These interact with the dye substrates causing dye degradation. Effects of composition of dye and catalysts, and their nature are studied. The trend in the Photocatalytic activity observed shows that CdS NPs are more efficient.

There are many Semiconductor photo catalysts such as CdS, ZnS, CdSe, TiO2, etc., have attracted extensive attention for the degradation of organic dye pollutants in water [13]. Among the semiconductors whose Photocatalytic properties have been studied, CdS is the most commonly used, because it is effective, stable, harmless, and inexpensive [14-16]. The present work Solution Combustion technique is chosen because it is low cost, simple technique and novel approach to study the transparent conducting materials and it is a potentially useful technique for large area deposition. In the present work we have synthesized CdS nanoparticles. The method of synthesis is found to have a pronounced effect on the catalytic activity of CdS. In the present investigation CdS has been synthesized by in presence of thiourea and its Photocatalytic activity was tested for degradation of Congo red.

2 Materials and Method

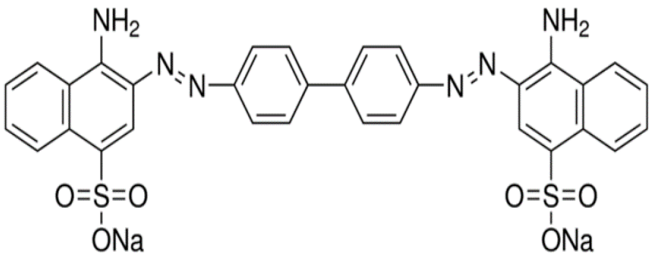

Figure 1. Structural Formula of Congo Red.

Cadmium nitrate Cd $(NO_3)_2.4H_2O$ and thiourea (NH_2CSNH_2) purchased from Hi-Media chemicals, Mumbai, India and distilled water. All chemicals were analytical grade. Dye sample (Congo red) was collected from S. D. Fine Chemicals. Dye was used without further purification. Full scan of dye was taken with the help of

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UV-vis spectrophotometer and maximum absorbance was observed at 497 nm. Molecular structure of Congo red dye is illustrated in Figure.1.

2.1 Synthesis procedures for CdS nanoparticles

Stoichiometric amounts of Cadmium nitrate and fuel as thiourea were calculated using the total oxidizing and reducing valences of the compounds which serve as numerical coefficients for Stoichiometric balance. Initially Cd $(NO_3)_2$ and NH_2CSNH_2 were dissolved in 100cm^3 silica crucible using double distilled water. Then the crucible was placed in a preheated muffle furnace at 500°C for calcinations. The resulting combustion product was grinded to make it amorphous.

2.2 Photocatalytic activity

Dye solution was prepared and stirred in dark for 15 minutes for it to attain equilibrium. Cadmium Sulphide was then added to the dye solution in order to catalyze the photocatalytic degradation. The solution with the catalyst was stirred in the dark for 15 minutes for the solution to attain equilibrium so that the loss of compound due to adsorption can be taken into account. It was then irradiated with visible light. The degradation process was continued for 2 hours in order to achieve complete degradation. Samples were removed at regular time intervals of 30minutes in order to measure the decrease in concentration of dye. The absorbance and concentration of the samples were checked using a UV-Visible Spectroscopy at the maximum wavelength of the dye. The dye chosen for degradation was Congo red. The concentration of the dye was 50ppm.

3 Results and Discussion

3.1 Fourier Transform Infrared (FTIR) Spectroscopy

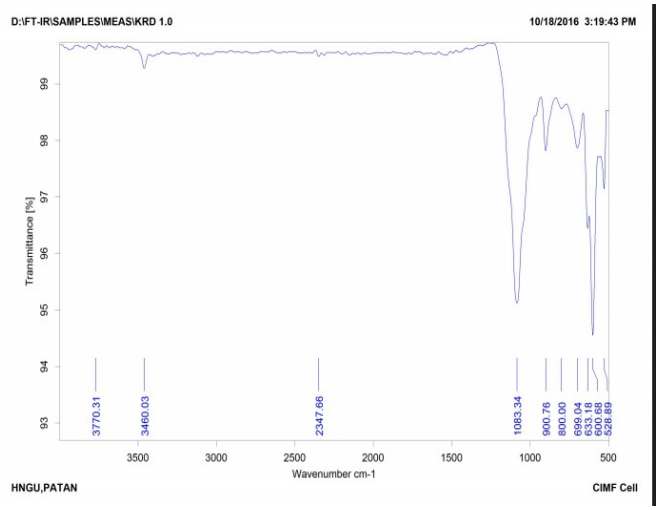

Figure 2. IR spectrum of CdS

The bands at 650 cm^{-1} and 730 cm^{-1} correspond to Cd-S stretching band. The bands at 1620 cm^{-1} and 3550 cm^{-1} are attributed to bending vibration of water and band at to O-H stretching of adsorbed water on surface of CdS.

3.2 X-ray diffraction studies

XRD is a very important experimental technique that has long been used to address all issues related to the crystal structure of solids , including lattice constants and geometry, identification of unknown materials etc., Fig 1 (a) shown the powder X-ray diffraction pattern of pure CdS Nanoparticle. The diffraction peaks positioned at 2θ values of 24.820, 26.520, 28.200, 36.620, 43.720 and 52.850 match well with hexagonal wurtzite phase of CdS (JCPDS card no.89-2944) and can be indexed respectively to the (1 0 0), (0 0 2), (1 0 1), (1 0 2), (1 1 0), (2 0 1) crystal planes. Figure: 4 shows x-ray diffraction pattern of CdS.

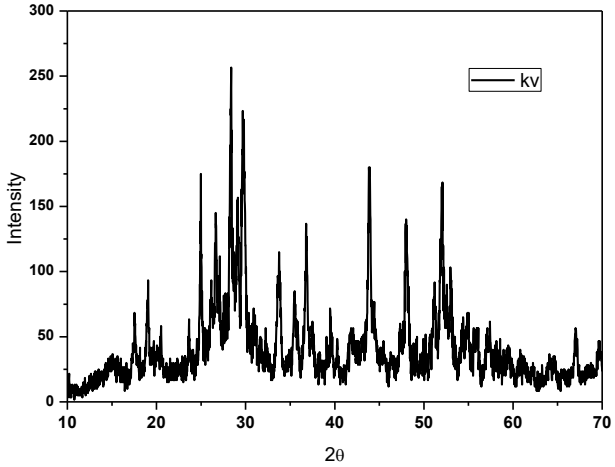

Figure 3. XRD pattern of pure CdS

The crystalline size D of the Nanoparticle was found from the peak width with the Scherer's formula,

$$D = 0.9\lambda / \beta \cos\theta,$$

Where D is the average crystalline size, λ is the X-ray wavelength, β is the full width half maximum (FWHM) of the diffraction peak, θ is the diffraction angle. The average particle size is found to be 27 nm.

3.3 Scanning electron microscopy

The SEM micrograph of the CdS calcined is shown in Figure 5. It can be seen that the particles adopt irregular morphology with different sized particle. From the image it is clear that the particles were highly agglomerated in nature. This might be due to the fact that the agglomeration may be induced during the crystal growth itself because of the small size regime which is evident from the XRD analysis.

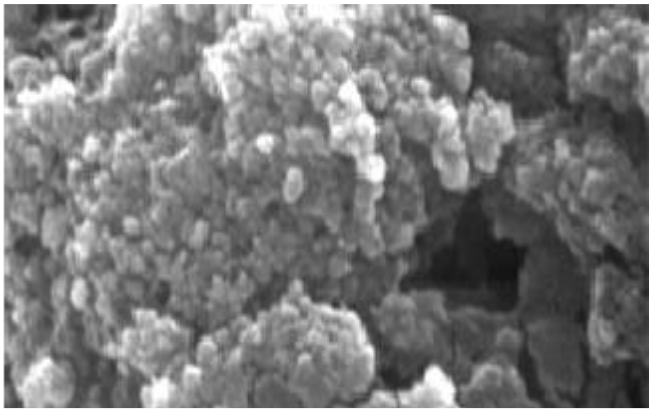

Figure 4. SEM image of CdS nanoparticles.

4 Photocatalytic activity of CdS Nanoparticles

4.1 Effect of CdS Loading

Table 1: Absorbance of CR Soln. With Irradiation time

Catalyst Dose of CdS (g/L)	Absorbance of 25 ppm CR soln.				
	0 Min.	30 Min.	60 Min.	90 Min.	120 Min.
0.1 gm.	0.68	0.27	0.20	0.16	0.12
0.2 gm.	0.68	0.20	0.12	0.05	0.04
0.3 gm.	0.68	0.16	0.09	0.06	0.04
0.4 gm.	0.68	0.10	0.06	0.02	0.01
0.5 gm.	0.68	0.20	0.16	0.12	0.08
0.6 gm.	0.68	0.26	0.18	0.14	0.12

The percent removal increases as the CdS dose increases. The results summarized in Table 2 and also illustrated in Figure: 3.

Table 2: % Dye Degradation with Irradiation time

Catalyst Dose of CdS (g/L)	% Dye Degradation				
	0 Min.	30 Min.	60 Min.	90 Min.	120 Min.
0.1 gm.	-----	60.29	70.58	76.47	82.35
0.2 gm.	-----	70.58	82.35	92.64	94.11
0.3 gm.	-----	76.47	92.64	97.05	94.11
0.4 gm.	-----	85.29	97.05	97.05	98.52
0.5 gm.	-----	70.58	76.47	82.35	88.23
0.6 gm.	-----	61.76	73.52	79.41	82.35

The optimum concentration of CdS required for the decolourization of 25 ppm Congo Red solution was examined with the catalyst amount from 0.0 - 0.6 g/L. The results summarized in Table 1 indicate that the required photons, when the concentration of CdS increased to 0.4 g/L, were thoroughly absorbed. This shows that an increase in the amount of catalyst to the level consistent with the optimized level of light absorption increases the amount of decolourization.

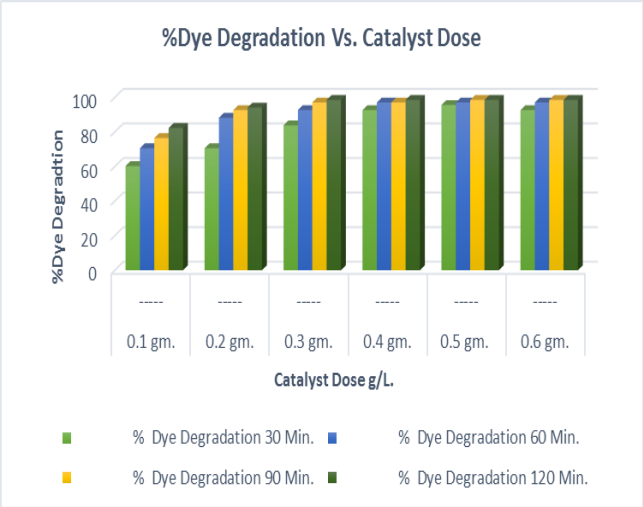

Figure 5. Effect of Catalyst Dose.

4.2 Effect of Initial Dye Concentration:-

Table 3: Effects on Initial Dye Conc. On Dye Degradation

Dye Concentration in ppm	Catalyst Dose (g/L)	% Dye Degradation
25 ppm	0.4 g/L	98.52
50 ppm		93.91
100 ppm		91.94
150 ppm		87.87
200 ppm		64.91

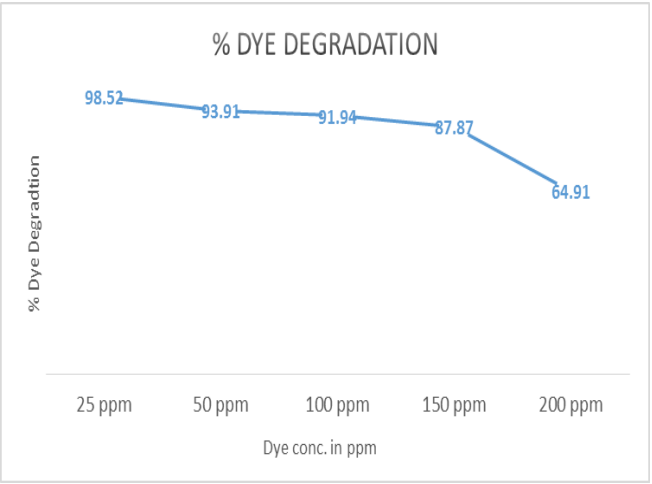

Figure 6 Effect of Initial Dye Conc.

Figure 6 illustrates the effect of initial dye concentration on the photo catalytic degradation rate of dyes in the range of 25 ppm to 200 ppm. As it can be observed, disappearance

rate was found to be inversely affected by initial concentration of dyes. The maximum concentration of dye that could be degraded by 0.4 g/L of CdS for CR.

The Degradation efficiency of CR is found to decrease with an increase in the initial dye concentration.

4.3 Effect of pH

The effect of pH was studied under equilibrium conditions. The pH of the solution was adjusted with either dilute HCI or NaOH before experimentation. The pH of the samples were maintained using a Digital pH meter. The pH meter was calibrated with 4.0 and 9.2 buffers. The dye concentration was maintained at 25 ppm. Figure 6 show the effect of pH on % decolourization of Congo red using CdS nanoparticles. It is observed that the removal of dye by CdS was maximum at pH 7 to 8.

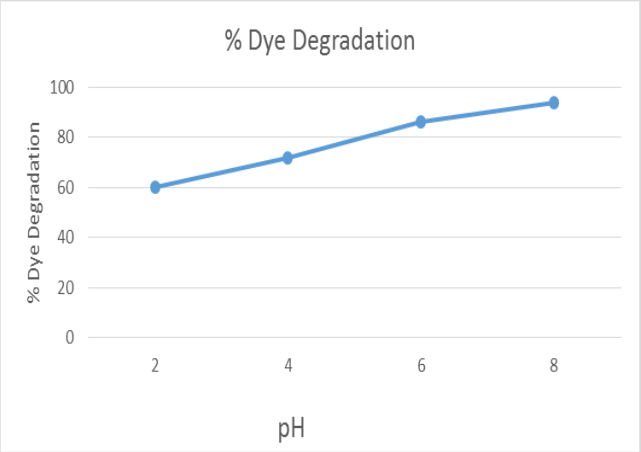

Figure 7: Effect of pH

5 Conclusion

CdS nanoparticles synthesized by using thiourea as source of Sulphide ions in presence of cadmium nitrate showed high activity for Photocatalytic degradation of Congo red. CdS nanoparticles can be efficiently applied for the degradation of Congo red. Following conclusions are drawn from the results of the present study. The Photocatalytic degradation process increased to some extend with increase in catalyst dose but decreased with increase in dye concentration. The maximum degradation was observed with 0.4 g/L dose of CdS. Photo degradation efficiency decreased with increase in reaction pH and the highest efficiency was observed at pH 8. The percentage of photo degradation of CR was 98.52% when the solution was irradiated by the visible light for 120 min.

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