Photocatalytic Degradation of Congo Red Dye on Thermally Activated Zinc Oxide

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Received 12 October 2014; Accepted 28 November 2014

Abstract. Congo Red in aquatic system has been recognized as a serious problem in living organism because of its toxic nature and therefore, its degradation is highly essential. In the present work, the efficacy of thermally activated ZnO was studied for photocatalytic degradation of Congo red (CR) dye. The batch operation was carried out by irradiating the aqueous solution of dye in the presence of semiconductor. The effect of various parameters such as catalyst loading, pH, oxidant dose, intensity variations and initial concentration of the dye on degradation was investigated. The maximum degradation capacity of ZnO for CR was found to be 11.8 mg g⁻¹ at optimized condition [303 K, 60 min, pH (8), dose (0.05g)]. The pseudo-second-order kinetic model described the CR degradation process with a good fitting. Thermodynamic parameters such as ΔH, ΔS, and ΔG were calculated, which indicated that the process was spontaneous and exothermic in nature, that was evident by increasing the randomness of the dye at the solid and liquid interface. The high CR degradation ability and regeneration efficiency of this photocatalyst suggest its applicability in industrial systems and data generated would help in further upscaling of the degradation process.

Keywords: Congo Red; Kinetic model; Photo-catalytic degradation; Thermodynamic study; Zinc Oxide

1. INTRODUCTION

Environmental problems associated with organic pollutants promote the development of fundamental and applied research in the area of environment (Ahmad et al., 2011). Several of these chemicals such as azo dyes, herbicides and pesticides are actually present in rivers and lakes, and are in part suspected of being endocrine-disrupting chemicals (Ohko et al., 2001; Coleman et al., 2000; Wang and Hong, 2000; Hong et al., 1998). Synthetic dyes are the major industrial pollutants and water contaminants (Modirshahla et al., 2007). These dyes are used extensively in the textile industry for dying nylon, cotton, wool, and silk, as well as for colouring oils, fats, waxes, varnishes, and plastics. The paper, food, cosmetic, and leather industries are also major consumers of these dyes (Chen, 2007).

The discharge of dye-coloured wastewaters into the aquatic ecosystem represents both environmental and public health risks because of the negative ecotoxicological effects and bioaccumulation in wildlife. Most importantly, many dyes contained in wastewaters can decompose into carcinogenic aromatic amines under aerobic conditions which can cause serious health problems to humans and animals (Akar et al., 2009; Kiran et al., 2009). Also, dyes can cause allergy, dermatitis, skin irritation and cancer in humans (Fiorentina et al., 2010). Additionally, colour in surface water may affect photosynthesis by preventing light penetration, thereby compromising aquatic life (Senturk et al., 2010).

Excess use of various dyes in the textile industry has led to the severe water contamination by releasing the toxic and coloured effluents, which are usually disposed by various physical and chemical methods, such as coagulation or flocculation (Golob et al., 2005; Allegre et al., 2004), electrocoagulation (Alinsafi et al., 2005), coagulation/carbon degradation process (Papic et al., 2004), adsorption, advanced oxidation process (AOP). However, most of the methods barely transfer the pollutants from one phase to another without destruction or have the other limitations (Wang et al., 2007). But advanced oxidation process (AOP), a photo-catalytic reaction is a promising and emerging process for the purification of water and air (Suzuki et al., 2008). In recent years, as a promising tool to surrogate the traditional wastewater treatment, semiconductor-assisted photocatalysis has fascinated the public concern for its ability to convert the pollutants into the harmless substances directly in the waste water (Kulkarni et al.,...
2014; Habib et al., 2013; Khattab et al., 2012). Till now, many kinds of semiconductors have been studied as photo-catalysts including TiO\textsubscript{2}, ZnO, CdS, WO\textsubscript{3}, and so on (Baruah et al., 2012; Ram et al., 2012; Yogendra et al., 2011) among them ZnO is the most extensively used effective photo-catalyst for its high efficiency, photochemical stability, nontoxic nature, and low cost. The wide spread use of ZnO as an effective photo-catalyst in practical application which is sensitive to UV light (Rao and Chu, 2009).

Therefore, continued study of ZnO is quite necessary when illuminated with an appropriate light source, the photocatalyst generates electron/hole pairs with free electrons produced in the empty conduction band leaving positive holes in the valence band. The photogenerated electron/hole pairs are capable of initiating a series of chemical reactions at the catalyst surfaces, which involve adsorbed organic pollutants and surficial water species that result in the decomposition of the organic compounds. The formations of relatively harmless end products represent another attractive feature of this process (Rego et al., 2009). It is well known that the highly reactive OH\textsuperscript{-} radicals and holes are generated on the surface of photocatalyst under the radiation of UV light. Due to the fact, the photocatalytic property is a surface reaction (Farbod and Jafarpoor, 2012), thermally activation process which can improve the high effective surface area. These nanoparticles have been reported as efficient photocatalysts against Congo Red dye at different conditions such as optimum catalyst dosage and effective pH level. A ZnO-assisted photocatalytic degradation study of these Congo Red dyes under UV light (254-nm) irradiation has been sparse.

In the last few decade, most of the removal techniques of Congo Red by normal zinc oxide with uv illumination were studied the optimized condition of different parameters, adsorption kinetics and isotherm models. But there have been no report regarding the thermal activation of ZnO with optimization of process parameters, adsorption kinetics and thermodynamic feasibility and spontaneity of photodegradation of Congo Red. Kinetic and thermodynamic data can be used to predict the rate and spontaneity of degradation respectively. Therefore the aim of the present study was formulated for degradation efficiency of CR onto thermally activated ZnO by the illumination of uv with optimized conditions.

2. MATERIALS AND METHODS

2.1. Thermally activation of ZnO

Thermally activated ZnO powder was obtained by heating the pure ZnO powder at 500°C in a muffle chamber by using nickel crucible. The sample was heated at 500°C for 30 minutes and cooled slowly at room temperature. After cooling the sample is ready for dye degradation study.

2.2. Adsorbate and other chemicals

All chemicals were used of analytical grade. Congo Red (CR), the typical anionic dye was selected as the adsorbate in the present study. A stock solution of 1000 ppm was prepared by dissolving 1000 mg of CR in 1L deionized water. The intermediate solution was prepared by appropriate dilution of the working solution. The pH of the solution was adjusted by addition of either 0.1M HCl or 0.1M NaOH solutions respectively.

2.3. Experimental procedure

In all experiments, the required amount of thermally heated ZnO was suspended in 100 ml of Congo Red dye using a magnetic stirrer. pH was measured by using pH meter (Eutech 700). At predetermined times; 5 ml of reaction mixture was collected and centrifuged (4,000 rpm, 15 minutes) in a centrifuge. The absorbance at 497 nm wavelengths of the supernatants was determined using ultraviolet-visible spectrophotometer (Systronics- 1203). Photocatalytic reaction was carried out in a homemade photoreactor equipped with a Philips120W, high pressure mercury lamp as a source for near-UV radiation. The reactor was consisted of graduated 400 cm\textsuperscript{2} Pyrex glass beaker and a magnetic stirring setup. The lamp was positioned perpendicularly above the beaker. The distance between the lamp and the graduated Pyrex glass was 10 cm. The whole photocatalytic reactor was insulated in a wooden box to prevent the escape of harmful radiation and minimized temperature fluctuations caused by draughts.

2.4. Degradation experiment

Degradation measurement was determined by batch experiment of known amount of the adsorbent with 100 ml of aqueous Congo Red solution of known concentration (4-10 ppm) in a series of 250 ml conical flasks. Dye concentration in the reaction mixture was calculated from the calibration curve. Degradation experiments curve were conducted by varying initial concentration of the dye, pH, contact time, catalyst
dose under the aspect of degradation kinetics. The amount of dye adsorbed onto zinc oxide powder at time t, qt (mg/g) was calculated by the following mass balance relationship.

\[
q_t = \frac{(c_0 - c_e) \times V}{m}
\]

The dye removal efficiency i.e., % of degradation was calculated as

\[
\% \text{ of degradation} = \frac{(c_0 - c_e) \times 100}{c_0}
\]

where \(c_0\) is the initial dye concentration (mg/L), \(c_t\) is the concentration of the dye at any time t, V is the volume of solution (L) and m is the mass (g) of zinc oxide powder (ZnO).

### Table 1: The dye removal capacity at different wave lengths

<table>
<thead>
<tr>
<th>Sl No.</th>
<th>Parameter</th>
<th>Removal (%)</th>
<th>Degradation capacity (mg/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Only UV(Without ZnO)</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>2</td>
<td>Only ZnO(without UV)</td>
<td>47.95</td>
<td>5.70</td>
</tr>
<tr>
<td>3</td>
<td>Both ZnO &amp; UV</td>
<td>96.45</td>
<td>11.57</td>
</tr>
</tbody>
</table>

### Table 2: Summery of parameters for various kinetic models

<table>
<thead>
<tr>
<th>Kinetic model</th>
<th>Equation</th>
<th>Constants</th>
<th>(q_{\text{exp}}) (mg g(^{-1}))</th>
<th>(q_{\text{cal}}) (mg g(^{-1}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pseudo first order</td>
<td>(\log(q_e - q_t) = \log q_e - k_L \frac{t}{2.303})</td>
<td>(R^2 = 0.995)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>(K_L = 0.0819 \text{ min}^{-1})</td>
<td>11.50</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>(R^2 = 0.9999)</td>
<td></td>
<td>12.03</td>
</tr>
<tr>
<td>Pseudo second order</td>
<td>(\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{t}{q_e})</td>
<td>(K_2 = 0.0586 \text{ g.mg}^{-1} \text{min}^{-1})</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

\(q_e\) and \(q_t\) are the amount of dye adsorbed (mg g\(^{-1}\)) at time t and at equilibrium and \(K_L\) (min\(^{-1}\)) is the Lagergren rate constant of first-order degradation and \(K_2\) (g mg\(^{-1}\) min\(^{-1}\)) is the second-order degradation rate constant.

### Table 3: Thermodynamic parameters for adsorption of Congo Red onto ZnO particles

<table>
<thead>
<tr>
<th>Temperature(K)</th>
<th>(\Delta G(KJ/mol^{-1}))</th>
<th>(\Delta H(KJ/mol^{-1}))</th>
<th>(\Delta S/(J/mol^{-1} K^{-1}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>303</td>
<td>-10.0680</td>
<td>-6.335</td>
<td>11.45</td>
</tr>
<tr>
<td>323</td>
<td>-9.6690</td>
<td></td>
<td></td>
</tr>
<tr>
<td>343</td>
<td>-10.1138</td>
<td></td>
<td></td>
</tr>
<tr>
<td>373</td>
<td>-10.8370</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

### 2.5. Photocatalysis

Photocatalysis may be termed as a photoinduced reaction which is accelerated by the presence of a catalyst (Mills et al., 1997). These types of reaction are activated by absorption of a photon with sufficient energy (equal or higher than the band gap energy of the catalyst (Carp et al., 2004). The absorption leads to a charge separation due to promotion of an electron (e\(^{-}\)) from valence band of the semiconductor catalyst to the conduction band, thus generating a hole (h\(^{+}\)) in the valence band (Gaya et al., 2008). The recombination of the electron and the hole must be prevented as much as possible if a photocatalyst reaction must be favoured. The ultimate goal of the process is to have a reaction between the activated electrons with an oxidant to produce a reduced product and also a reaction between the generated holes with a reductant to produce an oxidant product. The photogenerated electrons could reduce the dye or react with electron acceptors such as O\(_2\) adsorbed ZnO surface or dissolved in water, reducing it to superoxide radical anion O\(_2^-\) (Konstantinou et al., 2004). The photogenerated holes can oxidize the organic molecule to form R*, or react with OH\(^-\) or H\(_2\)O oxidizing them into OH radicals. The resulting OH radicals, being a strong oxidizing agent (standard
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reduction potential +2.8V) can oxidise most azo dyes to the mineral product.

3. RESULTS AND DISCUSSIONS

3.1. Degradation and photocatalytic degradation

CR degradation experiments were carried out by photocatalytic agent thermally activated zinc oxide into 100 ml of solution in the dark at 30°C with degradation accelerated by magnetic stirring at 400 rpm. Photocatalytic degradation was assessed in the presence or absence of UV irradiation provided by a 120 w middle lamp producing an intensity of 2.1 mw/cm² with the main emission wave length of 254 nm positioned 10 cm from the surface of the solution. To saturate the surface degradation capacity of the nanomaterials, each solution was determined by uv-visible spectro-photometrically.

![Graph 1: Effect of catalyst dose on photodegradation of CR dye (6 ppm), pH (8.0), Temp. (303K), λ (254 nm), Time (60 min)](image1)

![Graph 2: Effect of pH on photodegradation of CR dye (6ppm), ZnO (0.5mg/L), Temp. (303K), λ (254 nm), Time (60 min)](image2)

3.2. Effect of pH

The effect of initial solution pH on the degradation capacity at equilibrium conditions is shown in Figure 2. The results indicate that the dye degradation capacity increased from 11.2 mg/g to 11.8 mg/g with an increase in the value of pH from 5 to 8. However the dye degradation capacity was decreased from 11.8 to 11.3 when pH increased further from 8.5 to 10. This behaviour can be explained by the zero point charge of the photocatalyst. Since the pH_{zpc} of ZnO is 9.3±0.2 (Bahnemann et al.,1987), the surface of the
catalyst is positive below pH 9.3. Again the given pKa for Congo Red is 4.1, therefore Congo Red is negatively charged above pH 4.1 that might result in electrostatic attraction between the nanocatalyst and Congo Red and will increase the degree of photodegradation. It was further observed that the degradation efficiency increases with increasing pH value from 5 to 9 and above decreases. Hence the maximum degradation range was shown at the pH of 8 - 9. The final pH of photocatalytic treatment were 8.5 that means the photocatalytic removal of colour and degradation were observed to be faster in slightly alkaline pH than in acidic or neutral pH range (Neppolian et al., 2002; Tang et al., 1995; Bahnemann et al., 1994; Hustert et al., 1992). This is due to two reason: (a) at very low pH, ZnO particle agglomeration reduces the dye degradation as well as photon absorption and (b) in Congo Red, the azo linkage (\(-\text{N=N-}\)) is particularly susceptible to electrophilic attack by hydroxyl radical. However, in very low pH, the concentration of H\(^+\) ions is in excess and H\(^+\) ion interact with azo linkage decreasing the electron densities at azo group. Consequently the reactivity of hydroxyl radical by electrophilic mechanism is also decreased (Gladius et al., 2009). Hence at acidic pH range, degradation efficiency is minimum.

Finally the isoelectric point of ZnO (8.7 – 10.3) is the pH at which a particular molecule or surface carries no net electrical charge. In isoelectric point (IEP), ZnO surface in aqueous suspension are generally assumed to be covered with hydroxyl species, Zn-OH. At pH value above the IEP, the predominate surface species is Zn – OH\(^-\) which is less interacted with anionic dye(CR) while at pH values below the IEP, Zn – OH\(^+\) species predominate which is more interacted (Haruta, 2004; Brunelle, 1978).

3.3. Effect of Initial Dye Concentration on photocatalytic degradation of CR dye

The effect of initial dye concentration on the rate of photocatalytic degradation was studied by keeping all other experimental conditions constant at light intensity equal to 254nm, ZnO catalyst dosage was 0.05 g/100ml, pH dye solution was (8) temperature at 303K and changing the initial dye concentration in range (4-10). The results are plotted in Figure 3. These results indicate that the rate of photocatalytic degradation increased with the decreasing of initial dye concentration. As the initial concentration of a dye increases, the colour of dye solution becomes deeper which results in the reduction of penetration of light to the surface of the catalyst, decreasing the number of excited dye molecules. Due to increase of initial concentration of dye more and more organic substances are adsorbed on the surface of ZnO. Therefore, the generation of hydroxyl radicals is reduced, since there are only fewer active sites in the system causing little adsorption of hydroxyl ions, which in turn leads to the decrease in the generation of hydroxyl radicals (Daneshvar et al., 2004). Thus, the rate of hydroxyl radical generation on the catalyst surface, accordingly, will decrease. Similar results have been reported by other researcher for the photocatalytic oxidation of pollutants (Nikazar et al., 2007; Mrowetz and Selli, 2006; Mahmoodi and Arami, 2006; Muruganandham and Swaminathan, 2006; Mahmoodi et al., 2006; Kartal et al., 2001; Goncalves et al., 1999).

Fig. 3: Effect of concentration on photodegradation of CR dye pH (8.0), ZnO (0.5mg/L), Temp. (303K), \(\lambda\) (254 nm), Time (60 min)
3.4. Effect of catalyst loading

The amount of catalyst loading is one of the main parameters for the degradation studies. The results for the dye degradation using various amounts of ZnO (0.02-0.06 g/100ml) are shown in Figure 1. In order to avoid the use of excess catalyst it is necessary to find out the optimum loading for efficient removal of dye. The results showed that when catalyst dosage was increased from 20-50 mg, the decolourization increases from 41% to 98.5%. However, on further increase in dosage of the catalyst beyond 60 mg, there is a slight decrease in the dye removal rate. Table 1 also demonstrated that the colour removal was unchanged in the absence of ZnO indicating that CR dye is difficult to be oxidized by only UV light. The increase in degradation rate with increase in the catalyst loading is due to increase in total active surface area i.e. availability of more active sites on catalyst surface (Goncalves et al., 1999). However, it increases significantly upon addition of ZnO due to the production of higher amount of hydroxyl radical through the interaction of UV with ZnO. But above 50 mg of ZnO, the colour removal efficiency significantly decreased due to decrease of formation of hydroxyl radicals. It should be pointed out that, the catalyst loading affects both the number of active sites on photocatalysts and the penetration of UV light through the suspension (Daneshvar et al., 2003). With increasing catalyst loading the number of active sites increases, but the penetration of UV light decreases due to shielding effect (Gouvea et al., 2000). It should also be noted that the optimum value of catalyst loading is strongly depended on the type and initial concentration of the pollutant and the operating conditions of the photoreactor (Gogate et al., 2004). The optimum concentration of the catalyst for efficient UV photodecolorization and degradation is found to be 0.05 mg/100ml in Figure 1.
3.5. Effect of wave length of uv light

The influence of UV light intensity on the degradation efficiency has been examined on dye solution (6 ppm) at a pH 8.5 and ZnO loading of 0.05 g/100ml. It is evident from the results that the experiment was conducted by three different wave length (254, 312, 365 nm) and percentage degradation increases with decrease in wave length of uv light in Figure 4. The UV irradiation generates the photons required for the electron transfer from the valence band to the conduction band of a semiconductor photo catalyst and the energy of a photon is related to its wavelength and the overall energy input to a photocatalytic process is dependent on light intensity. The rate of degradation increases when more radiation falls on the catalyst surface and hence more hydroxyl radicals are produced. The degradation efficiency is found to be maximum (i.e. 25 W/m²) in the middle area of the photoreactor. Also similar results were reported by Habib et al. (2012); Karimi et al. (2011); Karimi et al. (2010); Akpon et al. (2009).
3.6. Effect of uv irradiation

The effect of UV irradiation on the decolorization of CR was also investigated. There was no observable loss of color when the dye is irradiated with only UV which suggests that the dye is resistant to direct UV-photolysis. The combined action of UV and ZnO however produced an increase from 47.95% to 98.5% in decolorization compared to that of ZnO alone due to the reaction of hydroxyl radicals generated upon photolysis. A higher percentage of removal by ZnO/UV showed that Zn-catalyzed decolorization with UV was more efficient than that of UV-catalyzed in producing the hydroxyl radical responsible for the oxidation of the dye. For ZnO/UV system, a complete color removal was observed after 60 min of irradiation. The high efficiency of this process is due to the formation of more hydroxyl radical which is attributed to zinc/UV-catalyzed (homogeneous and heterogeneous). Table 1 summarized the results for the color removal of CR by all system investigated in this work (Habib et al., 2012; Karimi et al., 2011; Karimi et al., 2010).

3.7. Effect of Irradiation Time

The contact time necessary to reach equilibrium depends on the initial dye concentration. The effects of contact time on the decolouration of Congo Red at 60 min are illustrated in Figure 6. Figure 6 clearly indicates that dye decolouration increase with contact time. Hence it appears that a rapid initial degradation occurs with equilibrium reached in 60 minutes. The first degradation of dye molecule is due to effective interaction of semiconducting materials and dye molecule is faster than the solute – solute interaction (Dogan et al., 2006). However, Movahedi et al., (2009) pointed out that (CR) is strongly adsorbed on thermally heated ZnO particles surfaces through the two oxygen atoms, of the sulphonate group of the dye molecules.

3.8.1. Pseudo first order kinetic model

In this study degradation data are applied to the pseudo first order kinetic models to find the rate constants of dye degradation according to Eq. 3

$$\log (q_e - q_t) = \log q_e - \frac{K_1}{2.303} t$$

(3)

The first order rate constant $K_1$ can be obtained from the slope of plot between $\log (q_e - q_t)$ versus time $t$ is shown in Figure 7.

3.8.2. Pseudo second order kinetic model

A pseudo second order model can be used to explain the dye degradation kinetics. The pseudo second order model can be expressed as

$$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{t}{q_e}$$

(4)

Where $t$ is the contact time (min), $q_e$ and $q_t$ are the amount of dye adsorbed (mg/g) at equilibrium and at any time $t$. A plot between $t/q_t$ versus $t$ gives the value of the constant $K_2$ is shown in Figure 8 and also $q_e$ can be calculated.

As it can be seen from Table 2, the degradation did not comply well with the pseudo first order model because of the absence of linearity between $\log (q_e - q_t)$ Vs t. In this study pseudo second order model fitted better when compare with the first order kinetic model in Table 2. From this study we can described that the experimental value of $q_e$ of first order model are not well matched with the calculated value whereas in case of second order model, the experimental value of $q_e$ are very close to the calculated value. So the photocatalytic degradation of (CR) by zinc oxide in aqueous phase is well fitted with the second order kinetic model.

3.9. Thermodynamic study

Thermodynamic parameters can be determined from the thermodynamic equilibrium constant $K_0$, where $K_0$ is the ratio of degradation capacity (mg.g⁻¹) and concentration at equilibrium state. The thermodynamic parameters ($\Delta G$, $\Delta H$ and $\Delta S$) of the degradation of CR on ZnO were calculated using equations and $\Delta G = -RT \ln K_0$, where $R$ is the ideal gas constant, $T$ is the temperature (K) and $K_0$ is the distribution coefficient calculated from the experiment. The values of $\Delta H$ and $\Delta S$ were calculated from the slope and intercept of the Van’t hoff plots in Figure 9 and listed in Table 3. The thermodynamics of Congo Red degradation has been investigated extensively. The negative value of free energy $\Delta G$ (Table 3) indicates the feasibility of the process and its spontaneous nature in Table 3. The experimental values of $\Delta G$ at different temperatures are negative. The negative values of $\Delta H$ for all the systems confirm the exothermic nature of process. The positive values of $\Delta S$ observed for the removal of CR molecule suggested increased the randomness during the process. The thermodynamic values of parameters for the CR reported in the present study and in good agreement with literature (Vijayakumar et al., 2012).

4. CONCLUSION

The active species generated in the degradation of CR under uv light irradiation were studied using thermally heated ZnO as model systems. The present investigation shows that zinc oxide can be utilized for the removal of hazardous dye from aqueous solution. The removal process is a function of shaking time,
pH, temperature and catalyst dose. It was observed that the removal process followed the pseudo second order kinetic model. The values of qe calculated from pseudo second order plot are in good agreement with the experimental value. The thermodynamic study showed that the process is spontaneous and exothermic in nature. Therefore, it can be concluded that the hazardous dye such as CR can be comfortable degraded by using thermally heated ZnO under uv light irradiation.

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