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Photocatalytic decolorization of crystal violet in aqueous nano-ZnO suspension under visible light irradiation

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Abstract

Nanosized ZnO was prepared through hydrothermal process and characterized by scanning electron microscopy, X-ray diffraction and laser-induced breakdown spectra measurement techniques. The as-prepared nanosized ZnO was used to investigate the decolorization/degradation of crystal violet, a cationic dye which is extensively used in dyeing/textile industries, under visible light through adsorption studies of the dye solution with ZnO in the dark. The results show that the adsorption of CV on ZnO takes about 200 min to reach equilibrium, and the equilibrium time at a certain concentration of the dye seems to be independent of temperatures that are used for the preparation of ZnO samples. The adsorption data follow the pseudo-first-order kinetic model (Lagergren), and the adsorption pattern follows the Langmuir model.

Prepared ZnO (300°C) was found to be a more efficient photocatalyst among others including pristine ZnO, to decolorize/degrade the dye. The decolorization rate is increased with the decreasing of the initial dye concentration and reached at a limiting value. The catalyst loading also influences the decolorization/degradation of the dye, and decolorization rate is increased with increasing the catalyst loading and reached at a limiting value. ZnO was found to be stable under visible light irradiation at solution pH = 6. The photocatalytic degradation of the dye followed zero-order kinetics, and the Langmuir-Hinshelwood mechanism was found to be valid.

Keywords: Nanosize ZnO; Photocatalyst; Lagergren model; Decolorization/degradation; Langmuir-Hinshelwood model

Background

Crystal violet (CV) is a triarylmethane dye which is extensively used in industries such as textile/dyeing, ball-point pen, paper, leather, additives, foodstuffs, cosmetics, and analytical chemistry [1,2]. CV is also used in biological staining. CV binds to DNA, so it has been used in cell viability assays in biochemistry. However, this binding to DNA will cause replication errors in living tissue, possibly leading to mutations and cancer. Toxicological investigations indicate that crystal violet has carcinogenic and mutagenic effects in rodents [3]. It has also been linked to increased risk of human bladder cancer. Therefore, it is treated as a serious pollutant in

wastewater and difficult to treat by common removal methods such as coagulation and biodegradation, but few studies on its treatment have been attempted [1,4]. The release of wastewater containing the dye poses a dramatic source of water pollution, eutrophication, and perturbation of aquatic life. Therefore, a method is highly required of treating wastewater containing CV.

In the last few decades, much attention has been paid to investigate degradation of organic pollutants mediated by TiO₂ particles in aqueous suspension under UV-light irradiation [5-12]. Recent studies indicate that ZnO can also be used as a promising photocatalyst to degrade such types of organic pollutants under visible/solar irradiation [13-16]. However, artificial UV light and TiO₂ particles tend to be somewhat expensive, and the UV component in sunlight reaching the earth surface and

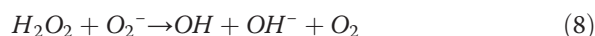
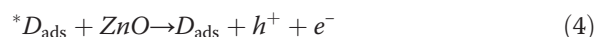
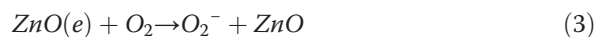
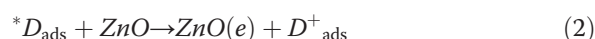
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available to excite TiO₂ is relatively small (ca. 3% to 5%), whereas solar spectrum has about 40% visible region.

Organic pollutants like dyestuffs have the ability to absorb visible light. The electron transfer process from dye to semiconductor, especially ZnO has been found to be very effective. Initially, the dye molecules adsorb on ZnO surface and excited through absorption light. The excited dye (D*) injects an electron to the conduction band of ZnO where it is scavenged by O₂ to form active oxygen radicals. The consequence of electron ejection into the conduction band results hole–electron pair formation. These hole–electron pairs are responsible of generating other reactive oxygen species like ·OH, HO₂, H₂O₂, and also ·O₂⁻. These active radicals are responsible for photodegradation or mineralization of organic compounds. The mechanism can be attributed as follows:



Recently, we reported [13] a comparative study of decolorization of Ponceau S (PS), an anionic dye, in pristine ZnO and prepared ZnO suspensions. The ZnO was prepared through the hydrothermal method. We found that the prepared ZnO (300°C) showed better efficiency to decolorize/degrade Ponceau S. In this report, CV, a cationic dye, has been taken as an example of wastewater pollutants in different industries. Photocatalytic degradation of CV has been reported recently in the presence of TiO₂ [1,8-11] under UV irradiation. So far,

no attempt has been taken to use ZnO instead of TiO₂ as a photocatalyst to decolorize/degrade CV. However, ZnO takes part in photocorrosion under UV radiation at lower solution pH (approximately 6.0). Therefore, the present study focuses on decolorization/degradation of CV in aqueous suspension of ZnO under visible light irradiation with a variety of conditions. In addition, an adsorption study of CV on ZnO is also discussed.

Results and discussion

Adsorption of CV from solution on ZnO

Adsorption studies of CV solution (2.50 × 10⁻⁵ M) with ZnO (0.20 g/40.0 mL) in dark are shown in Figure 1. The results show that the adsorption of CV on ZnO takes about 200 min to reach equilibrium. The equilibrium time at a certain concentration seems to be independent of temperatures that are used for the preparation of ZnO samples. The adsorption data have also been fitted to pseudo-first-order (Lagergren) and the pseudo-second-order kinetic equations of the adsorption processes (data not shown). The values of correlation coefficients (*r*²) for the pseudo-first-order kinetic model are between 0.994 to 0.998 and those for the pseudo-second-order kinetic model between 0.963 to 0.989. These results seem to suggest that adsorption of CV from solution on ZnO favors the pseudo-first-order adsorption model. Results further suggest that the commercial ZnO has been made at a temperature higher than 300°C.

The shapes of adsorption isotherms (30°C) indicate that adsorption of CV from solution on ZnO will reach plateau at its certain concentration as shown in Figure 2. In the range of CV concentration up to 2.50 × 10⁻⁵ M, *C_e/(x/m)_e* vs *C_e* plots are linear (data not shown) which suggests the Langmuir model, and the corresponding correlation coefficient are satisfactory (*r*² = 0.992 to 0.998). However, plots of ln(*x/m*)_e vs ln*C_e* (data not shown) suggest the Freundlich model and show straight lines, but *r*² values vary between 0.967 and 0.984, *r*² = 0.967 for ZnO prepared at 300°C. Therefore, it is suggested that the adsorption pattern of CV on ZnO favors the Langmuir model that also was observed for Ponceau S, an anionic dye, on ZnO [13].

The kinetic data (*k*) and Langmuir parameters (*Q_m* and *K_L*) (Table 1) are likely to suggest that increasing the heating temperatures during the preparation of ZnO samples to higher values causes agglomeration of ZnO particles, decreasing thereby the active adsorption sites. Accordingly, adsorption of CV on ZnO falls as the temperature of heating is raised during the preparation of ZnO samples. The amount of CV adsorbed (25% ([CV] = 2.50 × 10⁻⁵ M, Temp. = 30°C, pH = 6, ZnO = 0.20 g/ 40.0 mL), *Q_m* = 0.31 mg/g) at a particular time

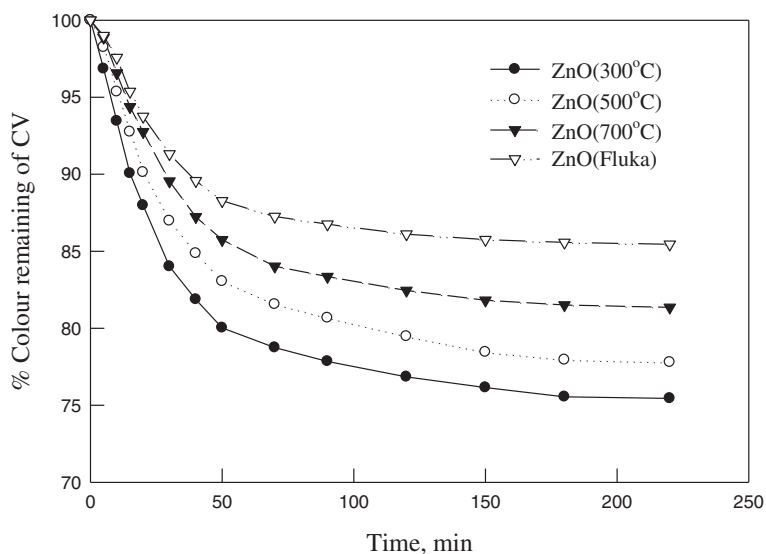


Figure 1 Absorption of CV from solution by ZnO. [CV] = 2.50×10^{-5} M, ZnO = 0.20 g/ 40.0 mL, pH = 6, Temp. = 30°C.

and concentration is the highest in the case of ZnO prepared at 300°C.

Adsorption of a dye on a semiconducting metal oxide surface, as on the surface of ZnO, is a prerequisite for the successful photodecolorization of the dye. Consequently, the adsorption studies were necessary.

The better adsorption characteristics of the CV from solution on ZnO made at 300°C, were the criteria for selecting this ZnO sample for studying the visible light-influenced degradation. The higher value of K_{L-H} compared to K_L (Table 1) suggests that there is some surface

activation by the visible light used in this study. This activation should be considerably low to promote valence band electrons to the conduction band as the band gap energy for ZnO is 3.2 eV ($\lambda_{max} \approx 388$ nm).

ZnO-mediated photodecolorization CV

The initial pH, in the range 3.75 to 6.02, of CV solutions did not affect on its UV-vis spectra. The spectrum of the CV solution before illumination with visible light (Figure 3) has characteristic prominent peaks at 590, 302, and 250 nm corresponding to conjugated triphenyl

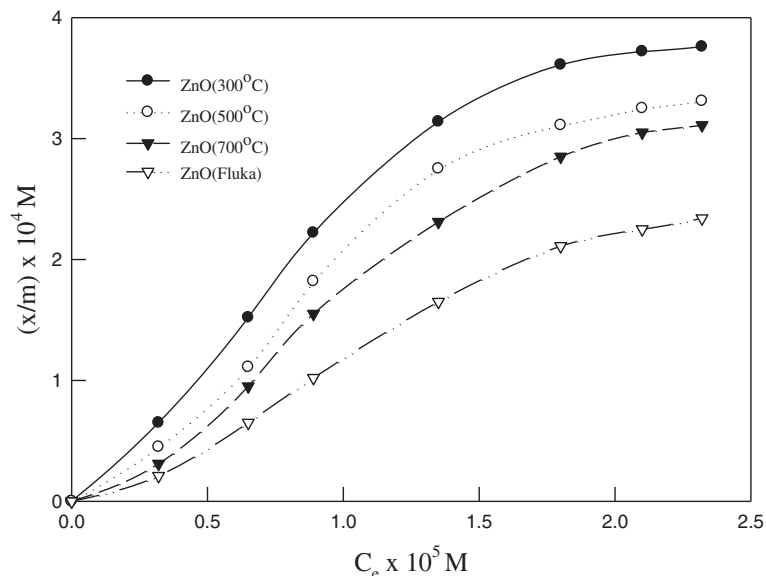


Figure 2 Adsorption isotherms of CV. ZnO = 0.20 g/ 40.0 mL, pH = 6, Temp. = 30°C.

Table 1 The kinetic and equilibrium parameters for adsorption of CV from solution on ZnO

ZnO	Rate constant		R_L	Equilibrium constant	
	$k_1 \times 10^2 / \text{min}$	$k_2 \times 10^3 / \text{g/mg/min}$		$K_L \times 10^{-3} / \text{L/mol}$	$Q_m / \text{mg/g}$
300°C	4.13	9.72		4.16	0.31
	$r^2 = 0.999$	$r^2 = 0.981$		$r^2 = 0.999$	
500°C	3.73	5.07		4.00	0.26
	$r^2 = 0.998$	$r^2 = 0.980$	0.91	$r^2 = 0.990$	
700°C	3.35	2.15		3.92	0.21
	$r^2 = 0.994$	$r^2 = 0.978$		$r^2 = 0.989$	
Commercial	2.77	0.98		3.83	0.16
	$r^2 = 0.995$	$r^2 = 0.988$		$r^2 = 0.985$	

Temp. = 30°C, pH = 6.02.

methane chromophore, diphenyl methyl type, $\phi_2C = (\phi, -C_6H_5)$, and benzene moieties, respectively. The decrease in absorbance at 590 nm with illumination is due to the breakdown of the chromophore responsible for the characteristic color of CV. This breakdown, caused by the visible light illumination of CV in the aqueous suspension of ZnO particles, leads to the formation of intermediates like benzophenone and benzene derivatives [17]. Some of the benzene derivatives are benzoic acid and aniline derivatives. All of these compounds are finally mineralized to CO_2 , H_2O , and NO_3^- . The hypsochromic shift of the peak at 590 nm of the chromophore to about 575 nm is of particular significance as it indicates N-dimethylation process [18] that ultimately generates NO_3^- ions [19,20].

Figure 3 shows that the characteristic peaks gradually disappear with the time of illumination. When the illumination was continued for about 360 min ($[CV] = 2.00 \times$

10^{-5} M), none of the peaks could be detected. The residual spectrum with a peak near about 200 nm disappears when very dilute solution of $NaNO_3$ is used as reference in the place of water. This suggested the presence of NO_3^- ions, as one of the products formed during visible light degradation of CV in the ZnO suspension.

Figure 4 shows how decolorization and disappearance of total organic carbon (TOC) progress with time of illumination of CV in ZnO suspension. The decrease of TOC with time of illumination is the actual mineralization of CV, that is, the conversion of CV molecules to CO_2 , H_2O , N_2 , and NO_3^- ions. TOC measures CO_2 formed from the carbon atoms of CV molecules. Accordingly, TOC measures complete degradation of CV molecules. With $ZnO = 0.20 \text{ g/40 mL}$ and $[CV] = 2.00 \times 10^{-5} \text{ M}$, it needs about 360 min for complete mineralization of CV molecules in the suspension. On the other hand, for complete decolorization, it takes about 200 min; therefore, it is concluded that decolorization is faster than degradation.

Influence of ZnO on decolorization of CV

The initial rate of decolorization of CV also increases with the increasing of the concentration of ZnO in the suspension (data not shown). However, this increase seems to be linear up to about 0.15 g ZnO/40 mL. Subsequently, deceleration of the initial rate of decolorization of CV starts. In our previous studies, it has also been reported that the initial rate of decolorization of an anionic dye, Ponceau S, increases with increasing of ZnO in suspension [13].

Figure 5 shows that after 5 min, about 28% decolorization takes place with 0.20 g ZnO. This decolorization is about 35% after 5 min when 0.40 g ZnO is used. Moreover, with 0.20 g ZnO, decolorization increases to

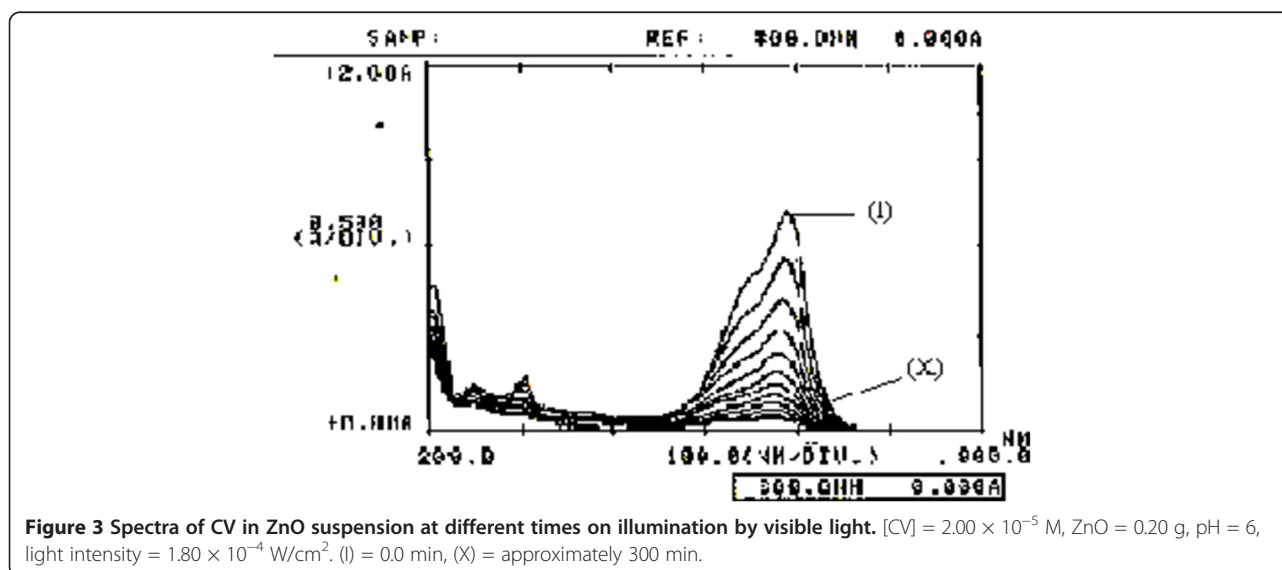


Figure 3 Spectra of CV in ZnO suspension at different times on illumination by visible light. $[CV] = 2.00 \times 10^{-5} \text{ M}$, $ZnO = 0.20 \text{ g}$, $pH = 6$, light intensity = $1.80 \times 10^{-4} \text{ W/cm}^2$. (I) = 0.0 min, (X) = approximately 300 min.

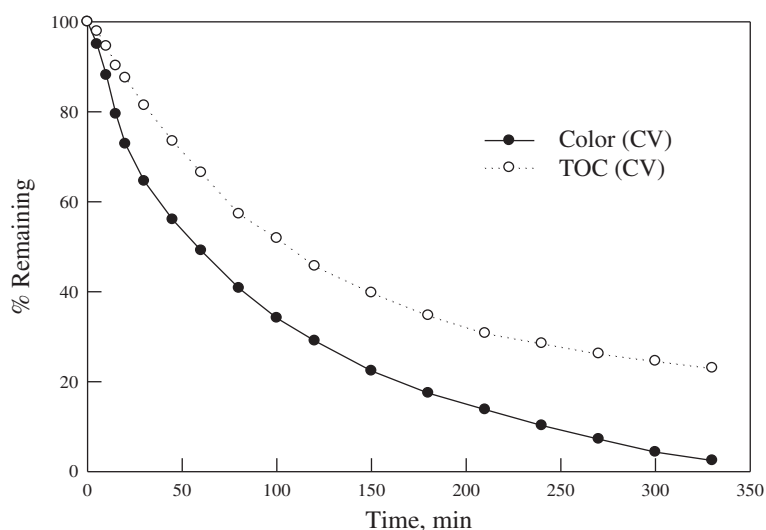


Figure 4 Disappearance of color and TOC of CV in ZnO suspension with time of illumination. [CV] = 2.00×10^{-5} M, ZnO = 0.20 g/40 mL, pH = 6.

about 43% after 10 min, 51% after 15 min, and about 62% after illumination for 30 min. Respectively, under identical times of illumination, with 0.40 g ZnO, about 46%, 55%, and 64%, decolorization takes place. These results suggest that increasing the time of illumination is much useful than increasing the concentration of ZnO in the suspension. The use of higher concentration, along with longer duration of illumination, does not appear to be that profitable (Table 2). For an example, after 60 min of illumination, there is about 5% enhancement

of decolorization when 0.40 g ZnO/40 mL is used instead of 0.20 g ZnO/40 mL (Table 2). The results in Table 2 further show that like PS [13], the increase in turbidity of the suspension, due to increased concentration of ZnO, does not affect the decolorization that much. The reason for this may be ascribed to the continuous stirring of the suspensions in the open air. This replaces ZnO particles with adsorbed dyes at the surface, exposed to the visible light and having the dye molecules decolorized, by fresh particles with adsorbed dyes from the bulk. The speed

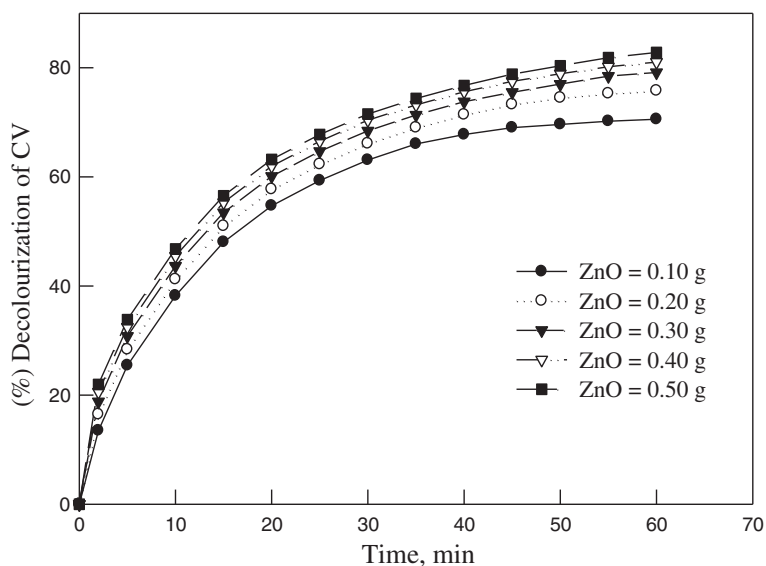


Figure 5 Influence of ZnO concentration on visible light decolorization of CV in its suspension. [CV] = 2.00×10^{-5} M, pH = 6, suspension = 40 mL.

Table 2 Influence of various parameters on visible light decolorization of CV in ZnO suspensions (Temp 30°C)

[CV] × 10 ⁵ M	ZnO g/40 mL	pH	Initial rate, R _i × 10 ⁶ mol/L/min	Decolorization after 60 min (%)	K _{L-H} × 10 ⁻⁴ L/mol	k _{i-H} × 10 ⁶ mol/ L/min
2.00	0.10	6.02	0.76	70.57		
			1.02	75.81		
			1.09	79.13		
			1.15	81.03		
			1.19	82.82		
0.50	0.20	6.02	0.41	93.83	0.77	2.37
1.00		0.69	86.79			
1.50		0.88	80.82			
2.00		1.02	75.81			
2.50		1.24	71.80			
2.00	0.20	3.75	1.02	76.30	r ² = 0.998	r ² = 0.998
		4.22	1.03	75.21		
		4.88	1.03	76.52		
		5.35	1.02	75.39		
		6.02	1.02	75.81		

of agitation may have been sufficiently high to help overcome the retarding effect of the higher ZnO concentration on the movement of the bulk particles to the surface.

Thus, the excess surface sites, provided by the increase in concentration of ZnO, for adsorption of fixed and a small number of CV molecules should have been useful [21]. There had been no loss of these surface sites due to aggregation when ZnO concentration increases. This loss of surface sites could be one of the dominant factors [20] that are responsible for the deceleration of the initial rate of CV decolorization with the increase in ZnO concentration in the suspension.

Various parameters related to CV decolorization (Table 2) are somewhat lower in comparison to those of PS, an anionic dye [13]. One of the inherent causes is likely to be that CV has adsorption parameters lower than those of PS.

Influence of concentration of CV

The trend in the decolorization of CV solution with its higher concentrations (Figure 6) seems to be similar to that with increasing amounts of ZnO (Figure 5), and the situation arises because of a small number of adsorption sites on ZnO in comparison to the increasingly higher

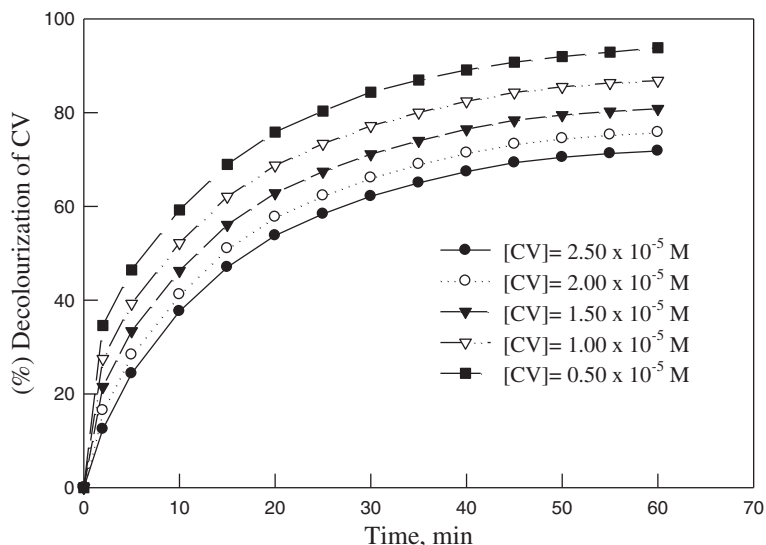


Figure 6 Influence of concentration of CV on its visible light decolorization in ZnO suspension. ZnO = 0.20 g/40 mL, pH = 6.

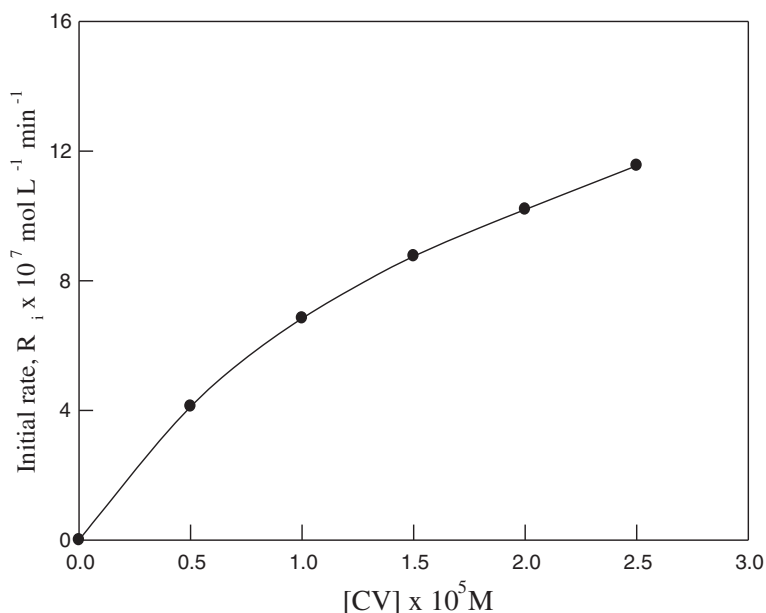


Figure 7 Influence of the concentration of CV on its initial rate of decolorization in ZnO suspension. ZnO = 0.20 g/40 mL, pH = 6.

number of CV molecules. The initial rate of decolorization of CV in solution by visible light in the presence of ZnO seems to remain linear up to the concentration of CV much less than 1.0×10^{-5} M (Figure 7). As like other dyes, inner filter effect [13,22,23] is also quite prominent in the case of CV as its concentration in solution increases. The data of variation of initial CV concentrations, $[CV]_i$, and initial rates, R_i , of disappearance of CV color from ZnO suspension, were applied to the Langmuir-Hinshelwood equation in the following form:

$$\frac{1}{R_i} = \frac{1}{k_{LH}} + \frac{1}{k_{LH}K_{LH}[CV]_i}$$

k_{LH} is a proportionality constant and provides a measure of intrinsic reactivity of CV adsorbed on ZnO. On the whole, k_{LH} offers an idea of the apparent rate constant of the reaction involving adsorbed CV with oxidizing radicals formed at the ZnO surface. It is the zero-order surface reaction constant [24]. K_{LH} is the apparent adsorption equilibrium constant (so called Langmuir-

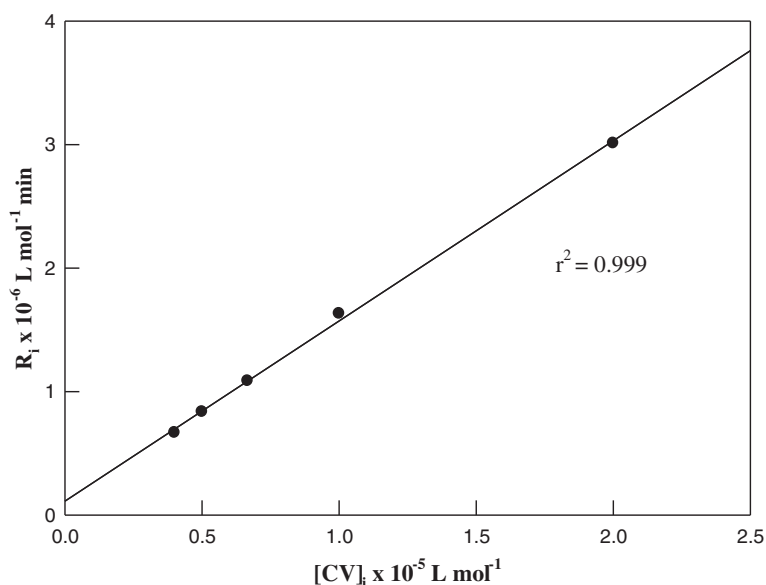


Figure 8 Langmuir-Hinshelwood plot of visible light decolorization of CV in ZnO suspension. ZnO = 0.20 g/40 mL, pH = 6, Temp. = 30°C.

Hinshelwood constant). R_i^{-1} vs $[CV]_i^{-1}$ is a nicely fitted straight line (Figure 8). K_{LH} and k_{LH} values are 7.70×10^3 L/mol and 2.37×10^{-6} mol/min, respectively (Table 1). The higher K_{LH} value, compared to that of K_L (4.16×10^3 L/mol; Table 1), suggests some surface activation of ZnO surface by visible light.

Conclusions

Adsorption of CV on ZnO aqueous suspension follows the pseudo-first-order kinetic model (Lagergren) while the adsorption pattern follows the Langmuir model; it is a prerequisite condition for semiconductor metal oxide-mediated photodegradation. Decolorization results show that ZnO can efficiently decolorize/degrade CV in the aqueous suspension under visible light irradiation. ZnO prepared at 300°C shows superior photocatalytic activity compared to the ZnO samples treated at 500°C and 700°C and also to the pristine ZnO. The prepared ZnO has been found to be quite stable under visible light illumination at a pH of approximately 6, and there is some activation of the surfaces of ZnO particles by this light. In addition, remediation of industrial effluents containing some azo dyes by the prepared ZnO under visible light is underway.

Methods

Materials

The dye CV, also known as gentian violet 10B, was obtained from local dye suppliers. The dye shows an absorption maximum at 590 nm. The structure of the dye is shown in Figure 9. Pristine zinc oxide (>99.9% purity,

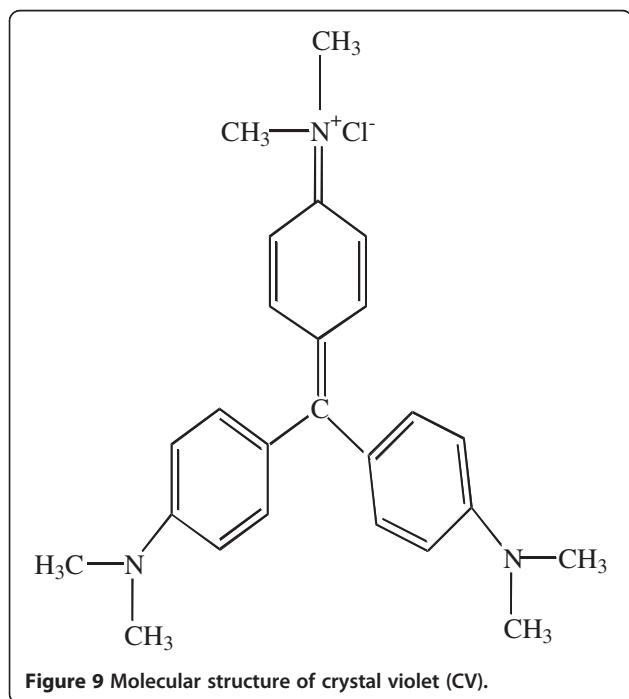


Figure 9 Molecular structure of crystal violet (CV).

MZ-500 with a surface area of 38.4 m²/g) was from Tayca Corporation, Japan. Zinc sulfate, ammonium carbonate, sodium hydroxide, and hydrochloric acid were purchased from BDH (Dubai, United Arab Emirates). All the chemicals were used without further purification. The pH of the solutions was adjusted using dilute solutions of HCl or NaOH. Deionized water was used throughout the experiment.

ZnO was prepared through hydrothermal method. The prepared ZnO was characterized using a scanning electron microscope (SEM; model S-3400N, Hitachi, Tokyo, Japan), X-ray diffraction (XRD; RINT 2200, Rigaku Corporation, Tokyo, Japan) and laser-induced breakdown spectroscopy (model LAB-170, pulsed Nd:YAG, Spectra-Physics Inc., Santa Clara, CA, USA) measurement. Preparation procedure has been described in detail in our previous study [13]. Briefly, ZnCO₃ was collected as a precipitate upon the addition of ammonium carbonate solution into zinc sulfate solution at 60°C. The precipitate was filtered and washed with water to remove sulfate ions and dry it at 105°C in an oven. The dry ZnCO₃ was further heated in a muffle furnace (OSK, Isuzu Seisa kusu Co., Ltd., Japan) at different temperatures to get desired thermally treated ZnO samples.

Adsorption studies

ZnO portions of 0.20 g were soaked overnight in water in stoppered bottles. The volume was made up to 40 mL by adding water and CV solution so that the desired concentration and an initial pH, approximately pH 6, of the dye solution were maintained. The bottles with their contents were shaken in a thermostat shaker (NTS-4000AL, Tokyo Rika Kikai Co., Ltd, Tokyo, Japan) at 30°C. Samples were withdrawn at definite intervals of time and centrifuged, and their dye concentrations were determined by measuring absorbance of the clear solution at 590 nm (UV-visible spectrophotometer, UV-160A, Shimadzu Corporation, Kyoto, Japan).

Procedure of decolorization

An experimental setup for photodecolorization of CV has been reported in our previous study [13,14]. Briefly, the visible light illumination source comprised two 40-W fluorescent lamps, each with a length of 20 cm, placed side by side. The lamp house was a wooden box with the internal surface covered with aluminum foil. The temperature inside the box was maintained at 30°C by regulated flow of cool air through it. The air entered the box at the bottom and flowed out at the top. The approximate intensity of the visible light at the position of the reactor was approximately 1.8×10^{-4} W/cm² as

measured using a spectroradiometer (model IL-588, International Light Technologies, Peabody, MA, USA).

Fifty-milliliter beakers, with an internal diameter of 4.0 cm, were used as reactors. Each reactor was placed on the magnetic stirrer, and the distance of the solution surface from the lower part of the lamps was kept fixed.

ZnO portions of 0.20 g were taken in each of the several reactors containing 10 mL of water each. They were left overnight, and CV solutions of desired concentrations were added to the reactors in each to reach the total volume of 40.0 mL. The contents of the reactors were agitated ultrasonically for 5 min prior to subjecting them to visible light illumination for definite times. The weight of each reactor was recorded. During the illumination, the suspensions were continuously stirred magnetically while the air passed through the lamp house. After illumination, the weights of the reactors were checked, and the weights were made up by the addition of water. Clear solutions, obtained after centrifugation, were analyzed spectrophotometrically at 590 nm to find out the concentration of the remaining CV in each reactor. The clear solutions (0 dye concentrations) were analyzed for free Zn^{2+} ions by AAS (Shimadzu, Kyoto, Japan) with reference to the standard $ZnSO_4$ solution.

Percent decolorization and mineralization

Decolorization or color removal of CV was calculated as $(X_t/X_o) \times 100$, where X_t is the absorbance at time 't' and X_o is that at $t = 0$. On the other hand, the percent color remaining was $[(X_o - X_t)/X_o] \times 100$.

In the case of mineralization/degradation, X_o is the TOC (ppm) of CV at $t = 0$, and X_t (ppm) is its TOC at time 't'. The total organic carbon analyzer (CPH/CPN and 638 - 4241C, Shimadzu) was used for this purpose.

The initial rate of decolorization

The equation $R_i = k[CV]_i$ was used to determine the initial rate (R_i) by multiplying the apparent first-order rate constant k with the initial concentration of CV, $[CV]_i$. k was determined from the slope of the plot $\ln([CV]_i/[CV]_0)$ vs time.

Competing interests

The authors declare that they have no competing interests.

Authors' contributions

MM is a PhD student and his work is the basis of the paper. AJM and TSAI are the supervisor and co-supervisor, respectively. MAH, MTS, MNI and IMII have helped with the instrumental analysis and final preparation of the paper. All the authors read and approved the final version of the manuscript.

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