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Photooxidation of 2-Butanol by using CdS semiconductor nano-particles

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Abstract
In present world photo catalysis is emerging as a new and advanced technique for oxidative removal of Organic and Inorganic waste from water. In this technique semiconductor Nano-particles are used as photo catalyst which on irradiation generates a pair of electrons (e^-) and holes (h^+) which act as a redox pair to mediate a chemical reaction on catalyst surface. In present work CdS Nano-particles are used as a semiconductor photo catalyst for photo catalytic oxidation of 2-butanol. The progress of reaction has been monitored spectrophoto metrically by noting the absorbance of reaction mixture at different intervals of time. The effect of various parameters like concentration of reactant, time of irradiation, amount of photo catalyst and various types of different photo catalyst on the rate of the reaction has been observed. The use of CdS as a semiconductor photo catalyst is due to its lower band gap energy (2.4 ev) due to which it majorly absorb solar radiations.

Keywords: Nanotechnology, semiconductor, 2-butanol, irradiation, Nano-particles, photo catalyst, CdS,

Introduction
The world is facing the challenge of purification of water and other resources. The use of synthetic chemicals as raw materials or intermediates in textile industries has resulted in the generation of hazardous waste as process effluents along with solid residues. The release of such compounds into water courses as a result of inadequate treatment could be detrimental to natural ecosystem and human health.

The main purpose of waste water treatment is the removal of toxic substances from water and to make water usable for industrial and domestic purposes, and sustainable for life. Semiconductor photo catalyst combined with nanotechnology is a fast expanding area of research. In photo catalysis light is used to excite a semiconductor material which is used in a wide range of reactions including mineralization of Organics, destruction of bacteria, and generation of fuels. Photo oxidation methods are the process in which semiconductor absorbs the photon of ultra-band gap energy due to which an electron (e^-_{cb})-hole(h^+_{vb}) pair is generated (Figure 1) in the bulk of the solution. These electrons hole pairs then move towards the surface where they react with an adsorbed hydroxyl ion to produce an adsorbed hydroxyl radical which can than oxidize the organic substrate.

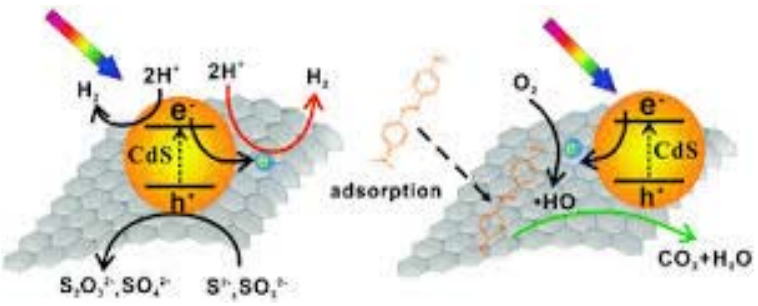

Fig 1: Photooxidation Process of CdS semiconductor photo catalyst.

The aim of present work is to access the photo catalytic activity of CdS semiconductor photo catalyst. Among the various heterogeneous (AOPs) photo catalysts available, the CdS is chosen as a photo catalyst for oxidation process due to its high effectiveness, low cost and

easy availability. Further CdS has very low band gap energy (E_{bg}-2.4 ev) due to which it majorly absorb solar radiation which makes it highly efficient and affordable photo catalyst as compared to other photo catalyst which have high band gap energy. In present study 2-butanol has been chosen as a model system for evaluating the efficiency of photo catalyst as an oxidant. Basic Interest has led on mechanistic questions such as Identify, Lifetime and mode of reactions of the dominant photo oxidizing species generated on the surface of photo catalyst by 2-butanol on visible irradiation.

Experimental

To the aqueous solution of 2-butanol (4.50 x 10^{-2}) M, CdS (0.500 gm) crystals are added in a 200 ml beaker. The reaction mixture was then exposed under visible light by placing 30 cm below tungsten lamp of intensity 200 W. Air was bubbled continuously into the reaction mixture for continuous stirring of the reaction mixture. The progress of the reaction was studied by photo metrically at different intervals of time and qualitative tests were also performed to test the ketone. After 4 hours of irradiation the reaction mixture start to give best of ketones which are further confirmed by preparation of its derivative i.e. 2, 4-Dinitrophenyl hydrazones (M.Pt.-176^{o}C). On the basis of physical, chemical and spectral data it was reported that the photo product of oxidation reaction of 2-butanol was 2-butanone. The appearance of peak at 1725 cm^{-1} in IR spectral analysis shows the presence of (R_2C=0) ketonic group in reaction mixture. The decrease in the intensity of Peak in the region 3650-3600 cm^{-1} shows the progress of the reaction i.e. the alcohol molecules were oxidized to ketonic group.

Results and Discussion

After completion of photo catalytic reaction the photo product was characterized by its usual qualitative test for ketonic group. The various qualitative tests are tabulated below in Table 1.

Table 1

Sr. No.	Test	Observation
1.	Sodium-nitro prusside test	Wine-red Coloration
2.	m-dinitro benzene test	Red Color
3.	2,4-dinitro phenyl hydrazine test	Orange ppt of 2,4-dinitro phenyl hydrazone

On the basis of above qualitative tests it was confirmed that the photo product of oxidation reaction of 2-butanol was a ketone.

3.1 Effect of Substrate

The percentage yield of photo product was estimated by varying the concentration of substrate on Photo catalytic reaction. The results are reported in Table 2.

Table 2

Sr. No.	[Concentration of Substrate] x 10^{-2}	Percentage Yield of Photo Product
1.	2.50	15%
2.	3.66	17%
3.	4.50	20%
4.	5.80	21%
5.	6.95	22%

3.2 Effect of Time of Irradiation

The effect of time of irradiation on Photo catalytic oxidation of 2-butanol was studied over the range of 2 to 7 hours. Obtained results show that maximum yield was obtained in first three hours. The minimum time of irradiation for maximum yield was found to be 5 hours. Beyond this the rate of photo oxidation of alcohol was almost constant.

Table 3

Sr. No.	Time (in Hrs)	%age yield of Photo product
1.	2	15%
2.	3	17%
3.	4	19%
4.	5	23%
5.	6	24%
6.	7	24%

The increase in the rate of reaction with increase in the time of irradiation can be explained on the basis that by increasing the time of irradiation, more number of photons will strike the catalyst surface so that the absorption of light increases which further increases the generation of electron (e^{-})-hole (h^{+}) pairs. These electron-hole pairs act as a redox pair which mediates the chemical reaction at the surface of the photo catalyst. Hence the rate of oxidation of alcohol increases with increase in the time of irradiation.

3.3 Effect of concentration of Photo catalyst

By keeping the amount of substrate and intensity of light constant, the effect of amount of catalyst has been studied on the rate of photo chemical reaction. The results are reported in table 3 as follows:

Table 4

Sr. No.	[Amount of Catalyst] x 10^{-3}M	% yield of photo product
1.	4.50	16%
2.	5.25	19%
3.	6.00	23%
4.	7.35	24%
5.	8.50	25%

Substrate = [4.50 x 10^{-2}] M
Intensity of Light = 200 W
Time of Irradiation = 4 Hours

The observed data shows that the increase in the yield occur only upto certain amount of photo catalyst. In present investigation maximum yield was obtained for [6.00 x 10^{-2}] M concentration of photo catalyst. Any further increase in photo catalyst showed no increase in the percentage yield of photo product. This observation can be explained on the basis that on initial stage an increase in the amount of catalyst increases the number of active sites for the substrate molecules to react and to perform the photo catalytic reaction, but further increase in catalyst concentration does not lead to increase in the product concentration because on increasing the catalyst amount beyond a certain limit there are no substrate molecules left for adsorption on catalyst surface.

3.4 Effect of nature of Photo Catalyst

The effect of the nature of photo catalyst on photo catalytic reaction was studied by using different photo catalyst like ferric oxide, cadmium sulphide, titanium oxide, zinc oxide and zinc sulphide. The results are summarized in Table 4.

Table 5

Sr. No.	Type of Photo Catalyst	Band Gap (ev.)	Wavelength (nm)	% yield of Photo Product
1.	Fe_2O_3	2.2	564	14%
2.	CdS	2.4	516	15%
3.	TiO_2	3.1	400	17%
4.	ZnO	3.2	388	17%
5.	ZnS	3.6	345	19%

Substrate = [4.50 x 10^{-2}] M
Intensity of Light = 200 W
Time of Irradiation = 4 Hours
Amount of Photo Catalyst = 0.500 gm

From the above data it was observed that the yield of Photo Product increases with the increase in the band gap of semiconductor. The yield was higher in the case of ZnS. This can be explained on the basis that the for the semiconductor having more band gap energy, absorb more efficiently the radiation and hence the rate of formation of electron hole pairs increases which further increases the rate of photo chemical reactions.

Conclusion

From the observed data it has been concluded that the rate of photo oxidation of alcohol increases with increase in the concentration of substrate and photo catalyst up to a certain limit and a further increase in the rate of reaction has been observed by increasing the time of irradiation. The photo catalytic oxidation of 2-butanol into 2-butanone by using photo catalytic semi-conductor Nano particles is a low temperature, non-energy intensive approach. Photooxidation using Nano catalyst has offered very effective, efficient, durable and eco-friendly approach. This method is more cost effective, less time and low energy consuming with very less waste generation than conventional method using oxidizing agents. From the present study it has been concluded that the above technique of photooxidation by semiconductor Nano particles can be used for chemical waste remediation for purification of water and other resources. The organic matter present in water as a waste material can be oxidized to simple inorganic compounds (CO_2 and H_2O) on the surface of semiconductor Nano particles by absorbing solar Radiations. However certain precautions are to be taken to avoid any threat to human health or environment due to Nano particles.

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