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Synthesis and Application of CdS nanocrystalline thin films

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ABSTRACT

In present investigation CdS nanocrystalline thin films were deposited on glass substrate by using two different methods i.e. sol gel and spray pyrolysis. Optical characterisation of prepared nanocrystalline films was performed by UV Visible spectroscopy. Optical characterization indicates that film deposited by sol gel has band gap 3.25 and spray deposited film shows band gap 2.87eV. Prepared thin films then applied to the photocatalytic experiment of reduction of Cr (VI) from aqueous solution. Various process parameters have been studied in order to optimise efficient photocatalytic reduction of Cr (VI).

Keywords: CdS, Sol gel, Spray pyrolysis, Photocatalytic.

INTRODUCTION

Extensive use of chromium containing compounds in industrial processes releases chromium result in contamination of surface water, sub surface water and soil. Waste waters resulting from metal plating, textile, pulp and paper, leather tanning, metal finishing, pigment, and dyes, photography, wood preserving and metallurgical industries contain heavy metals particularly chromium in higher concentration [1]. Though chromium is essential nutrient for plant and animal, however accumulated higher concentration it causes serious disorders and diseases and it can ultimately become lethal [2]. ISO and WHO fixed, considering the toxicity, The maximum tolerance limit for total chromium for public water supply and for bathing ghats, at 0.05 mg/L [3]. The application of activated carbon for the removal of chromium and other heavy metals, colours, odour and taste, other organic acids by adsorption process from the industrial wastewater is common practice. In industrial wastewater chromium is mostly found in two forms one is hexavalent and the other is trivalent whereas the hexavalent form is more common and hazardous to biological activities. [3] At higher concentrations all compounds of chromium are toxic. Exposure of human beings to chrome bearing dust for longer period will cause skin irritation and corrosion of skin, ulcer formation, liver damage, respiratory tract infection, nausea,

vomiting, severe diarrhea, [1]. Considerable work was done on the removal of chromium from wastewater by different methods like chemical precipitation, electrochemical reduction, ion exchange, evaporation and concentration, electrolysis and electroplating [4-6]. These all methods are not economical, when the effluent contains chromium in very large quantities or resulting from small scale industries. Several workers have also tried for water treatment of dyes by adsorption over low cost materials [7-9]. Each method has its own disadvantages for examples, the use of charcoal is technically easy but has high waste disposal cost while in filtration low molar mass dyes can pass through the filter system. Photocatalytic reduction and oxidation receives much attention due to ability of the photocatalytic technique to completely degrade organics and inorganic pollutants into water and CO₂ without generating any harmful byproducts. The technique has been widely employed for the photomineralisation of a large number of organics and inorganics. In photocatalytic degradation generally two forms of semiconductor catalyst are used one in powder form and another is solid supported material i.e. thin film. The use of powder catalyst has some disadvantages like separation of catalyst after each run, non reusability, continuous stirring during the reaction etc. Therefore thin film photocatalysis attains more popularity than other techniques.

Several methods are reported for the preparation of CdS thin film such as electrodeposition, [10] Pulse laser deposition [11], Physical vapor deposition [12], vacuum evaporation [13], close space sublimation [14] But all these methods have sophisticated requirements in order to precise temperature control, high pressure etc. Besides all above methods Sol gel and spray pyrolysis techniques are simple, convenient and cheaper.

Hence in this research we have selected these two methods for the deposition of CdS thin film and applied in photocatalytic experiment for removal of dye.

MATERIAL AND METHODS

All reagents used are of analytical purity, CdCl₂ and Thiourea is used as precursor for Cd and S respectively and which were purchased from LOBA Chemie, Mumbai, India, Polyethylene glycol, Ethanol and Acetic acid was also purchased from LOBA and used without further purification.

2.2. Thin Film Preparation:

2.2.1) Sol-gel Method:

For the deposition of CdS thin film by sol gel dip coating method two solutions have been prepared 1: Polyethylene glycol was dissolved in ethanol and acetic acid was added to ethanoic solution under stirring, which was continued for one and half hour. 2: Cadmium chloride and thiourea were dissolved in ethanol under stirring and the stirring was for one hour. The prepared solution 2 was mixed to solution 1 and was stirred again for five hours to obtain the final sol for deposition of thin film. The dip coating technique was used to deposit thin film using above sol. After deposition annealing of the film is necessary in order to remove solvent and residual organics and film densification. So the film has been post annealed in air at 150 °C.

2.2.2.) Spray pyrolysis:

CdS thin films were deposited onto glass substrates by spray pyrolysis method at 250 °C substrate temperature. 0.025M solutions of cadmium chloride hydrate and thiourea were used as starting materials. Glass microslides of the size 7.5 cm 2.5 cm were used as substrates. Prior to deposition these substrates were washed with water, then boiled in concentrated (2M) chromic acid and kept in distilled water for 24 h. Finally the substrates were ultrasonically cleaned in ultrasonic bath (SYSTRONICS) for 10 min. The temperature of substrate was controlled by an iron constantan thermocouple. The spray rate employed was 4 ml/min and kept constant throughout the experiment. Air was used as a carrier gas. After deposition, the film was allowed to cool at room temperature.

The Optical properties of thin films deposited by both methods have been studied by using double beam spectrophotometer (SYSTRONICS 2203).

2.3.) Photocatalytic Experiment:**2.3.1) Instrumentation:**

The photocatalytic degradation was carried out in photocatalytic reactor, which consists of a cylindrical pyrex glass reactor, a double walled quartz cooling water jacket and 400 W medium pressure Hg lamp with nominal wavelength range 220-1400 nm. The cooling water jacket was set up inside the reactor to maintain the temperature to be within range of 25-310C, preventing excessive heating of the reaction mixture. The reaction solution was stirred with magnetic stirrer at a constant speed to maintain well mixed during the experiment. The pH metric measurements were made on equiptronics digital pH meter (Model-E610) fitted with glass electrode which was previously standardized with buffer of known pH. The change of dye concentration was measured by UV Visible spectrophotometer (SYSTRONICS 2203).

RESULT AND DISCUSSION

3.1) Optical Properties: The optical absorbance spectra of CdS thin films deposited by Sol gel and Spray pyrolysis are shown in Fig 1, from the spectrograph the absorption edge of sample found to occur in the range 450-550 nm the optical band gap E_g can be estimated from tauc plot

$$\alpha h\nu = A(h\nu - E_g)^n$$

Where E_g is the band gap corresponding to particular transition occurring in film, A is a constant, ν is transition frequency and the exponent n characterizes the nature of band transition. The graph between $h\nu$ vs. $(\alpha h\nu)^2$ plotted and shown in Fig 5 the extrapolation of straight line to $(\alpha h\nu)^2 = 0$ axis gives the value of energy band gap of CdS thin film. The band gap of thin films observed to be 3.25 and 2.87 eV for sol gel and spray deposite film respectively.

3.2.) Photocatalytic Behavior:

Photocatalytic property of thin films were studied by applying both films in photocatalytic reduction of Cr (IV) from aqueous solution. Different process parameters have been studied in order to optimise efficient reduction of Cr (VI) from aqueous.

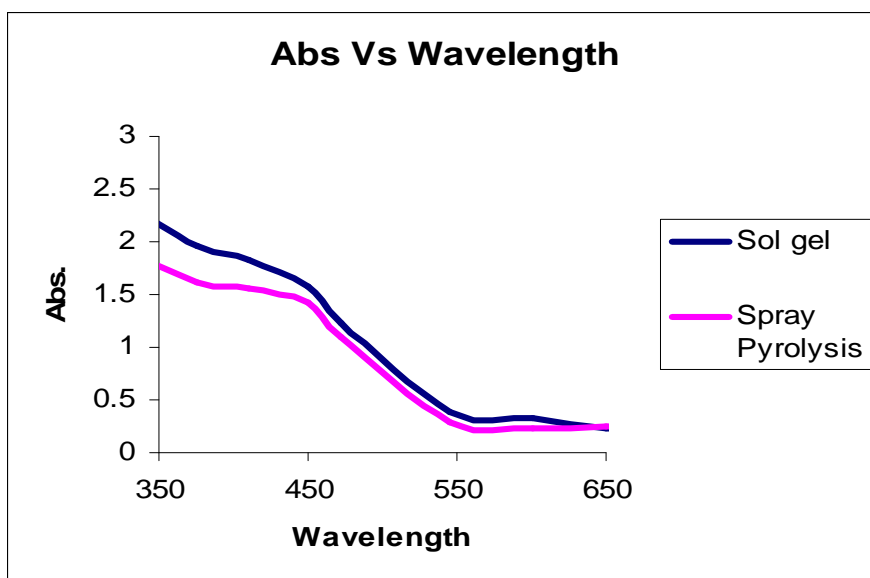
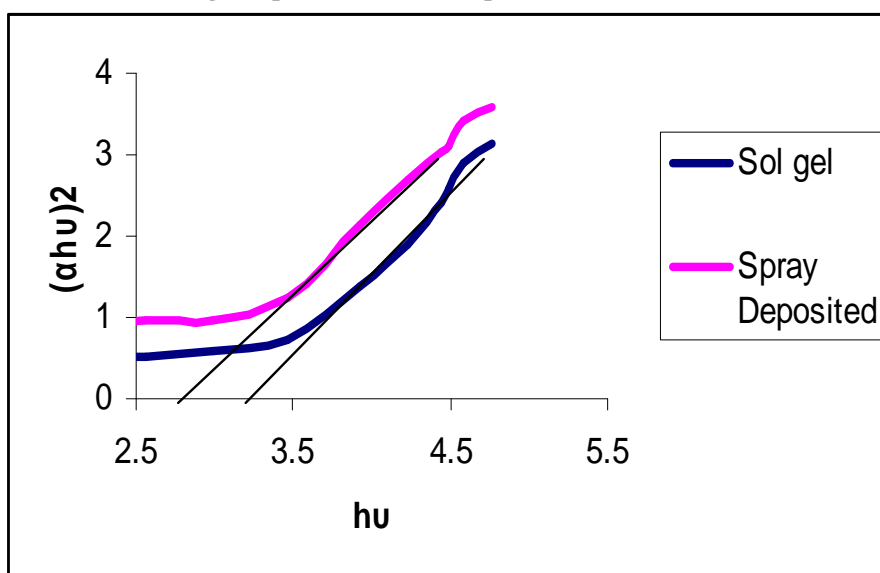
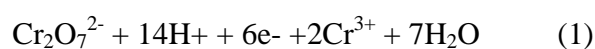


Fig.1. Optical absorbance spectra CdS thin films

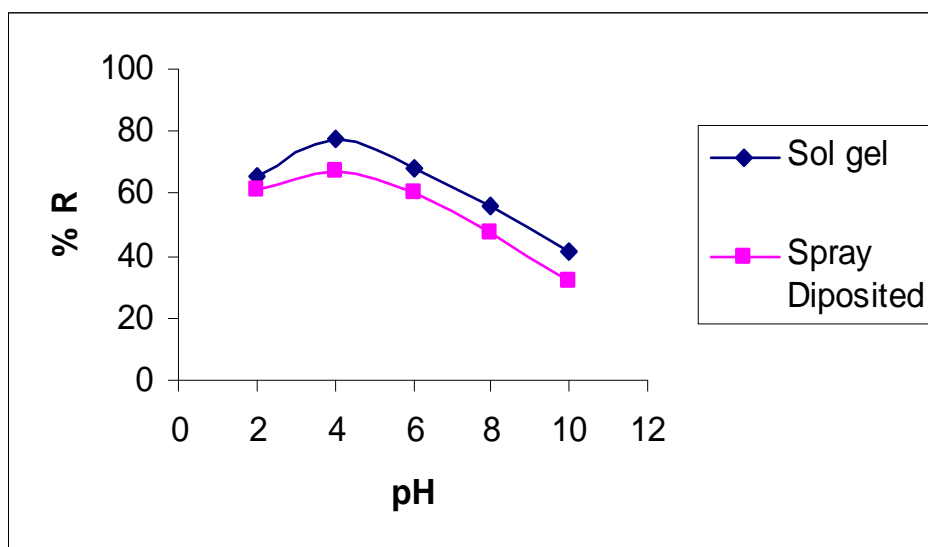
Fig 2. Energy band gap determination of CdS thin films from $h\nu$ vs. $(\alpha h\nu)^2$

3.2.1) Effect of pH:

A series of experiments were conducted at different pH values ranging from 2 to 10, (Fig.3). The figure shows the change of concentration for the photocatalytic reduction only (i.e., the results shown are obtained after it was ascertained that an adsorption equilibrium has been attained and the lamp was turned on) and indicates that reduction rates increase with decreasing pH. Because $\text{Cr}_2\text{O}_7^{2-}$ ions are the predominant species at medium to low pH values, the reduction of Cr(VI) by Photogenerated electrons can be described as follow



pH 4 was optimum pH observed.



11

Fig.3. Effect of pH on percent removal of Cr (VI) by CdS thin films.

3.2.2.) Effect of Contact time:

Effect of contact time and concentration for the removal Cr (IV) from aqueous solution for CdS thin film. The experiments were carried out at room temperature, pH 4.0 and initial concentrations (60 mg /L) for different time intervals up to 180 min. Percentage of Cr (VI) removal increases with increasing in contact (fig.4).The percentage removal increases with increase in contact time and reaches equilibrium after 30 min. Therefore 30 min is optimum contact time for better Cr (VI) ion removal.

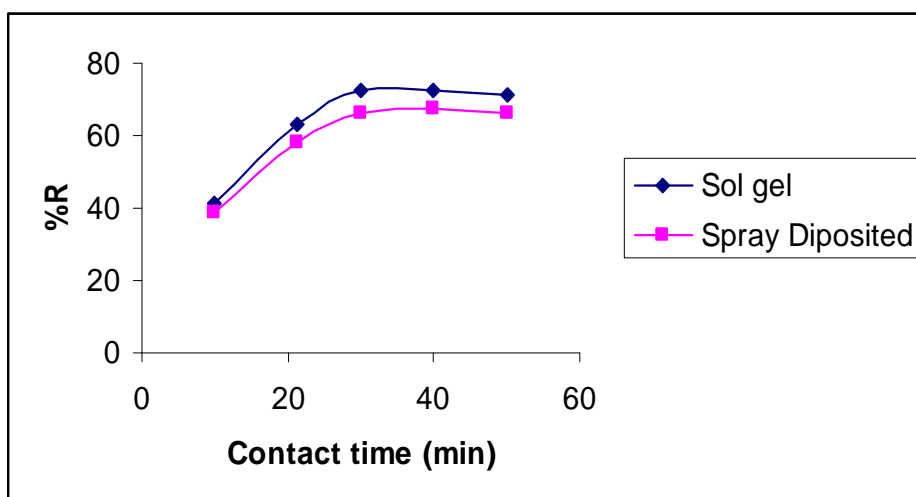


Fig.4. Effect of contact time on percent removal of Cr (VI) by CdS thin films.

3.2.3.) Effect of Initial concentration:

The rate of photodegradation of Pb(II) was studied by varying the metal ion concentration from 10 to 70 mg/L (Fig.5) because for fixed catalyst concentration active sites remains the same ,the number of substrate ions was accommodating the inter layer space increases so that the

degradation decreases. Moreover, the photodecomposition efficiency of Cr(VI) is much higher with thin film prepared by sol gel method than prepared by spray pyrolysis.

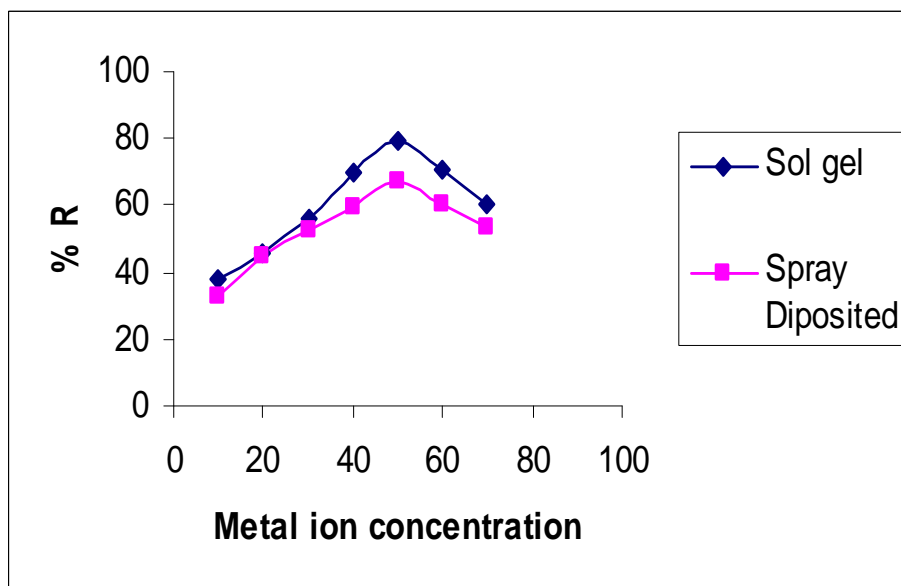


Fig.5. Effect of initial metal ion concentration on percent removal of Cr (VI) by CdS thin films

CONCLUSION

CdS nanocrystalline thin film can be easily deposited by Sol gel and Spray pyrolysis technique. Optical study indicates that sol gel derived thin film shows higher band gap than that film deposited by spray pyrolysis. Hence sol gel derived thin film exhibits better photocatalytic property than spray deposited film. In reduction of Cr(VI) at pH 4, contact time 30 min., initial metal ion concentration 40 ppm found to be optimum conditions for better metal ion reduction. Sol gel derived thin film shows better photocatalytic property than spray deposited thin film; it may be due to the spray deposited film having a smaller band gap, which results in a higher electron-hole pair recombination rate, hence decreasing its semiconducting property. Findings from this research can provide fundamental information on nanocrystalline CdS thin film preparation for other environmental pollution control applications.

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