

Photodegradation Efficiency of Prepared and Commercial ZnO to Remove Textile Dye from Aqueous Solution

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ABSTRACT

Commercial ZnO (*c*-ZnO) and prepared ZnO (*p*-ZnO) were applied under UV and solar light irradiation for evaluating the heterogeneous photocatalytic degradation of Remazol Black B (RBB) solutions. A comparative study of the experimental results demonstrates that, *c*-ZnO is more efficient for removing RBB than *p*-ZnO. The effects of various operational parameters such as the concentration of RBB, amount of ZnO and irradiation time have been studied and the optimized conditions for maximum removal of the RBB were determined and compared. The photodegradation kinetics was studied with modified Langmuir-Hinshelwood mechanism, which reveals the pseudo first order kinetics for photodegradation of RBB in the presence of *p*- and *c*-ZnO. Furthermore, Experimental results also demonstrate that photocatalytic activity for both types of ZnO is higher under solar light irradiation as compared to UV light.

1. Introduction

Wastewater contaminated with industrial effluents or residual dyes from textile, leather, paper, printing, food, solvent, rubber, plastic, cosmetics, petroleum, pesticide, wood-preserving chemical, paint, pigment, pharmaceutical and other industries are the main source of environmental pollution [1] and are the severe setback for the environmental protection issues [2]. Especially in third world countries, waste from various factories, industries, agricultural and domestic effluents are drained directly to different bodies of water sources without prior treatment generating pollution and secondary effects on the environment [3]. Most of the dyestuffs contain a large amount of unfixed azo groups and are difficult to decompose due to relatively stable chemical structures of aromatic systems [4]. Thus disposal of these industrial and house hold effluents into the open sources of water cause severe contaminations to the drinking water, irrigation systems and even in the in groundwater in some regions that cause disruption on human health and ecosystems by changing the reproduction of animals and plants. Therefore, the removal of dyes from effluent is essential not only to protect human life but also from the environmental point of view for securing water resources.

Conventional treatments of such wastewater usually involve adsorption [5, 6], reverse osmosis [7], and chemical coagulations [8]. These methods, however, just transfer dyes from one phase to another (liquid- to solid-phase) and causing secondary pollution [9]. Hence further treatments are required, which are relatively expensive due to repetitive procedures. In this respect, advance oxidation or semiconductor mediated photocatalysis become a potential alternative treatment methodology, which can be applied effectively and efficiently for the degradation of dyes from industrial effluents [10-12]. The efficient oxidation reaction of contaminants under irradiation in water and air using semiconductive particles or photocatalysts like TiO₂, ZnO and their modified versions, has attracted significant attention for several decades [13-15].

Advance oxidation methods of wastewater treatment e.g. photocatalysis not only transfer the pollutants from one phase to another but also mineralized the pollutant into harmless products such as CO₂, water and salts. When a photocatalyst is irradiated with light of energy

equal or greater than the bandgap energy of the semiconductor, the photons are absorbed and electron-hole pairs are generated. These electrons and holes, then, either recombine or drift towards the surface participating in several redox reactions. Subsequently, producing reactive oxygen species (ROS) such as [•]OH radicals, O₂⁻ and H₂O₂ [16-18]. These ROS can activate and induce a number of reactions, which end up in complete mineralization of the pollutants. TiO₂ is one of the mostly used photocatalysts that absorbs merely the UV region of the solar spectrum, which make it poor contestant for harnessing solar energy. On the other hand, zinc oxide (ZnO) has almost similar band gap ($E_g = 3.37$ eV) as TiO₂ and large exciton binding energy (60 meV) at room temperature [19] which lead to the better optical [20] electrical [21, 22] photocatalytic [23], photonic [24], and chemical properties [25]. Intriguing properties such as non-toxicity, higher photocatalytic efficiency, good sensing behavior, ease of production and low production cost makes ZnO excellent alternating counterpart of TiO₂. Although ZnO suffers some disadvantage of the rate of recombination of photogenerated electron-hole pairs [26], larger quantum efficiency than TiO₂ and higher photocatalytic efficiencies have been reported for ZnO [27, 28].

In this study, we have used prepared and commercial ZnO as a photocatalyst for the degradation of dye (Remazol Black B, (RBB)) from aqueous solution and compared the effectiveness of the two ZnO for removing of the RBB. We have also studied the photodegradation efficiency of the RBB under irradiation of UV as well as sunlight in the presence of both types of ZnO.

2. Experimental Methods

RBB was obtained from Dystar, Germany and commercial ZnO was purchased from Merck, Germany and were used without further purification. Preparation and characterization of the *p*-ZnO was described elsewhere [29]. Photodegradation of RBB was carried out in the presence of both *p*-ZnO and *c*-ZnO. Photodegradation of RBB was studied under different experimental conditions. In a typical experiment, a definite amount of the ZnO was taken in a 100 mL beaker. 20.0 mL deionized water was added and kept overnight for soaking and smoothening of the ZnO. 80.0 mL of the specified concentrated dye solution was added to the suspension to obtain a desired concentration of the dye solution. Using a magnetic stirrer solution was stirred constantly 500 rpm. Then, the UV-lamp was turned on and this was taken as "time zero" for the degradation reactions. After definite interval 5 mL of the irradiated solution was

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withdrawn and before analysis, the aqueous samples were centrifuged at 4000 rpm to remove suspended solid ZnO. The absorbance of the clear solution was measured at the $\lambda_{\text{max}} = 595 \text{ nm}$ using UV-visible spectrophotometer (UV-1650 PC, Shimadzu, Japan). The photodegradation of RBB solutions in the presence of sunlight was carried out in the same beaker with all the experimental conditions remaining the same.

3. Results and Discussion

3.1 Effect of Illumination Time

The photodegradation of dye RBB ($5 \times 10^{-5} \text{ M}$) was investigated under UV-light irradiation in the presence of both ZnO. The absorption spectra of the supernatant solution taken from the reaction suspension after various time of degradation are given in Fig. 1. The figure shows the absorption peaks at 595 nm corresponds to the dyes absorption maxima in aqueous solution decreased with irradiation time and a maximum 98% degradation is observed after 130 minutes for *p*-ZnO and 70 min for *c*-ZnO, respectively.

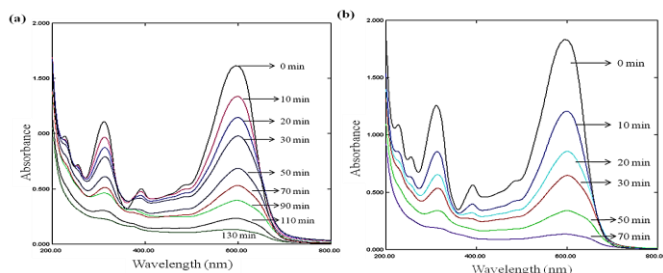


Fig. 1 UV-visible spectra of photodegradation of RBB as a function of time using 0.1 g/100 mL (a) *p*-ZnO and (b) *c*-ZnO, respectively.

3.2 Effect of Catalyst Dosages on Dye Degradation

In order to observe the effect of catalyst loading on the degradation of RBB, a set of experiments were executed by varying the dosage of ZnO from 0.1 – 1.1 g/ 100 mL with other parameters remaining constant ($[\text{RBB}] = 5 \times 10^{-5} \text{ M}$, $\text{pH} = 5.83$). Fig. 2 shows the degradation of RBB as a function of time with the various amounts of ZnO. It is observed that with an increase of ZnO from 0.1 to 1.1 g/100 mL the percent of RBB degradation increase from 65 to 95 % and 80 to 98 % after 90 minutes irradiation of UV-light for *p*-ZnO and *c*-ZnO, respectively. The increase in degradation efficiency with the increase of ZnO loading may be attributed to the higher number of interaction sites on the surface [30]. Moreover, photon absorption capacity also increased with the increase of ZnO which results in higher degradation [31].

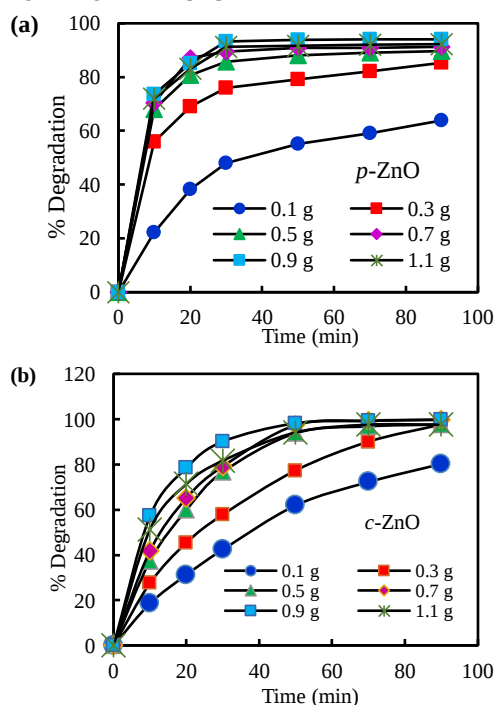


Fig. 2 Effect of catalyst dosage on the degradation of RBB (a) *p*-ZnO and (b) *c*-ZnO, $[\text{RBB}] = 5 \times 10^{-5} \text{ M}$, $\text{pH} = 5.83$

Fig. 3 shows an initial rate of RBB degradation with the increase of ZnO loading. It is found that the rate of degradation of RBB increase steadily with the increase of ZnO loading, reaches an optimum at 0.9 g/100 mL and then decrease again. This observation can be explained in terms of availability of active sites on the catalyst surface and the penetration of UV light into the suspension. Formation of reactive $\cdot\text{OH}$ and other reactive oxidative species (ROS) increase with the amount of ZnO due to the availability of a higher number of active surface sites and more photon absorption. Therefore, there will more frequent and efficient interaction between the ROS and RBB, which enhanced degradation. When the optimum dosage is reached, any further increase of an amount of ZnO leads to scattering of light and reduced the passage of light in the sample. Moreover, at higher concentration, there is a possibility of the aggregation of catalyst particles resulting in decreased number of active surface sites for interaction between RBB and catalyst [31]. Furthermore, at higher concentration of catalyst, some of the activated ZnO might collide with ground state catalyst and get deactivated, which also reduced the photocatalytic activity [32].

According to Fig. 2(a) and (b), photodegradation efficiency is higher for *c*-ZnO than *p*-ZnO, because 98% of dye is degraded after 90 minutes of UV-illumination. But initial rate of degradation is always higher for *p*-ZnO than *c*-ZnO as shown in Fig. 3. This possible reason might be adsorption i.e., in case of *p*-ZnO some extent of adsorption of RBB occurred, but for *c*-ZnO no adsorption was found as reported previously in our previous report [29].

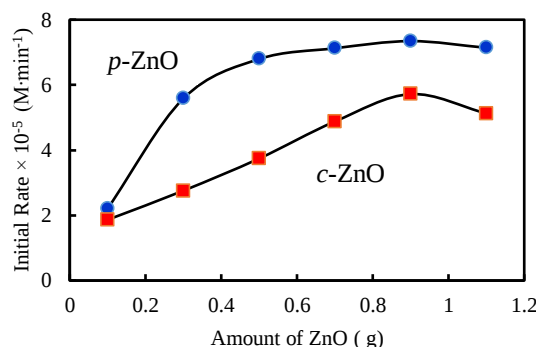


Fig. 3 Initial rate of degradation as a function of ZnO loading for *p*-ZnO and *c*-ZnO

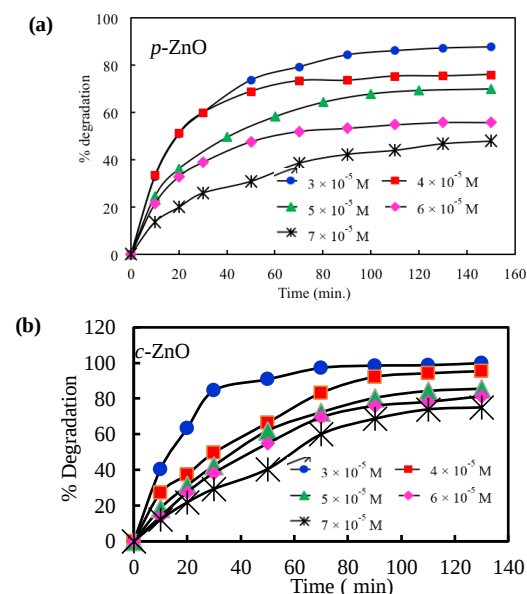


Fig. 4 Effect of initial concentration of RBB on degradation under UV irradiation (a) *p*-ZnO and (b) *c*-ZnO with amount of 0.9 g/100 mL

3.3 Effect of Initial Concentration of RBB

Fig. 4 shows the effect of initial concentration of RBB on photodegradation by using 0.9 g/100 mL of *p*-ZnO and *c*-ZnO. The concentration of RBB solution was varied from $3 \times 10^{-5} \text{ M}$ to $7 \times 10^{-5} \text{ M}$ for both ZnO with $\text{pH} = 5.83$. It is found that when the concentration RBB is low, the percentage of degradation is high and percent of degradation is decreased at higher concentration of RBB. On the other hand, Fig. 5 shows the rate of degradation increase steadily and reaches limited values with

the increase of concentration. Initial increase in the rate of photodegradation with the increase in concentration is due to availability of higher number of RBB molecules in the bulk as well as in the vicinity of the catalyst surface for interaction with the surface generated ROS. But as the concentration of RBB reaches a sufficient values to interact with the optimum amount of available ROS and other reactive species, any further increase of concentration, then, does not enhanced the reactions and thereby rate of degradation becomes independent of concentration of RBB. Furthermore, at higher concentration more reaction intermediates might be formed and a few of them might remain in the system or loosely attached to the surface for relatively longer period, which might hinder interaction between fresh RBB molecules and the ROS [31]. Again, at higher concentration the more number of RBB molecules adsorbed on the catalyst increases the light absorption rather than catalyst surface or the intermediates that are formed and loosely stayed in the system might absorb more photons, thereby decreasing the photons available for ZnO activation. Moreover, the path length of photons entering the solution is decreased and less number of photons is reached on the catalyst surface due to dominant interaction between reactant/intermediate/product results decrease in the generation of ROS species and hence the removal efficiency decreased [33, 34].

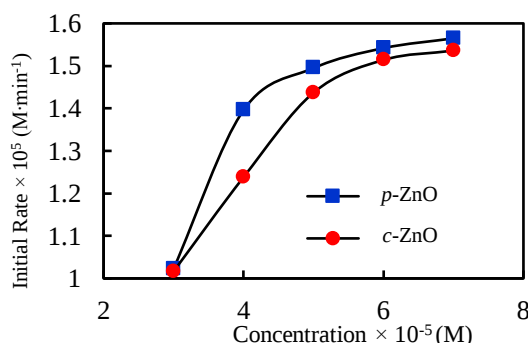


Fig. 5 Effect of concentration of RBB on the initial rate of photodegradation using *p*-ZnO, and *c*-ZnO with amount of 0.9 g/100mL

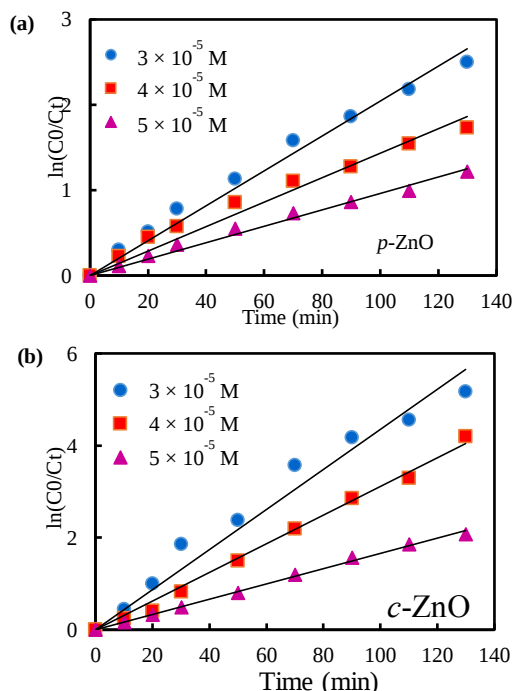


Fig. 6 Kinetics of photocatalytic degradation of RBB by using (a) *p*-ZnO and (b) *c*-ZnO with amount of 0.9 g/100mL

3.4 Kinetic Analysis

The photocatalytic degradation rate of most organic compounds is followed the pseudo-first order kinetics and the first order kinetics models is derived from the modification of Langmuir-Hinshelwood model assuming that there is no interaction between dye and the reaction intermediates for the surface sites as well as the ROS. The final modified pseudo-first order kinetic models for photodegradation is expressed [35, 36] by,

$$\ln \left(\frac{C_0}{C_t} \right) = kt \quad (1)$$

where, C_0 is the initial concentration of the contaminant (mg/L), C_t is the concentration (mg/L) at time t , t is the irradiation time and k is the limiting reaction rate at maximum coverage for the experimental conditions (min^{-1}). According to the Eq. (1) plot of $\ln(C_0/C_t)$ against t will give a straight line for first order reactions and slope of this line will be the pseudo first-order rate constant.

The linearity of the plot of $\ln(C_0/C_t)$ vs t for the photodegradation of RBB in presence of both types of ZnO demonstrates that photocatalytic degradation follows pseudo first-order kinetics (Fig. 6). From the slope of the straight lines the rate constants were calculated and are given in Table 1. It is observed that the rate constant for the photocatalytic degradation of RBB decrease with the increase of concentration of dye. For a definite amount of catalyst and intensity of photon irradiation, the number of surface-generated active species available for interaction is finite. But the number of RBB molecules is excessive at higher concentrations, which reduces a fraction of dye molecules to interact successfully with the ROS and consequently decrease the rate constant [31].

Table 1 Pseudo first order rate constants for the photodegradation of RBB by ZnO, at 25°C and pH = 5.83

Concentration of RBB	Amount of ZnO	k (<i>p</i> -ZnO) min^{-1}	k (<i>c</i> -ZnO) min^{-1}
3×10^{-5} M	0.9 g	0.018915	0.040297
4×10^{-5} M	0.9 g	0.012832	0.032271
5×10^{-5} M	0.9 g	0.009037	0.016528

3.5 Effect of Light Sources

Fig. 7 shows that the rate of degradation of dye RBB in solar light is higher than in UV light for both the ZnO samples. The intensity of this UV light source was measured previously, which was $0.14 \text{ mW cm}^{-2} \text{ s}^{-1}$ [37]. Although the intensity of the sunlight was not measured, this value is about $700\text{--}750 \text{ W m}^{-2} \text{ s}^{-1}$ [38]. This indicates that the intensity of the solar light is much higher than the UV source used in this study. Thus the higher effectiveness of the sunlight compared to the UV light should be related to the higher intensity of the former light sources. It is also noted that although initial rate of degradation of *p*-ZnO is higher than *c*-ZnO due to a content of adsorption, after 90 minutes of irradiation percentage of degradation of RBB is higher for *c*-ZnO compared to the *p*-ZnO, which indicates that *c*-ZnO is more effective for removal of RBB from aqueous solution.

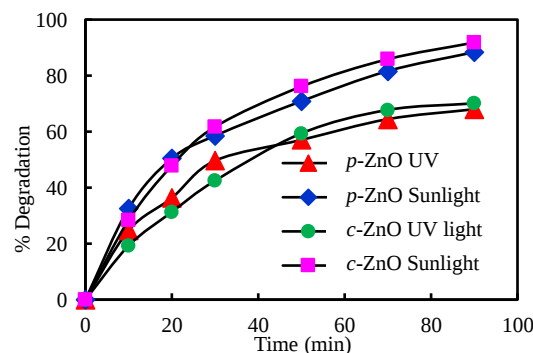


Fig. 7 Effect of different light sources on the rate of photodegradation of RBB using 0.1 g/100 mL of ZnO

3.6 Determination of Intermediate during Photodegradation

To find out any intermediate formed during photodegradation and adsorption, FTIR spectra were recorded after adsorption and 150 minutes of photodegradation. The original FTIR spectrum of shows a number of lines in Fig. 8(a). However, after photodegradation with *p*-ZnO and *c*-ZnO, the peak intensities of almost all the peaks decrease and only a few peaks are available in the spectra including the peak at around 1630 (Fig. 8(b) and Fig. 8(c)), which should be due to the benzene or fused benzene ring derivatives [39]. This indicates a few content of fused benzene type compounds still persistent of after almost complete decolourization of dye.

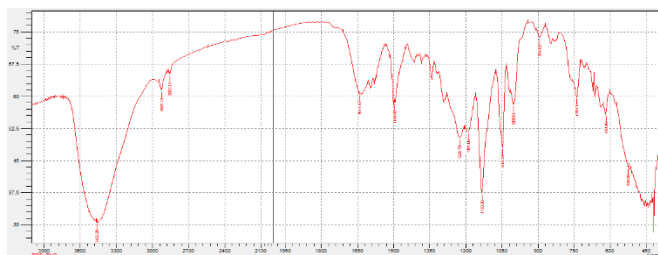
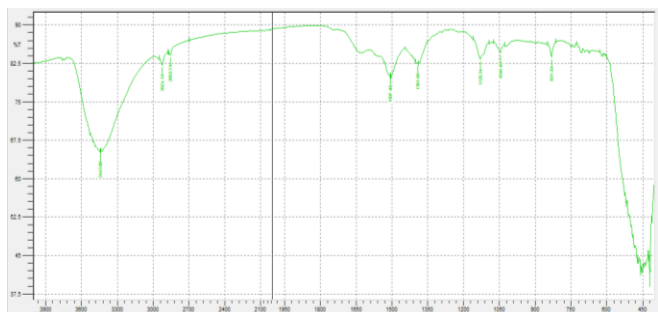
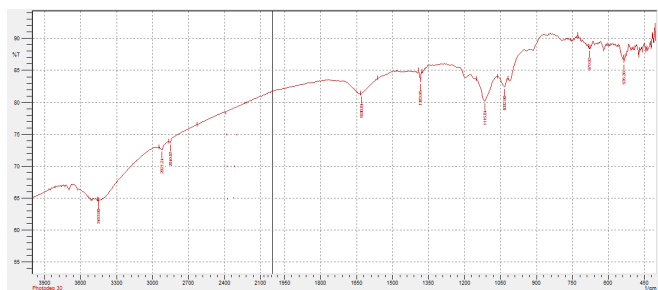


Fig. 8(a) FTIR spectrum of pure RBB

Fig. 8(b) FTIR spectrum of degraded RBB solution after 150 minutes by *p*-ZnOFig. 8(c) FTIR spectrum of degraded RBB solution after 150 minutes by *c*-ZnO

4. Conclusion

A comparative study of photocatalytic activity between two different types of ZnO such as *c*-ZnO and *p*-ZnO were executed. The experimental results suggest that *c*-ZnO is more effective than *p*-ZnO for dye degradation under illumination of both sunlight and UV-light. The optimum parameters studies showed that maximum RBB degradation (98%) occurred at 0.9 g/100 mL of ZnO loading after 90 minutes of irradiation and photocatalytic degradation follows pseudo first-order mechanism. Photodegradation under solar light is found more efficient as compared to UV-light, might be due to higher intensity of the sunlight.

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