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# ZnO composite mediated photocatalytic decolourization of Coralene Red F3BS dye in presence of solar light

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# ABSTRACT

An attempt has been made to develop innovative synthesis method of ZnO composite involving hydrazine hydrate as combustion agent (by using solution combustion method) and synthesized composite was characterized by Scanning Electron Micrographs and X-Ray Diffraction. The ZnO composite was employed as catalysts for solar light induced photocatalytic decolourization of Coralene Red F3BS, disperse azo dye. Photodecolourization of aqueous solution containing Coralene Red F3BS dye was investigated by comparing absorbance readings taken at regular intervals of time (15 minutes). Photo-catalytic experiments were conducted systematically at different pH levels and catalyst dosages on dye solution of different dye concentrations to understand and evaluate the respective effects of these parameters on the photo-decolourization of dye solution. The decolourization of Coralene Red F3BS was recorded (99.65 %, 99.02% and 99.80 % for the dye concentrations of 50ppm, 75 ppm and 100 ppm respectively) at neutral pH level in a relative short period of 105 minutes. © 2011 Trade Science Inc. - INDIA

#### **INTRODUCTION**

The presence of the synthetic and non-biodegradable compounds such as azo dyes in textile effluents are of main environmental concern due to their widespread use, potential to produce toxic aromatic amines and low removal rate during various wastewater treatment facilities. The effluents from dyeing processes of textile fibers and fabric can modify the ecosystem by reducing the transparency of water and

# KEYWORDS

ZnO composite; Decolourization of dyes; Coralene Red F3BS; Photo-catalytic decolourization.

the sunlight penetration thereby altering the photosynthetic activity and the natural diffusion of gases<sup>[1]</sup>. In addition, azo dyes and their derived products are proved to be carcinogenic agents<sup>[2]</sup>. Traditional methods of coloured wastewater treatment facilities, such as adsorption on activated carbon, flocculation and electro-coagulation have the major disadvantage of being phase transfer methods which again require subsequent treatment and disposal. On the other hand, biological treatments take a long time for the

> Full Paper

effluent to reach the required standards, and also produce a large quantity of sludge, which normally cannot be reused<sup>[3]</sup>.

Recently photocatalytic decolourization process is one among the emerging technologies, aimed at degradation of water pollutants, especially refractory organic substances<sup>[4-12]</sup> because of its high effectiveness and sustainability in the long run. The main advantages over other competing processes include complete mineralization, no disposal problems, low energy consumption and low  $cost^{[7]}$ . During the past few decades, photocatalytic process involving TiO<sub>2</sub> and ZnO semiconductor particles under UV light illumination have been shown to be potentially advantageous and useful in degrading dyes in textile effluents<sup>[12-14]</sup>. Many studies have shown that photocatalytic activity of ZnO has more photosensitivity than TiO<sub>2</sub> for the dye degradation<sup>[13-16]</sup>.

Although many ZnO nanoparticles have been reported as photocatalyst, in the present investigation, an attempt has been made to develop innovative synthesis method of ZnO composite involving hydrazine hydrate as combustion agent. The ZnO composite particles obtained from this method have shown high photosensitivity for the decolourization of aqueous Coralene Red F3BS dye solution under natural solar radiation.

# **MATERIALS AND METHODS**

The commercially available water soluble disperse azo dye Coralene Red F3BS (Colourtex Limited, Surat, Gujarat, India), 99% LR grade Hydrazine hydrate (S D fine-chem limited, Mumbai, India) and Dry Zinc Chloride (Jobal Chemie, Mumbai, India) were procured and used without further purification. UV-VIS Spectrophotometer (Systronics) was used for recording the absorbance and  $\lambda_{max}$  (wavelength of light





at which maximum absorbance have been recorded for Coralene Red F3BS dye).

# Preparation of ZnO composite

Aliquot of Hydrazine Hydrate was added drop wise at room temperature to Zinc Chloride in 1:1 molar ratio under vigorous stirring. The obtained white precipitate after complete combustion of  $ZnCl_2$  with  $N_2H_4$ . $H_2O$  was suspended in 50 ml of distilled water for hydrolysis and heated to 50° C with constant stirring for 2 hours in order to achieve destruction of the agglomerates and redispersion into primary particles. The residue was filtered and dried in hot air oven at 100° C for 3 hours and investigated for photocatalytic decolourization of Coralene Red F3BS dye in presence of natural sunlight.

# X-ray diffraction and scanning electron micrographs of prepared ZnO composite

The X-Ray Diffraction pattern (figure 2) of the synthesized composite was recorded and it confirms the presence of other contaminants such as Zinc Chloride [Joint Committee for Powder Diffraction Standards (JCPDS) card no 74-1752] and Zinc Perchlorate Hydrate (Zn (ClO<sub>4</sub>), XH<sub>2</sub>O) (JCPDS card no 88-0309) along with ZnO (JCPDS card no 80-0075) in the synthesized composite. According to X-Ray Diffraction report, the crystallite size of the ZnO composite particles calculated by using Scherrer's formula was found between 18nm to 82 nm. The Scanning Electron Micrograph images of composite samples have shown the typical texture and morphology of ZnO composites. It also indicated the presence of some vestigial inorganic biproduct from the combustion media and starting material.

# Photocatalytic experimental procedure

Initially the 50 ppm, 75 ppm and 100 ppm Coralene Red F3BS dye solutions were prepared by dissolving 50 mg, 75 mg and 100 mg of Coralene Red F3BS dye in 1000 ml distilled water respectively. Photocatalytic decolourization experiments were carried out in the presence of ZnO composite with different dosages of catalyst, for each of standard dye solutions. Primarily, reaction volume of 100 ml (of 50 ppm dye concentration) was taken in clean beakers (Borosil<sup>®</sup>) and various doses (50 mg, 100 mg, 150 mg, 200 mg, 250 mg, 300



# Full Paper

mg, 350 mg, 400 mg) of ZnO composite particles were dispersed directly to the beaker in presence of direct sunlight. Experiments were repeated for 75 ppm and 100 ppm dye solutions separately. Absorbance readings of the dye solution were recorded at regular intervals (15 minutes) by UV/VIS spectrophotometer. Further, the experiments were carried out at 3, 7 and 12 pH levels (adjusted by dilute hydrochloric acid and sodium hydroxide) to study the effect of pH on photodecolourizaton of various concentrations (50 ppm, 75 ppm and 100ppm) of Coralene Red F3BS aqueous solution. The percentage of decolourization was calculated by using the following equation,

Decolourization (%) =  $\frac{A_o - A_t}{A_o} \times 100$ 

Where,  $A_o$  is the initial absorbance of dye solution and





A<sub>t</sub> is absorbance at time 't'.

# **RESULT AND DISCUSSION**

# Effect of initial dye concentration

The effect of dye concentrations were investigated by varying the initial standard concentration (from 50 mg/l to 100 mg/l) and results obtained are shown in figure 3. It is observed that the rate of decolourization of Coralene Red F3BS dye was inversely affected by the concentration of dye i.e., higher the dye concentration decreases the rate of decolourization process. The experimental results depicted that the rate of photo decolourization was slow at 75 ppm and 100 ppm dye solutions in comparison with decolourization in 50 ppm dye solution.





Figure 2 : Scanning electron micrographs and x-ray diffraction pattern of ZnO composite.

Materials Science Au Indian Journal



Figure 3 : Effect of initial dye concentration (50 ppm, 75 ppm and 100 ppm) on the decolorisation of Coralene Red F3BS dye solution at normal pH (8.2) at differet catalyst load; 0.05g, 0.1g, 0.15g, 0.2g, 0.25g, 0.3g, 0.35g and 0.4g.

#### Effect of catalyst loading

To determine the effect of the catalyst loading, a series of experiments were carried out by varying the amount of catalyst (from 0.05 g/100 ml to 0.4 g/ 100 ml) and decolourization is depicted in figure 4(a), 4(b), and 4(c). Decolourization efficiency was maximum at the high catalyst dosage from 0.25 g/100ml to 0.4 g/100ml when compared to lower loading levels from 0.05 g/100ml to 2.0 g/100ml. The initial decolourization rate was very fast and decreases exponentially with time at all catalyst dosages for the prepared standard dye solutions. In the first rapid phase (of 45 minutes), 40-80% decolourization was

recorded for lower dosage (0.05 g/ 100ml to 0.2 g/ 100ml) of catalysts and 85-90 % decolourization observed at higher dosages (0.25 g/100 ml to 0.4 g/ 100 ml). In the second phase of the reaction (from 45 minutes to 105 minutes), decolourization was increased by 19-40% and 12-14% at lower catalyst dosages and higher catalyst dosages respectively (figure 4). The recorded experimental data depicted that, in all dye concentrations (50 ppm, 75 ppm and 100 ppm) at neutral pH condition, catalyst dosage of 0.3 g/100ml was found to be successful in achieving highest (99.48 %, 99.02 % and 99.60 % respectively) decolourization.



Figure 4 : Effects of catalyst load on percentage of decolourization at dye concentrations (a) 50 ppm, (b) 75 ppm and (c) 100 ppm with respect to time and catalyst load.

#### Effect of pH

F3BS by synthesized ZnO composite at different pH The decolourization efficiency of Coralene Red level has been analysed in presence of solar light (figure



# Full Paper

5). It was found that the decolourization of dye was negatively pH sensitive, i.e., at pH 12.9 (alkaline condition) and 3.0 (acidic condition) the decolourization

was less (61.31 % and 59.59 % respectively) where as at pH 7.0 (normal) the percentage of decolourization (99.65 %) was too fast (105 minutes) for all the stan-



Figure 5 : Effect of pH of dye solution on percentage of decolorisation of 50ppm Corelene Red F3BS dye solution at differet catalyst load; 0.05g, 0.1g, 0.15g, 0.2g, 0.25g, 0.3g, 0.35g and 0.4g.

dard concentrations of dye solutions.

# CONCLUSION

In the present work, ZnO composite of various size and shape have been synthesized and characterized by X-Ray Diffraction and Scanning Electron Micrograph studies. These composite particles were prepared through a simple combustion method and were tested for their efficiency of photocatalytic decolourization of solution containing disperse azo dye Corolene Red F3BS under natural sunlight. It was observed that, at neutral pH level, the synthesized ZnO composite was highly photosensitive and effective in complete decolourization (~99.5%) of selected azo dye solution (50 ppm, 75 ppm and 100 ppm) in a shorter period of time (105 minutes) and found to be dependent on the pH of dye solution. This protocol can be employed effectively in the treatment of textile effluents, as it is economically feasible compared to any other traditional oxidative processes.

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An Indian Journal

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