

Research Article

Comparison between Solar and Artificial Photocatalytic Decolorization of Textile Industrial Wastewater

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Received 5 February 2012; Revised 15 April 2012; Accepted 24 April 2012

Academic Editor: Jianguo Yu

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The photocatalytic decolorization of industrial wastewater was investigated by using TiO_2 and ZnO photocatalysts. Heterogeneous photocatalytic processes applied under natural weathering conditions, in the presence of solar radiation show a promising degradation capability. The complete removal of color could be achieved in a relatively short time of about 20 minutes, when ZnO was used and about 100 minutes when TiO_2 was used under solar irradiation. However, in the presence of artificial UV-light, complete decolorization of textile industrial wastewater was obtained after less than one hour of irradiation when ZnO was used and in less than two hours, when TiO_2 was used at the same temperature. The results indicate that the degree of photocatalytic decolorization of textile industrial wastewater was obviously affected by different parameters. These parameters include catalyst mass, type of catalyst, type of reactor, type of dye, dye concentration, and temperature. The procedure used in this research can be used as an efficient technology for solar photocatalytic decolorization of the colored wastewater discharged from the textile industry under the climatic conditions of most countries.

1. Introduction

Treatments of industrial wastewater, especially textile wastewater, will provide huge amount of water to face water scarcity around the world. Moreover the reliable treatments of wastewater will reduce contamination of soils, surface, and ground water, and as a result public health will be protected. Textile wastewaters are strongly colored and contain high amounts of organic matter depending on forms of dyes and auxiliary chemicals [1, 2].

Titanium dioxide and zinc oxide are widely and economically available. These semiconductors can be excited with light of a wavelength in the range of the solar spectrum ($\lambda > 310 \text{ nm}$). Using of solar irradiation is very attractive technology from the economical point of view.

Titanium dioxide and zinc oxide are universally considered as the most important photocatalysts, due to their considerably low bandgap energy ($\sim 3.2 \text{ eV}$) and their lower cost. However, various methods have been developed to reduce the band gap and the electron-hole recombination. Jing et al. [3] reported that the photocatalytic activity of

the semiconductor is strongly dependent on its crystallite size, specific surface area, morphologies, and textures. The photocatalytic activity of ZnO hollow spheres with porous crystalline shells was increased significantly compared with untreated samples of ZnO at ambient temperature [4]. The hierarchical organization of nanosheets together with annealing-induced carbon doping within the ZnO lattice accounting for the improved visible light photocatalytic activity of porous carbon self-doped ZnO consequently gave better photocatalytic activity of ZnO products [5]. The photocatalytic properties of titanium dioxide are improved significantly in the presence of high energy facets, making this material attractive for various environmental applications [6]. The surface structure at the atomic level plays an important role in tuning the adsorption selectivity and, consequently, photocatalytic activity of semiconductor [7]. Graphene-based semiconductor photocatalysts also have important environmental and energy applications [8]. Titanium dioxide response toward visible light was increased after the addition of silver using the hydrothermal synthesis due to the formation of Ag aggregation on the TiO_2 surface

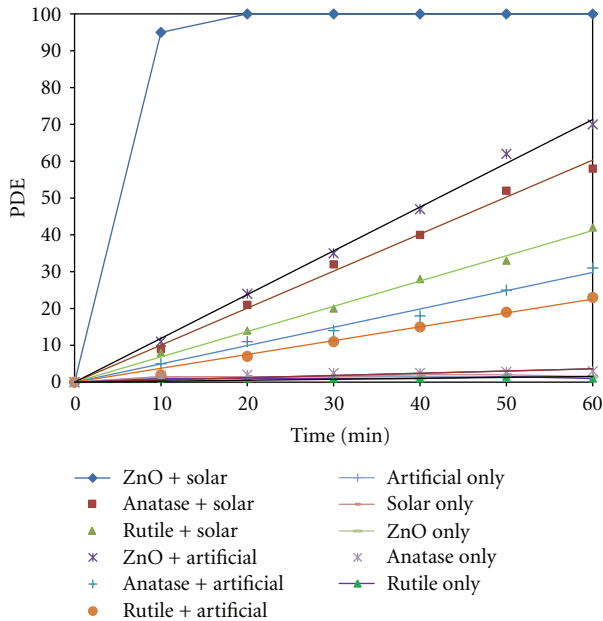


FIGURE 1: Effect of types of catalyst and irradiation on photocatalytic decolorization efficiency of real industrial wastewater.

[9]. The photocatalytic activity of titanium dioxide is also significantly enhanced by those embedding gold nanoparticles in the Au-TiO₂ nanocomposites. The prepared Au-TiO₂ nanocomposites exhibit a highly visible light photocatalytic activity, and their photocatalytic activity is higher than that of the pristine TiO₂ nanoparticles due to the surface plasmon resonance [10]. Wang et al. [11] reported that photocatalytic activity of TiO₂ powders calcined at 500°C was two times higher than that of the uncalcined TiO₂. The authors explained that this was due to the enhancement of anatase crystallization and the optimal mass ratio (ca. 1:2) of rutile to anatase.

The band gap of metalized titanium dioxide prepared by a simple impregnation method was reduced to 2.80 eV. Moreover, the solar decolorization and mineralization rates for Ta/TiO₂- and Nb/TiO₂-mixed oxide photocatalysts were improved by about 140% and 237%, respectively compared with untreated Degussa P25 TiO₂ [12].

The prepared carbon-modified TiO₂ powders by impregnation method using a commercial available titania powder, Hombikat UV100, as matrix material, showed better photoactivity of nitrogen oxides degradation than that of unmodified TiO₂ [13]. Comparing with the untreated TiO₂, anatase mesoporous titanium dioxide codoped with nitrogen and chlorine (N-Cl-TiO₂) greatly improves the photoreponse of TiO₂, thereby reducing the band gap [14]. The authors explained the enhancement of photocatalytic activity due to the small crystalline size, intense light absorption in visible region, and narrow band gap [14].

Wang et al. [15] in a recent review mentioned that doping of metal and nonmetal elements can improve the photocatalytic activity of titanium dioxide.

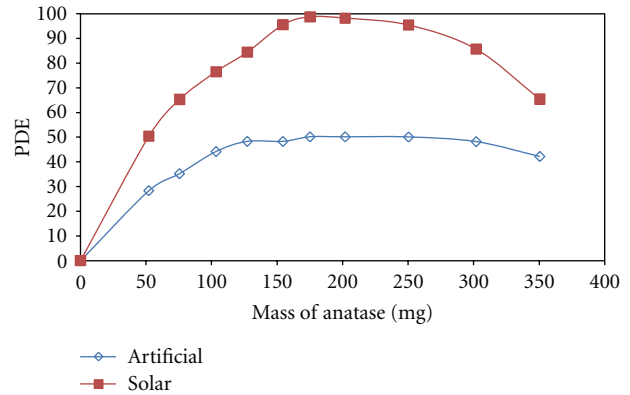


FIGURE 2: Mass effect of TiO₂ (anatase) on photocatalytic decolorization efficiency of real textile industrial wastewater for different times of irradiation under solar and artificial radiation.

Solar disinfection (SODIS) is a simple and low-cost technique used to disinfect drinking water. It was found that this important technique was enhanced in the existence of photocatalysts by far more effective way than SODIS alone for the inactivation of both the total coliforms and the faecal coliforms [16].

Low pH and high concentration of Cl⁻ solutions were found beneficial to the photocatalytic activity of titanium dioxide [17].

By monitoring the surface chemistry of Pt-TiO₂ microspheres before and after photocatalytic reactions, it was found that the degradation of methyl orange molecules was more complete in the presence of platinum particles on the surface of titanium dioxide [18].

We have investigated previously, prolifically, the decolorization, photodegradation, and phytoremediation of many water-soluble toxic compounds in real and simulated industrial wastewater. The treated wastewaters could be recycled in the same industry or reused in another industry or for agricultural fields. The efficiency of these methods of treatments is between 70 and 95% [19–29].

The aim of the present paper is to investigate photocatalytic decolorization of real and simulated textile wastewater using TiO₂ and ZnO as photocatalysts with irradiation with solar and artificial radiation at different conditions.

2. Experimental Procedure

Real textile industrial wastewater sample was collected at the mid of December 2008 from textile factory in Hilla (Babylon Governorate, Iraq) at the discharging point. The sample was collected in hydrochloric acid-washed polyethylene container, 10 dm⁻³ in volume, which previously washed for several times with nitric acid (0.05 N) and rinsed for several times with redistilled water.

Experiments were carried out during December 2008 till July 2009. Solar irradiation experiments have been performed at the floor of the Chemistry Department building in the College of Science, Babylon University, in an open atmosphere between 11.00 a.m. and 1.00 p.m.

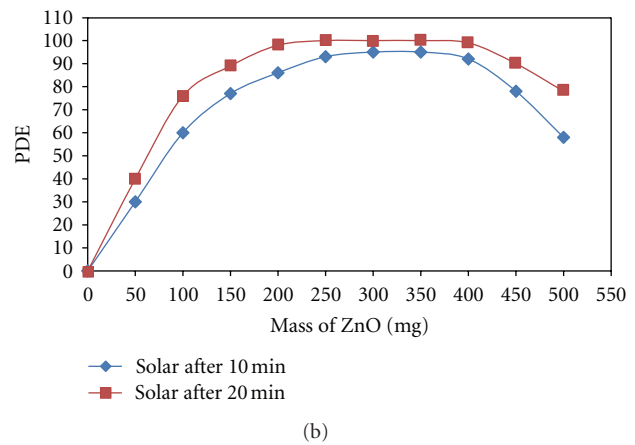
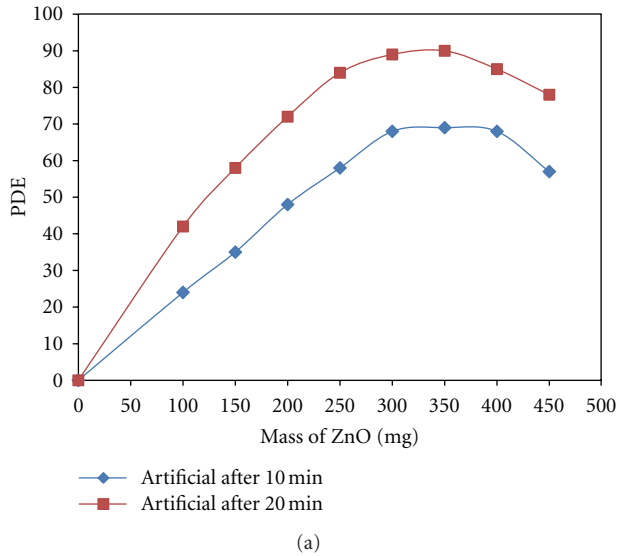


FIGURE 3: Mass effect of ZnO on photocatalytic decolorization efficiency of real textile industrial wastewater for different times of irradiation under artificial radiation (a) and solar irradiation (b).

Sunlight illuminations were accomplished in a 300 cm³ glass container containing 100 cm³ of the industrial wastewater solution. The sunlight radiation was collected using converging lens with a focal length of 14 cm. Artificial irradiation experiments are performed in a homemade reactor. The reactor consists of graduated 400 cm³ Pyrex glass beaker and a magnetic stirring setup. The radiation source was a Philips 125 w/542 high-pressure mercury lamp (Holland). The lamp was positioned perpendicularly above the beaker. The mercury lamp was allowed to warm up for 3 minutes to ensure a stable light intensity before commencing a reaction.

Titanium dioxide P-25 anatase (commercial TiO₂), of surface area 50 m² g⁻¹, was purchased from Degussa. Titanium dioxide rutile was obtained from Fluka (Assay 97%) and zinc oxide with 99.5% purity, supplied by Carlo ERBA.

In all experiments, the required amount of the catalyst, titanium dioxide (anatase or rutile) or zinc oxide, was suspended in 100 cm³ of industrial wastewater using a magnetic stirrer. At predetermined times, 1.5 cm³ of reaction mixture was collected and centrifuged (4,000 rpm, 15 minutes) in an 800B centrifuge. The supernatant was carefully removed by a syringe with a long pliable needle and centrifuged again at same speed and for the same period of time. This second centrifugation was found necessary to remove fine particles of ZnO or TiO₂. After the second centrifugation, the absorbance at certain wavelengths of the supernatants was determined using ultraviolet-visible spectrophotometer, type UV-1650 PC, Shimadzu and visible spectrophotometer type v-1000, T-Chromatech.

Photocatalytic decolorization efficiency (PDE) of the dye was followed, spectrophotometrically, by a comparison of the absorbance, at specified interval times, with a calibration curve accomplished by measuring the absorbance, at known wavelengths, with different concentrations of the dye solution. PDE is calculated from a mathematical equation

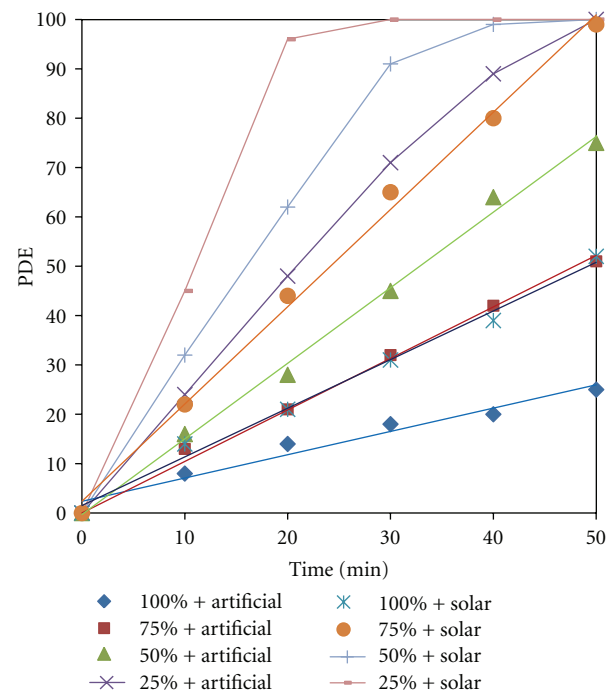


FIGURE 4: Effect of initial dye on photocatalytic decolorization of real textile industrial wastewater on TiO₂ (anatase) and solar radiation.

adapted from measurements of decolorization used before [30, 31].

$$\text{PDE} = \frac{(\text{absorbance})_0 - (\text{absorbance})_t}{(\text{absorbance})_0} \times 100, \quad (1)$$

where (absorbance)₀ is the absorbance at 422 nm before irradiation and (absorbance)_t is the absorbance at time *t*. To

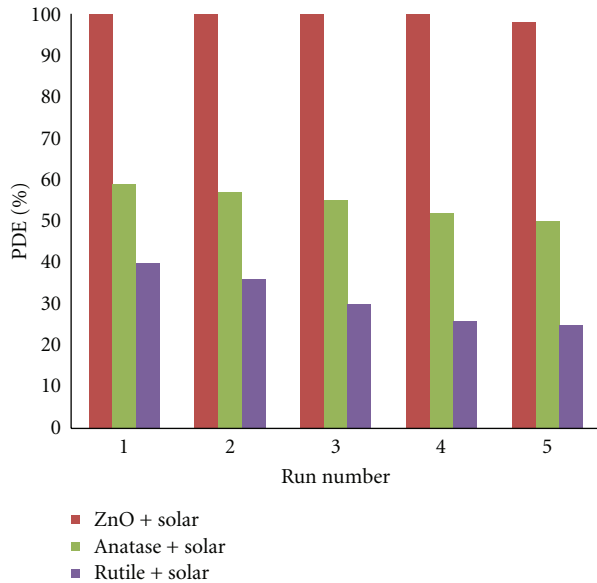


FIGURE 5: Effect of TiO₂ (anatase and rutile) and zinc oxide reuse on photocatalytic decolorization efficiency after 60 minutes of solar irradiation.

TABLE 1: Effect of temperature on photocatalytic decolorization efficiency of real textile industrial wastewater on TiO₂ and solar radiation.

Time/min.	Temperature/K			
	293.15	298.15	304.15	310.15
0	0	0	0	0
10	9.11	11.23	12.43	14.22
20	18.23	20.88	23.54	26.44
30	26.66	30.43	34.05	39.76
40	36.55	40.22	46.12	55.45
50	45.65	51.65	58.54	70.34
60	55.24	58.34	65.45	78.85

check the validity of the previous equation for used textile industrial wastewater, the photodegradation percentage of the dye was followed, spectrophotometrically, by a comparison of the absorbance, at specified interval times, with a calibration curve accomplished by measuring the absorbance, at known wavelengths, with different concentrations of the dye solution.

3. Results and Discussion

3.1. Photocatalytic Decolorization of Real Textile Industrial Wastewater at Different Conditions. Figure 1 shows the change in photocatalytic decolorization efficiency (PDE) of real textile industrial wastewater in the present and absent of catalyst and in the present and absent of solar or artificial irradiation.

The results indicate that the activity of different catalysts fell in the sequence:

TABLE 2: Effect of temperature on photocatalytic decolorization efficiency of real textile industrial wastewater on ZnO and artificial radiation.

Time/min	Temperature/K			
	290.15	298.15	313.15	319.15
0	0	0	0	0
10	10.22	12	17.25	18.22
20	21.4	14.12	35.25	38.04
30	32.8	36.22	52.33	56.22
40	42.44	48.35	68.88	77.25
50	52.3	62.55	85.25	100
60	63.55	72.85	97.88	100

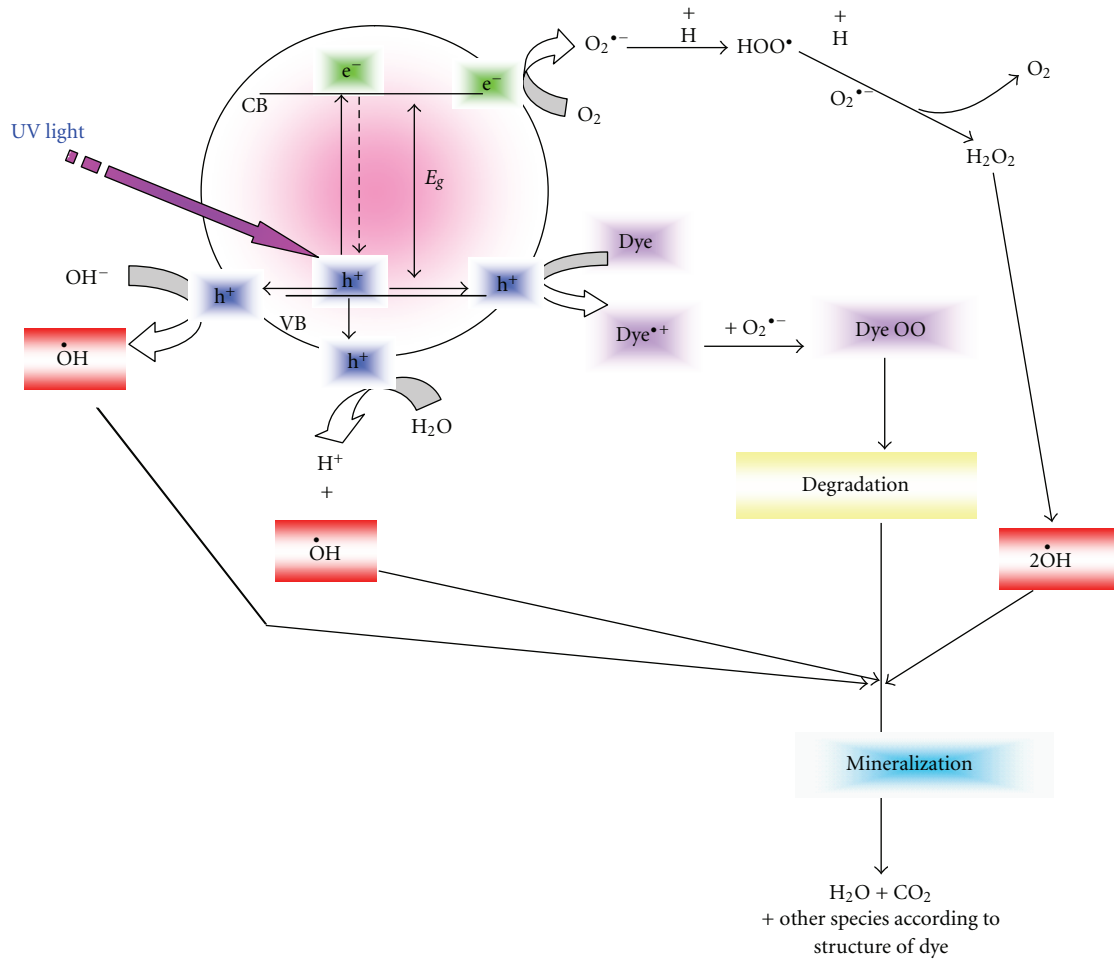
ZnO (solar) > ZnO (artificial) > Anatase (solar) > Rutile (solar) > Anatase (artificial) > Rutile (artificial) \gg No catalyst = No solar or artificial irradiation = 0.

These results also indicate that there was no dark reaction. Incubations of colored industrial wastewater without solar or artificial radiation and/or without catalyst was performed to demonstrate that decolorization of the dye was dependent on the presence of both; light and catalyst.

Results indicate that ZnO in the existence of solar irradiation is most active. Sakthivel et al. [32] explained the higher activity of ZnO due to absorption of large fraction of the solar spectrum and absorption of more light quanta by ZnO than TiO₂. However, the amount of zinc oxide required to reach the optimum activity is two times more than that for titanium dioxide (anatase or rutile) [25, 26]. In another study, Attia et al., [23], observed that ZnO is less active than anatase when the same weight of catalysts is used for photocatalytic degradation of textile wastewater. Akyol et al., [33] reported that ZnO is more active than TiO₂ for the decolorization efficiency of aqueous solution of a commercial textile dye due to the band gap energy, the charge carrier density, and the crystal structure.

All types of catalysts used in this research showed higher photocatalytic activity under sunlight irradiation. This may be due to high light intensity of solar radiation in IRAQ. Neppolian et al. [34] reported that solar energy may emerge as a viable method for textile wastewater treatment because of its eco-friendliness and cost effective where 96% of textile industrial wastewater was photodegraded during April–June; peak summer period of the year in Chennai, India. However, Akbal [35] concluded that the photocatalytic decolorization rate of methylene blue and methyl orange with UV light irradiation was higher than that with solar light irradiation.

3.2. Effect of Catalyst Mass. Figure 1 shows that 175 mg of anatase is sufficient for maximum rate of decolorization when mercury lamp was used for irradiation. Moreover the same mass was found also sufficient when the reaction vessel was illuminated with solar irradiation. These observations proved that the optimum mass to achieve maximum decolorization percentage is independent on type of irradiation



SCHEME 1: Dye/semiconductor/UV light system.

source and solar irradiation in this system is more efficient than 125 w/542 high pressure mercury lamp.

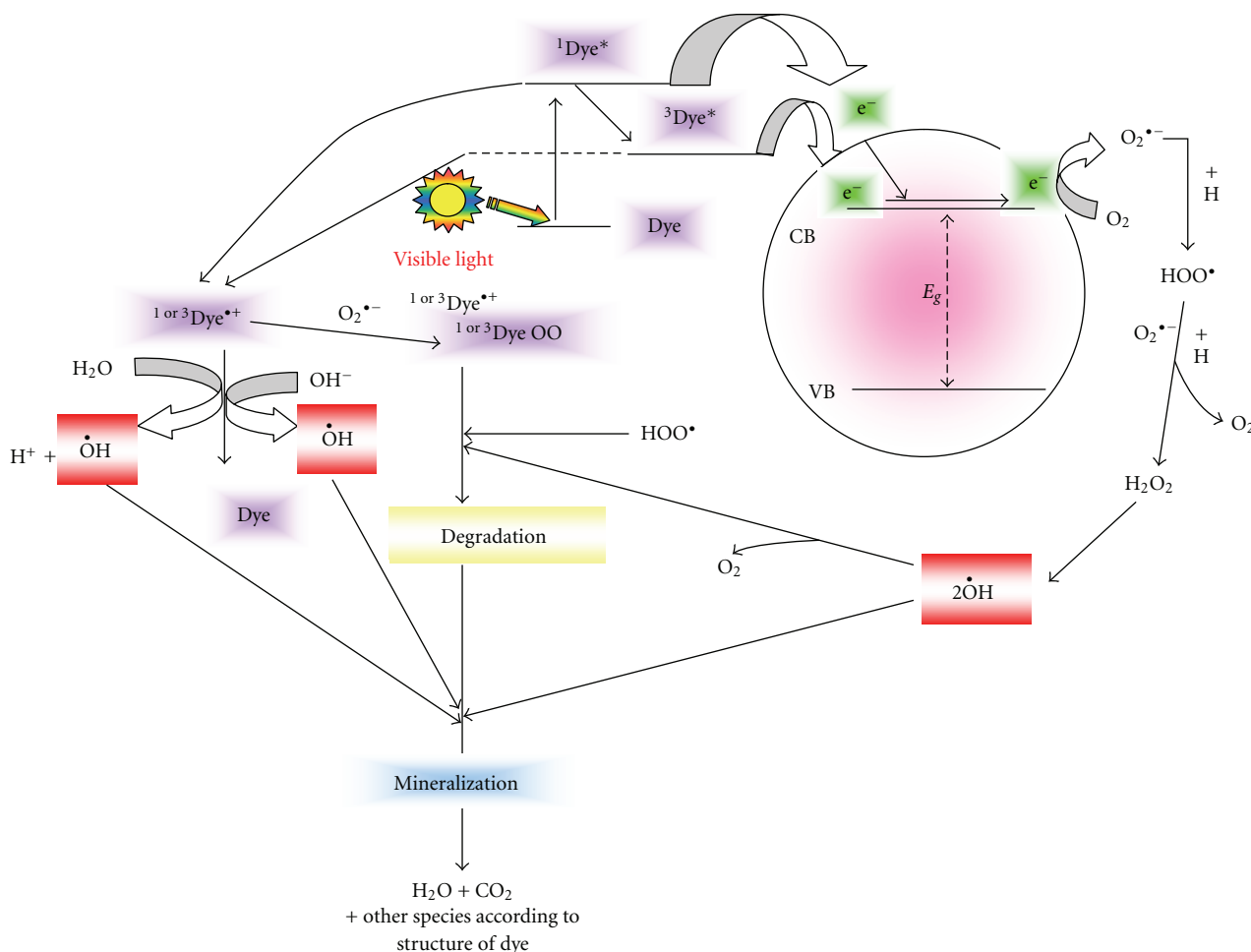
The same observations were noted when ZnO was used as photocatalyst. However the optimum mass to achieve maximum PDE is 350 mg. These results are shown in Figure 2.

Mass of catalyst is regarded as the major parameter affecting the photocatalytic degradation efficiency [36]. The results plotted in Figures 2 and 3 shows that the photocatalytic degradation efficiency of real industrial wastewater increases linearly with the increasing in catalysts mass at the first stages. This behavior may be due to an increase in the amount of active site on surface of photocatalyst particles and as a result, the number of dye molecule that adsorbed on the surface of photocatalyst will be increased and that will lead to an increase in the density of particles in the area of illumination [37]. However, after reaching maximum photocatalytic degradation rate, addition of excess amount of catalysts has no effect on photocatalytic rate, so a plateau region was observed. After the plateau region is achieved, the activity of photocatalytic decolorization decrease with increase of catalyst concentration for titanium dioxide and zinc oxide. This behavior is more likely due to Light

scattering by catalyst particles at higher concentration which leads to decrease in the passage of irradiation through the sample leading to poor light utilization [38, 39]. Deactivation of activated photocatalyst molecules colliding ground state molecules with increasing the load of photocatalyst may be also cause reduction in photocatalyst activity [37]. High concentration of loading catalyst also decreases the number of surface active sites [40].

3.3. Effect of Temperature. Reaction was followed at four different temperatures in the range 293.15–315.15 K using 175 mg of anatase under solar radiation. The results in Table 1 indicate that the PDE of real textile industrial wastewater increases with increasing of temperature.

Reaction was also followed at four different temperatures in the range 290.15–319.15 K using mercury lamp and 350 mg of zinc oxide. The results are listed in Table 2. The results indicate that PDE of real textile industrial wastewater increases with increasing of temperature. However it is clear that temperature is the least active parameter in the photocatalytic decolorization of real textile industrial wastewater.



SCHEME 2: Nonregenerative dye/semiconductor/visible light system.

It is well known that the most desirable system for complete mineralization of a wide range of organic substrates is that which operates under natural weathering conditions without producing of harmful byproducts [41]. Photocatalytic treatments and especially with solar irradiation offer that because they are mostly proceeding under natural weathering conditions. Adsorption of reactants on the surface of catalysts is a spontaneous exothermic phenomenon so it is enhanced by reduction of temperature [42].

3.4. Effect of Initial Dye Concentration. The results in Figure 4 show the changing of rate of decolorization of real textile industrial wastewater on 175 mg of anatase by using solar and artificial irradiation at 298.15 K with the initial direct dye concentrations (25%–100%) at different times. The results indicate that decreasing of dye concentration decreases the time of decolorization.

This behavior related to decreasing of the path length of photons entering the solution as the initial concentration of dye increases, and as a result the number of photon absorbed by the catalyst decreases.

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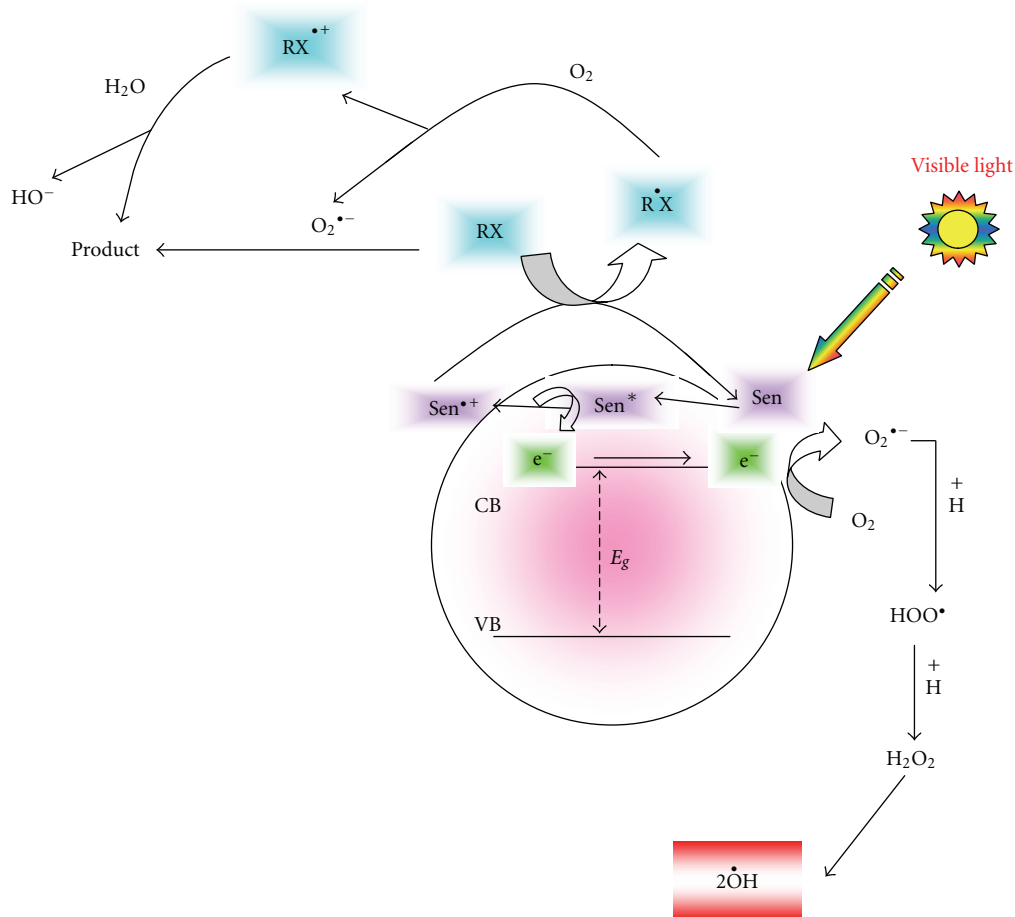
dye increases, and as a result the number of photon reached the catalyst surface decreases [43–45].

3.5. The Reusability of Catalyst. The reusability of the titanium dioxide (anatase or rutile) and zinc oxide was examined. The recovered catalysts were reused for five consecutive runs. Figure 5 shows only a slight decrease in the activity of these photocatalysts after five consecutive uses. However, rutile showed more decrease than anatase and zinc oxide.

4. Kinetics of Decolorization

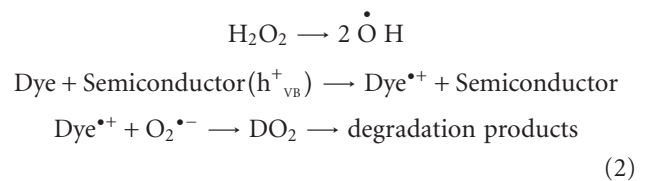
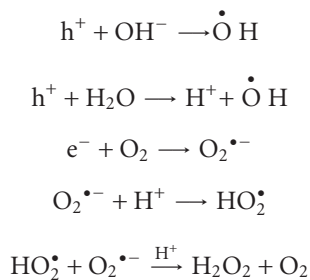
In photosensitization process, which is defined as the conversion of light energy to chemical reactivity [46], a colored organic or inorganic material is added to a semiconductor. The added sensitizer (Sens) absorbs light in to the visible range to yield an excited state of the sensitizer (Sens)*. The excited sensitizing molecule injects an electron from excited singlet (S1) or triplet (T1) states into the conduction band of the semiconductor [47].

The illumination of suspended semiconductor in an aqueous solution of dye with unfiltered light (polychromatic light) leads to the possibility of the existence of two pathways [21, 48].

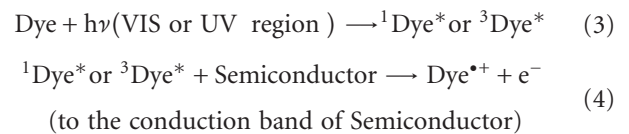


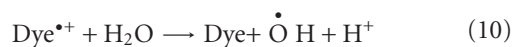
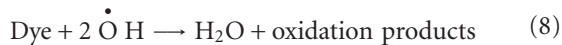
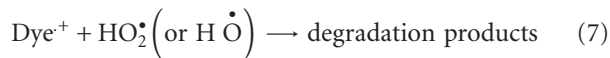
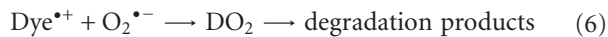
SCHEME 3: Regenerative dye/semiconductor/visible light system.

(1) In the first pathway, the part of light with energy equal to or more than the band gap of the illuminated semiconductor will cause a promotion of an electron to conduction band of the semiconductor, and as a result, a positive hole will be created in the valence band. The formed photoholes and photoelectrons can move to the surface of the semiconductor in the presence of light energy. The positive hole will react with adsorbed water molecules on the surface of semiconductor producing $\cdot\text{OH}$ radicals, and the electron will react with adsorbed oxygen on the surface. Moreover, they can react with deliquescent oxygen and water in suspended liquid and produce perhydroxyl radicals ($\text{HO}_2\cdot$) with high chemical activity [49]. The processes in this pathway, as shown in Scheme 1, can be summarized by the following equations:

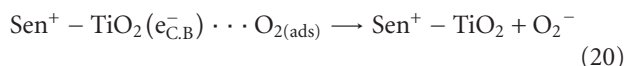
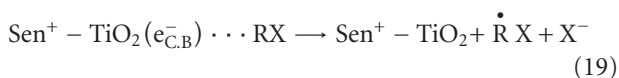
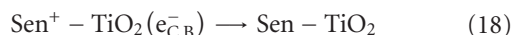
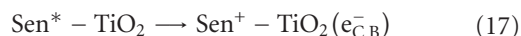
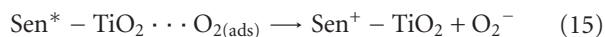
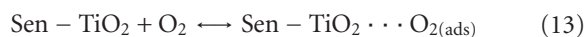
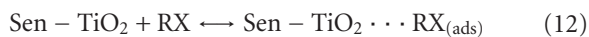
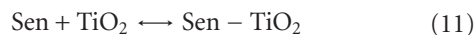


(2) In the second pathway, the other part of light with energy which is less than the band gap of the illuminated semiconductor will be absorbed by the adsorbed dye molecules. Dye molecules will be decolorized by a photosensitization process. The photocatalytic decolorization of dyes, which is described as photosensitization processes, is also characterized by a free radical mechanism. In this process, the adsorbed dyes molecules on the surface of the semiconductor can absorb a radiation in the visible range in addition to the radiation with short wavelengths [19, 50, 51]. The excited colored dye (dye^*) (in the singlet or triplet state) will inject an electron to the conduction band of the semiconductor [24]. The processes in this pathway, as shown in Scheme 2, can be summarized by the following equations:





The mechanism above is favoured by nonregenerative organic dye where dye/semiconductor/visible light system and the sensitizer itself degrade. However, in regenerative semiconductor system, (Scheme 3), the following mechanism may be followed:



Cho et al. [52] conclude that there is no direct electron transfer between an excited sensitizer and CCl_4 molecules, in homogeneous solution, and the existence of semiconductor is essential for sensitized photocatalysis. Platinum supported on titanium dioxide acts as an excellent sensitizer and could have practical advantages as a mild and convenient photocatalyst for selective oxidation processes [53]. The addition of rhodamine B, as sensitizer to TiO_2 dispersion system, increases the rate of photooxidation properties [54]. The authors explained that due to the fact that more light absorbed by rhodamine B between 460 and 580 nm, then the energy transfer from sensitizer to TiO_2 or to any other active species hence promotes the photocatalytic activity of titanium dioxide.

5. Conclusions

- (1) The existence of catalyst and lights is essential for photocatalytic degradation of colored dyes.

- (2) Solar photocatalytic treatment is an efficient technique for decolorization of industrial wastewater through a photocatalytic process, and the transformation is practically complete in a reasonable irradiation time.

- (3) Visible light/ ZnO and visible light/ TiO_2 systems could be efficiently used for photodegradation of textile industrial wastewater. The results indicate that the degree of photodegradation of textile industrial wastewater was obviously affected by different parameters. The complete removal of color could be achieved in a relatively short time of about 20 minutes, when ZnO was used under solar irradiation.

- (4) The procedure used in this research can be used as an efficient technology for solar photocatalytic degradation of the colored wastewater discharged from the textile industry under natural weathering conditions.

Acknowledgments

The author is gratefully acknowledging the financial support provided by the Arab Science and Technology Foundation (ASTF) and the US Civilian Research and Development Foundation (CRDF) without which this research would not have been possible. The author also expresses sincere appreciation to all the staff working at ASTF, Baghdad Office. This paper was Published in Proceedings of ISES, Solar World Congress, 2011: Kassel-Germany (28th August–2nd September, 2011).

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