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Zinc oxide-mediated photocatalytic decolorization of Ponceau S in aqueous suspension by visible light

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Abstract

ZnO, comprising nanosize particles (approximately 40 nm) has been prepared by heating (300°C) ZnCO₃, which was obtained as precipitate by mixing ZnSO₄ and (NH₄)₂CO₃ solutions. The prepared ZnO was characterized by X-ray diffraction, scanning electron microscopy (SEM), laser-induced breakdown spectroscopy, and adsorption studies. It has been used to catalyze the decolorization of Ponceau S (PS), a model diazo dye, in an aqueous suspension under visible light ($I \approx 1.8 \times 10^{-4} \text{ W cm}^{-2}$). This ZnO was found to be more efficient as a photocatalyst compared to pristine ZnO. ZnO samples with higher temperatures (500°C and 700°C) show less catalytic activity. SEM images show that the particle size of ZnO increases with the increase in calcined temperature of ZnO through agglomeration, resulting in a decrease in surface area. Photodecolorization of PS is affected by its and ZnO concentrations, but unaffected by the initial pH of the solutions in the range of 4 to 7. Illumination for a sufficiently long time completely mineralizes the dye, but no Zn²⁺ can be detected in the clear solution. Photodegradation kinetics in the ZnO suspension obeys the Langmuir-Hinshelwood equation, and some activation of the ZnO surface by light is indicated.

Keywords: Nanosize ZnO, Ponceau S (PS) dye, Adsorption, Photodecolorization, Mineralization, Inner filter effect, Langmuir-hinshelwood equation

Background

A substantial amount of dyestuff is lost during the dyeing process in the textile/dyeing industries. The discharged wastewater is highly colored and is of complex and variable nature [1]. This wastewater has been a long-standing concern, and different methods of removal of the color are still under trial. After the successful demonstration of the photocatalytic ability of titania (TiO₂) [2], studies on its use for removal of pollutants, particularly dyes, from wastewater attracted wide attention. The source of light has been mostly ultraviolet (UV). The recent studies on various parameters to make TiO₂ as an effective photocatalyst for this purpose, even with solar light, have been reviewed [3-6].

Zinc oxide (ZnO), with comparable bandgap energy as that of TiO₂ (rutile), has hardly drawn as much attention

as a photocatalyst. The reluctance to use ZnO probably originates from a study of the effect of UV illumination of its aqueous slurry [7]. ZnO dissolves forming Zn²⁺(aq) and simultaneously produces H₂O₂ in the presence of air. On the other hand, TiO₂ is photochemically stable. However, one of the early studies of ZnO as a photocatalyst shows that it has high reaction and mineralization rates [8] because of its more efficient hydroxyl ion generation efficiency [9]. Subsequent studies discover that ZnO absorbs a large fraction of the solar spectrum and more light quanta than TiO₂ [10-14]. These studies have shown that ZnO acts as a better photocatalyst than TiO₂ under the given conditions. ZnO has been found to satisfactorily degrade an azo dye, brilliant orange [11], and a non-azo dye, Bractive T Blue [12], from aqueous suspensions at the normal pH, approximately 6, of the dyes by visible light with an intensity of approximately 0.18 mW cm⁻². Many more examples of the use of ZnO as a photocatalyst are available

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in the literatures [15-17]. One of the examples is the study showing that ZnO can be used as an alternative photocatalyst to TiO₂ [17].

The recent trend in the study of ZnO photocatalysis is predominantly based on nanosize ZnO and its amenability of structural modification for improved visible light absorption [18]. ZnO nanocrystals have been investigated in terms of operational parameters and kinetics of photocatalytic degradation [19]. A photocatalytic paper using zinc oxide nanorods has also been developed [20].

Photocatalysis using suspended particles to remove contaminants in aqueous systems is an efficient process, but the subsequent removal of the particles from the systems adds extra process steps. In this regard, use of supports for the catalysts is found to be helpful. However, there is an inverse relationship between the adhesion of a catalyst to a support and its photocatalytic activity [21].

Ponceau S (PS), a diazo dye, is used in the textile dyeing industry and also in coloring leather and paper [22]. It is used in clinical laboratories as a protein-binding dye for staining blood serum proteins [23]. Its biotransformation products have toxic effects on aquatic organisms, and it is a suspect as a carcinogen for the human body [24]. Some methods of its decolorization/degradation are reported [22,25-27]. Most of these methods need further development in terms of time and cost.

In the present study, nanosize ZnO powder has been prepared and used as a photocatalyst to decolorize/degrade PS in the presence of visible light from fluorescent lamps. Suspended nanosized ZnO particles in the aqueous medium have been preferred to make the process simple to operate. The photodecolorization pattern of PS in this process is expected to be the same as that which will be obtained by a previously made nanosize ZnO attached to a suitable support.

Methods

Materials

PS, acid red 112 (C.I. 27195), was obtained from local dye suppliers. The structure of the dye is shown in Figure 1.

Its absorption maximum, λ_{\max} , at 520 nm in the visible range is quite sharp ($2.8 \times 10^4 \text{ M}^{-1} \text{ cm}^{-1}$ at 30°C).

Pristine zinc oxide (>99.9% purity, MZ-500 with a surface area of 38.4 m²/g) is 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 to values between 4 and 7 using dilute solutions of HCl or NaOH. Deionized water was used throughout the experiment.

Preparation of zinc oxide

ZnCO₃ was precipitated from 10% zinc sulfate solution at 60°C by ammonium carbonate solution. The precipitate was filtered and washed with water to remove sulfate ions. The dry (at approximately 105°C) ZnCO₃ was heated at 300°C in a muffle furnace (OSK, Isuzu Seisa kusu Co., Ltd., Japan) until being in constant weight. Portions of this ZnO were further thermally treated separately at 500°C and 700°C for 1 h.

Characterization of the prepared ZnO

X-ray diffraction (XRD) was done using RINT 2200 (Rigaku Corporation, Tokyo, Japan). Some ideas about the particle sizes of ZnO samples were obtained from scanning electron microscope (SEM; model S-3400N, Hitachi, Tokyo, Japan) images. For further characterization of ZnO, a Spectra-Physics laser source (model LAB-170, pulsed Nd:YAG, Spectra-Physics Inc., Santa Clara, CA, USA) was used to get laser-induced breakdown (LIB) spectra of the samples. The SpectraPro 2758 spectrometer (Spectra-Physics Inc.) and Princeton Instruments PIXIS 100B CCD detector (Trenton, NJ, USA) were used.

Nanosize ZnO was also characterized by 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 PS solution so that the desired concentration and an initial pH, approximately pH 6, of the dye solution were maintained. The bottles

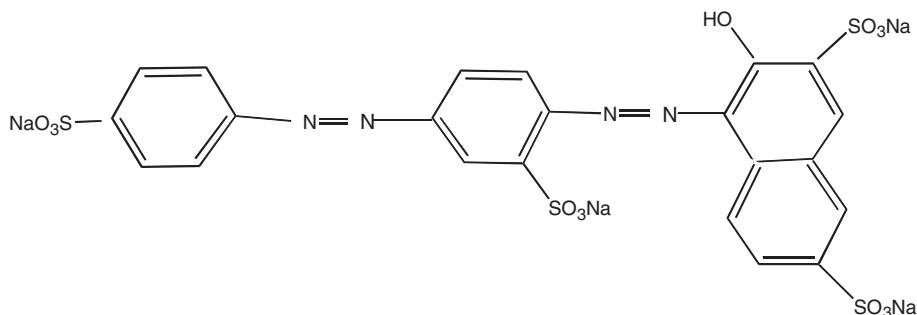


Figure 1 Structure of Ponceau S (PS).

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 520 nm (UV-Visible spectrophotometer, UV-160A, Shimadzu Corporation, Kyoto, Japan).

Decolorization experiment

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 $1.8 \times 10^{-4} \text{ W cm}^{-2}$ as measured by 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 PS solutions of desired concentrations were added to the reactors to make the total volume in each to 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 520 nm to find out the concentration of the remaining PS in each reactor. The clear solutions (0 dye concentrations) were analyzed for free Zn^{2+} ions by AAS (Shimadzu) with reference to the standard ZnSO_4 solution.

Percent decolorization and mineralization

Decolorization or color removal of PS 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 total organic carbon (TOC, ppm) of PS 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 $R_i = k[\text{PS}]_i$ was used to determine the initial rate (R_i) by multiplying the apparent first-order rate constant k with the initial concentration of PS, $[\text{PS}]_i$. k was determined from the slope of the plot $\ln([\text{PS}]_i/[\text{PS}]_t)$ vs time.

Results and discussion

Characterization of ZnO

Figure 2 shows the XRD pattern of the prepared ZnO and commercially available ZnO (Tayca Corporation, Osaka, Japan). Results show that the prepared ZnO are crystalline and identical with wurtzite ZnO, and ZnO at 300°C contains a minor amount of $\text{Zn}(\text{OH})_2$.

Figure 3 shows the SEM images of thermally treated ZnO powders. ZnO particles made at 300°C are of approximately 40 nm in sizes. Agglomeration of the particles of ZnO made at 300°C to larger sizes results from heating.

Adsorption of PS on the prepared nanosize ZnO particles from aqueous suspension reveals some of their additional characteristics. The Langmuir equation for adsorption in the present system at equilibrium is expressed here as follows:

$$\frac{C_e}{x/m} = \frac{1}{K_L Q_m} + \frac{C_e}{Q_m},$$

where K_L is the Langmuir constant (L/mg) or adsorption constant. The ZnO-PS system obeys the Langmuir isotherm as the plots of $C_e/(x/m)$ vs C_e are straight lines in all cases. Q_m and K_L values were computed from the slopes and intercepts, respectively, of the straight lines. The maximum amount of adsorption of PS corresponding to the complete monolayer formation on ZnO particles is represented by Q_m (mg/g), and it is also known as the Langmuir constant related to the capacity of adsorption. C_e (mol/L, M) is the equilibrium concentration of PS in the suspension, and x/m (mg/g) is the amount of PS

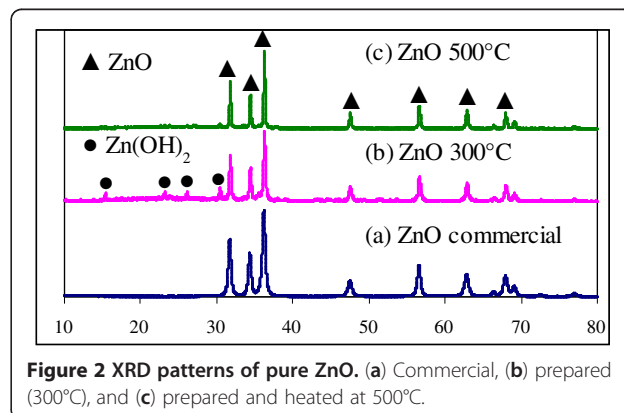


Figure 2 XRD patterns of pure ZnO. (a) Commercial, (b) prepared (300°C), and (c) prepared and heated at 500°C.

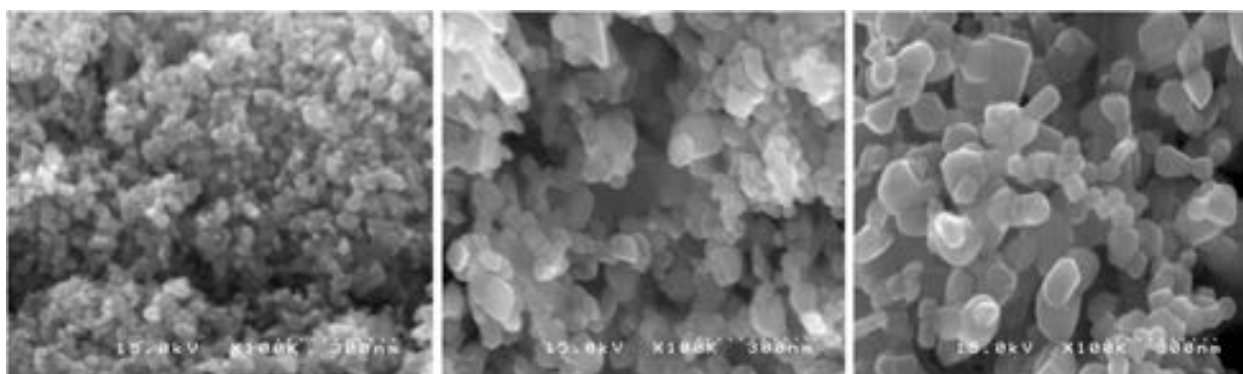


Figure 3 SEM images of ZnO (a) prepared (300°C), (b) heated at 500°C and (c) at 700°C.

adsorbed per gram ZnO, at equilibrium, for its different initial concentrations.

PS molecules from the aqueous suspension, with $[PS]_i = 8.50 \times 10^{-5}$ M, $ZnO = 0.20$ g/40 mL, $pH = 6.02$, and $temp = 30^\circ C$, take about 100 min for their adsorption on ZnO (300°C) particles to reach equilibrium. The equilibrium times, under these conditions, are longer for other ZnO samples. Q_m is the highest in the case of the prepared ZnO (300°C; Table 1). This is due to large surface area of the nanosize ZnO at 300°C. Previous studies also show that a larger surface area provides more surface active sites for the adsorption of the dye molecules resulting in efficient photocatalytic activity [28-30].

LIB spectra (Figure 4) of both the commercial and prepared ZnO (300°C) show three peaks with different intensities (in au). The peak positions as well as intensities of the commercial ZnO are very much identical to those of the prepared sample.

On the basis of the above results, the prepared nanosized ZnO (300°C) has been chosen for studying the photocatalytic decolorization of PS.

ZnO-mediated visible light decolorization of PS

No detectable decolorization or degradation of PS was observed under the longest illumination by the light source in the absence of ZnO. About 30% decolorization of the dye from the aqueous suspension took place by adsorption on the ZnO surface under the specified conditions

Table 1 Equilibrium parameters for adsorption of PS on ZnO from suspensions $pH = 6.02$, $temp = 30^\circ C$

| ZnO | Equilibrium parameters | |
|------------------|---|--------------|
| | $K_L \times 10^{-3}$ (L mol ⁻¹) | Q_m (mg/g) |
| Prepared (300°C) | 8.23 $r^2 = 0.995$ | 1.18 |
| Heated at 500°C | 8.01 $r^2 = 0.996$ | 0.64 |
| Heated at 700°C | 7.86 $r^2 = 0.995$ | 0.57 |
| Commercial | 7.44 $r^2 = 0.990$ | 0.44 |

(Figure 5). However, visible light decolorization is much faster as it accompanies degradation. About 35% Yellow direct 12 has also been reported [31] to be decolorized by ZnO in the dark under similar experimental conditions. Visible light generates reactive oxygen species (e.g., $\cdot OH$, O_2^-) on the surface of ZnO in the aerated aqueous suspension containing the dye, and these species are responsible for the degradation of PS molecules [32,33]. Of these species, $\cdot OH$ is the most prominent and most active.

Effect of solution pH on decolorization

It is to be noted that the initial pH of the PS solution in the range of 4 to 7 did not affect its photodecolorization. This is because the zero point charge of ZnO (made at 300°C) in the aqueous medium is 8.9. PS is an anionic dye, and the surface of the nanosized ZnO particles carries predominantly a positive charge up to the solution pH of <8.9. The initial pH in most of the present studies has been fixed at a pH of approximately 6. After complete decolorization, the pH of the clear solution has been found to decrease (≥ 4).

In a previous study, decolorization of brilliant golden yellow in ZnO suspension has also been found to decrease [14].

Decolorization mechanism

The spectrum of the PS solution (Figure 6) has characteristic peaks at 520, 345, 306, and 235 nm corresponding to the chromophore containing two azo linkages, a naphthalene moiety, benzene rings, and benzene and naphthalene rings, respectively. The decrease in absorbance, hence decolorization, of the solution at 520 nm with visible light illumination of PS in the ZnO suspension is due to the breakdown of the chromophore which is responsible for the characteristic solution color.

The -N=N- bonds of the chromospheres of PS molecules are the most susceptible sites for oxidative attack [34-36]. Figure 6 shows that the characteristic peaks gradually

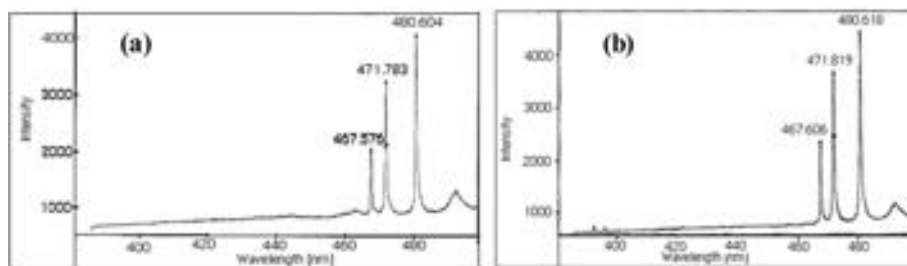


Figure 4 LIB spectra of ZnO samples. (a) ZnO (commercial) and (b) ZnO prepared (300°C).

disappear with the time of illumination. When the illumination is continued for sufficiently long time, none of the peaks can be detected. The residual peak in the spectrum, observed when water was used as reference, near about 200 nm disappeared when the very dilute solution of Na_2SO_4 was used as reference in the place of water. This suggests the presence of SO_4^{2-} ions, one of the products formed during degradation of PS in the ZnO suspension.

PS molecules are adsorbed on ZnO predominantly through their negatively charged $-\text{SO}_3^-$ ends. The molecules of this dye, like some dye molecules containing $-\text{SO}_3\text{Na}$ groups in their structures [37,38], release SO_4^{2-} ions as one of the initial products of mineralization during photocatalytic degradation. CO_2 is unlikely to be produced until some simple carboxylic acid species are available for the photo-Kolbe reaction [39]. When the azo bonds are ruptured, the naphthalene and benzene moieties are expected to be separated releasing some SO_4^{2-} ions in the suspension. These species need to be remained adsorbed on the ZnO surface to be further oxidized during photolysis to the precursors of CO_2 , and

the subsequent processes lead to the formation of CO_2 from them. Accordingly, mineralization of PS molecules in the ZnO suspension, indicated by the formation of CO_2 , is much slower than its decolorization.

Figure 7 shows how decolorization and disappearance of TOC progress with time of visible light illumination of PS in the ZnO suspension. The decrease of TOC with time of illumination is the actual measure of mineralization of PS, that is, its conversion into CO_2 , H_2O , N_2 , SO_4^{2-} , and Na^+ ions.

With 0.20 g ZnO in 40 mL of 5.00×10^{-5} M PS solution, it needs visible light illumination for about 300 min for its complete decolorization (Figure 7). Photodecolorization of PS is faster than its photodegradation. Obviously, the photodecolorization of PS is related to its degradation. Accordingly, studies on photodecolorization gave a measure of the photodegradation of PS.

Generation of active species

The principal agent for degradation of dyes by light in the presence of photocatalysts is the $\cdot\text{OH}$ radical. This radical, along with others, is expected to form by visible light illumination of the ZnO suspension containing PS through sequences of steps, some of which are likely to be as follows:

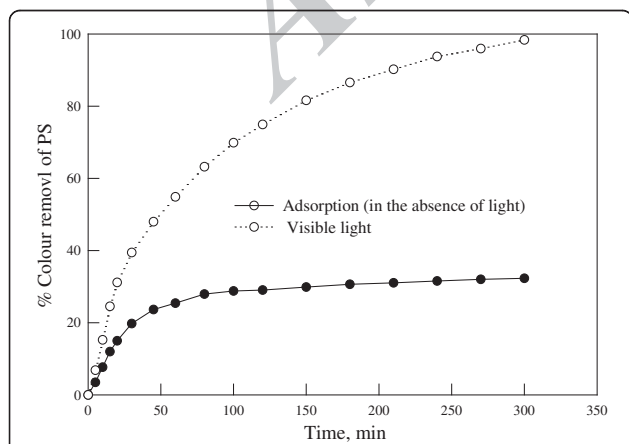


Figure 5 Decolorization of PS in ZnO suspension with time. [PS] = 5.00×10^{-5} M, pH = 6.02, ZnO = 0.20 g/40 mL, and light intensity $\approx 1.8 \times 10^{-4}$ W cm^{-2} .

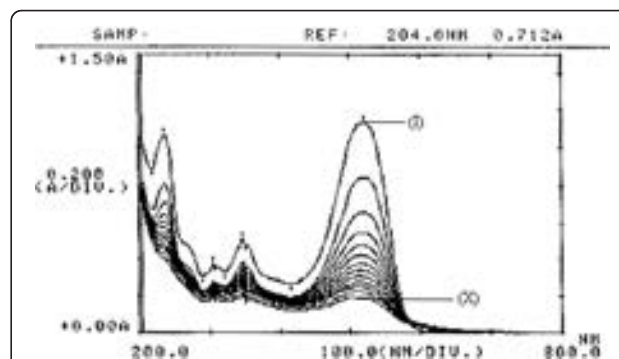
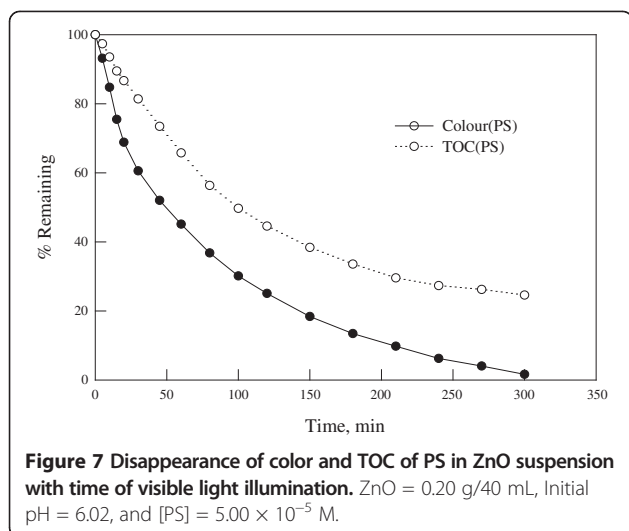


Figure 6 Spectra of PS in ZnO suspension after illumination with visible light. [PS] = 4.00×10^{-5} M, initial pH = 6.02, ZnO = 0.20 g/40 mL, (l) = 0.0 min, and (X) = 250 min.



The first step is the excitation of the dye (PS) adsorbed on the ZnO surface, D_{ads} ,



* D_{ads} stands for an excited PS molecule which is adsorbed on ZnO.

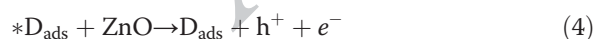
The excited molecule on the ZnO surface injects an electron to its conduction band.



The trapped electron, $ZnO(e)$, converts O_2 adsorbed on the surface into O_2^- ,



The excited dye can also create an electron (e^-)-hole (h^+) pair in ZnO.



This hole can react with a water molecule adsorbed on the ZnO surface to produce the $\cdot OH$ radical.



Another route of generation of $\cdot OH$ can be through the formation $HO_2\cdot$, which leads to formation of H_2O_2 .



The H_2O_2 adsorbed on ZnO can also lead to the formation of $\cdot OH$.



Accordingly, the overall degradation of PS molecules in the suspension of ZnO by visible light can be summarized as follows:

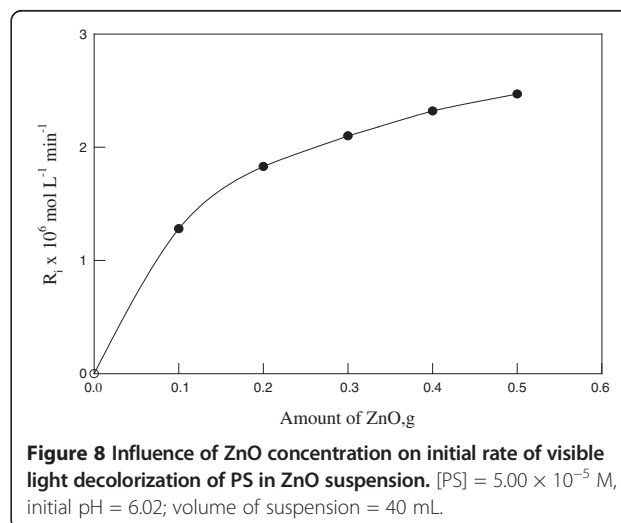


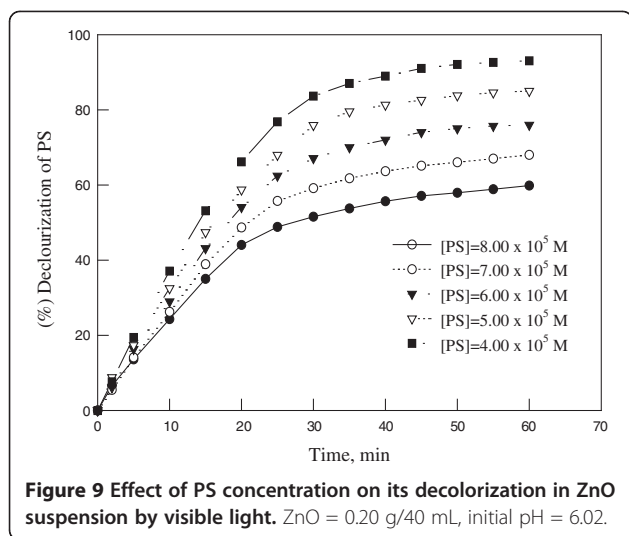
The step 11, involving $\cdot OH$, is likely to be the most favorable. However, this does not exclude the possibility of the oxidative attack at this stage by other species, such as dissolved oxygen, $HO_2\cdot$, and radicals that are subsequently formed from intermediates. The azo linkages, $-N=N-$, of PS are, of course, the most active site for oxidative attack by $\cdot OH$ radicals.

Effect of catalyst concentration

Increasing the amounts of ZnO in the aqueous suspension increases the PS decolorization.

The initial rate of decolorization increases with the increase of concentration of ZnO in the suspension (Figure 8). Figure 8 suggests that the initial rate of decolorization may increase linearly up to about 0.15 g ZnO. Afterwards, deceleration becomes prominent as the concentration of ZnO in the suspension increases. Accordingly, PS molecules in a particular concentration need an optimum amount of ZnO for their decolorization





with the visible light. The time for complete decolorization will certainly depend upon the intensity of the visible light.

An important aspect of increasing the concentration of ZnO in the aqueous suspension is to increase its turbidity. This should have affected the intensity of the visible light photons that cause excitation of the dye molecules adsorbed on the ZnO particles in the aqueous suspensions. However, such excitation by visible light photons does not seem to be spectacularly affected. This could be due to the efficient stirring of the suspension, as it caused fresh surfaces to be continuously exposed to the light of the same intensity.

Effect of initial dye concentration

The trend (Figure 9) in the decolorization of PS in the ZnO suspension with its concentrations seems to be similar to that with the increase of concentrations of ZnO, and the situation arises because of a small number of adsorption sites on ZnO in comparison to the increasingly larger number of PS molecules. Moreover, the inner filter effect [40,41] becomes predominant with increasing PS concentration in the suspension. The decolorization of PS drops from about 93% to 60% when its concentration is increased from 4.00×10^{-5} M to 8.00×10^{-5} M (Table 2). Of the various factors that cause inner filter effect during the dye decolorization process by visible light in the presence of ZnO particles, the concentration seems to be the most prominent [42].

The kinetic data were applied to the Langmuir-Hinshelwood equation in the following form:

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

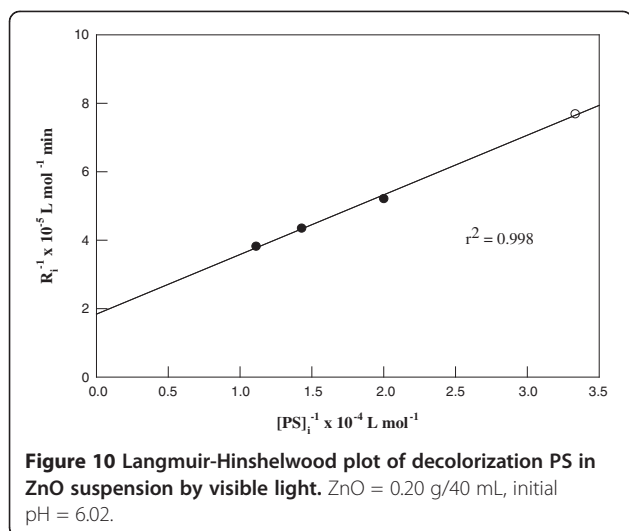
R_i is the initial rate of disappearance of the PS color from ZnO suspension; k_{L-H} is a proportionality constant and provides a measure of intrinsic reactivity of PS adsorbed on ZnO [43]. K_{L-H} is the apparent adsorption equilibrium constant (so-called Langmuir-Hinshelwood constant).

R_i^{-1} vs $[PS]_i^{-1}$ is a nicely fitted straight line (Figure 10). K_{L-H} and k_{L-H} values are 10.50×10^3 L mol⁻¹ and 8.67×10^{-6} mol min⁻¹, respectively. The higher K_{L-H} value compared to that of K_L (8.23×10^3 L mol⁻¹) suggests some surface activation of the ZnO surface by visible light.

Table 2 Influence of various parameters on decolorization of PS in ZnO suspension under visible light irradiation

| [PS] × 10 ⁵ M | ZnO g/40 mL | pH | Initial rate $r_i \times 10^6$ mol L ⁻¹ min ⁻¹ | % decolorization after 60 min | $K_{L-H} \times 10^{-4}$ L mol ⁻¹ | $k_{L-H} \times 10^6$ mol min ⁻¹ |
|--------------------------|-------------|------|---|----------------------------------|--|---|
| 5.00 | 0.20 | 3.69 | 1.83 | 85.62 | 1.05 $r^2 = 0.995$ | 8.67 $r^2 = 0.995$ |
| | | 4.38 | 1.84 | 84.42 | | |
| | | 5.26 | 1.83 | 85.82 | | |
| | | 6.02 | 1.84 | 85.02 | | |
| | | 7.25 | 1.83 | 84.32 | | |
| 5.00 | 0.10 | 6.02 | 1.28 | 79.35 | | |
| | | | 1.84 | 85.02 | | |
| | | | 2.10 | 89.28 | | |
| | | | 2.32 | 92.48 | | |
| | | | 2.47 | 94.61 | | |
| 4.00 | 0.20 | 6.02 | 1.45 | 93.07 | | |
| 5.00 | | | 1.84 | 85.02 | | |
| 6.00 | | | 2.28 | 75.99 | | |
| 7.00 | | | 2.52 | 68.02 | | |
| 8.00 | | | 2.64 | 59.85 | | |

Temperature is 30°C.



Conclusion

In the presence of visible light, ZnO can efficiently catalyze the decolorization of PS in the aqueous suspension. 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. Photodecolorization of PS is unaffected by the initial pH of its solution in the range of 4 to 7. 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.

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 and IMI 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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