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Synthesis and characterization of ZnO-TiO₂ nanocomposites and their application as photocatalysts

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Abstract

Nanocomposite ZnO-TiO₂ powders of varying ZnO/TiO₂ molar ratios have been prepared from their salt/compound by heating at 600°C and 900°C and characterized using scanning electron microscope and X-ray diffraction techniques. The nanosized powders can decolorize/degrade brilliant golden yellow (BGY), an azo dye extensively used in textile industries, in water under solar irradiation. The effects of various parameters such as photocatalyst loading, molar ratio of ZnO/TiO₂, pH of the solution, initial dye concentration, and irradiation time on the photodecolorization have been investigated. ZnO-TiO₂ nanocomposite (6 g/L) in the molar ratio of 1:1 or 3:1, prepared at 900°C, can efficiently decolorize about 98% of 20 mg/L BGY at pH of about 7 by 2-h illumination in sunlight. The initial dye decolorization follows pseudo-first-order kinetics. Finally, trial experiments were done using real textile wastewater to find out the effectiveness of the photocatalysts to a more complex system.

Keywords: Nanocomposite ZnO-TiO₂, Photocatalysts, Dye pollution, Solar photocatalytic decolorization

Background

Effluents containing dyes often create severe environmental pollutions because of their direct disposal into the nearby water bodies [1]. Most of the textile synthetic dyes are azo compounds, that is, they contain -N = N- linkage in their structure. About 15% of the dyes are lost in wastewater during dyeing operation. This affects the esthetic merit of surface water and reduces light penetration, hampering aquatic lives and hindering photosynthesis. Moreover, some dyes are either toxic or mutagenic and carcinogenic [2]. Consequently, many approaches have been attempted to find out an effective method for remediation of industrial wastewaters. Unfortunately, the conventional methods for treating dye-containing wastewaters are usually ineffective in terms of complete mineralization. Biodegradation of dyes is generally selective and incomplete; some dyes with aromatic structure are difficult to biodegrade [3]. Therefore, an effective method is needed to overcome this problem.

In recent years, the photocatalytic degradation of various kinds of organic and inorganic pollutants using semiconductors as photocatalysts has been widely studied [4-7]. Among these semiconductors, TiO₂ has been extensively investigated as the most promising photocatalyst due to its high photocatalytic activity, non-toxicity, high photochemical stability, and low-cost. However, it divulged some drawbacks. For example, TiO₂ (band gap 3.2 eV corresponds to 388 nm) can only absorb the UV region (only about 4%) of the solar radiation, whereas solar spectrum has about 40% visible region (400 to 700 nm), and it shows high recombination rate of photo-induced electron-hole pairs which may affect negatively the degradation ratio and rate [8]. In order to make TiO₂ suitable for receiving solar energy efficiently in a wide range from UV to visible, many methods such as dye sensitization, metal- or nonmetal-doped TiO₂-based nanoparticles, and modification of TiO₂ by the addition of another metal oxide-based semiconductor have been used. It has been shown that coupled semiconductors seem to be simple and viable photocatalysts. Photocatalytic process is based on the generation of electron-hole pairs by means of band-gap

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radiation that can give rise to redox reactions with the species adsorbed on the surface of the photocatalysts. In principle, the coupling of different semiconductor oxides seems useful in order to absorb a wide range of solar radiation (both UV and visible regions) and to achieve a more efficient electron-hole pair separation, and consequently, a higher photocatalytic activity will be attained [9]. Various composites formed by TiO_2 and other inorganic oxides such as ZnO [10], SnO_2 [11], SiO_2 [12], In_2O_3 [13], Cu_2O [14], MgO [15], WO_3 [16], MoO_3 [17], and so on have been reported.

Note that ZnO is a suitable alternative to TiO_2 because its photodegradation mechanism has been proven to be similar to that of TiO_2 . In fact, in comparison to TiO_2 , ZnO has been reported to have higher photocatalytic efficiency [18,19]. ZnO can absorb over a larger fraction of UV radiation, and the corresponding threshold of ZnO is 425 nm [20]. Therefore, nanocomposites of ZnO/TiO_2 can be used as photocatalysts to degrade/decolorize organic dyes under solar irradiation in aqueous system. In this paper, nanocomposite/nanosized bicomponent $\text{ZnO}-\text{TiO}_2$ of various molar ratios of ZnO/TiO_2 were prepared through thermal decomposition of their corresponding salt/compound. The nanocomposites were characterized by scanning electron microscope (SEM)/X-ray diffraction (XRD) technique, and then the as-prepared nanocomposites were used as photocatalysts to decolorize the brilliant golden yellow (BGY), an azo dye which is extensively used in textile/dyeing industries, under solar irradiation.

Methods

Materials

Zinc carbonate was purchased from E. Merck, Mumbai, India. Metatitanic acid was purchased from Chengdu XiYa Chemical Technology Co., Ltd., Chengdu, China. Sodium hydroxide, sulfuric acid, sodium dodecyl sulfate, and Triton X 100 were purchased from BDH Chemicals Ltd., East Yorkshire, UK. BGY (Figure 1) was obtained from local dye suppliers. Wastewater was collected from local textile factory in Dhaka, Bangladesh. De-ionized water was used throughout the experiments.

Preparation and characterization of ZnO/TiO_2 nanocomposites

Metatitanic acid was first neutralized by adding concentrated NH_3 solution (28%) under ice-cold temperature. Then, the neutralized metatitanic acid was spray-dried using air to obtain spherical shape granules ($\text{TiO}_2 \cdot x\text{H}_2\text{O}$) [21]. For the preparation of ZnO/TiO_2 nanocomposite, the requisite amount of zinc carbonate was mixed with granules $\text{TiO}_2 \cdot x\text{H}_2\text{O}$ in a mortar in such a way that the mixture satisfied the desired molar ratios of ZnO and TiO_2 [22]. Each sample of the powered mixtures was taken in a platinum crucible and heated in a muffle furnace at pre-set temperatures for 1 h. Initially, samples were heated batch-wise from 200°C to 900°C for 1 h. The calcined samples were heated further at the desired temperatures for constant weight. SEM (JSM-6490LA, JEOL, Tokyo, Japan) was used to study the particle sizes, and XRD technique (RIINT 2100, Rigaku Corp., Tokyo, Japan) was used to study the structural characteristics of the as-prepared samples.

Photocatalysis studies

Photocatalytic experiments were carried out in a 50-mL capacity borosilicate glass as photochemical batch reactor with dimensions 6×4 cm (height $\times$ diameter). All photocatalytic experiments were carried out under the similar conditions on sunny days of June to July between 10 A.M. and 2 P.M. when the solar intensity fluctuations were minimal. A series of 20 mg/L BGY solutions were prepared. The pH of the dye solutions was adjusted to the desired value using either dilute H_2SO_4 or NaOH . The reaction suspension was prepared by adding appropriate quantity of photocatalyst into 50 mL of BGY aqueous solutions, and then the mixture was magnetically stirred in dark for 15 min to establish adsorption-desorption equilibrium. The suspension containing BGY and photocatalyst was then kept under sunlight, and the photocatalytic reaction timing was started. During the experiment, the reaction mixture was stirred vigorously using magnetic stirrer. The reaction mixture was withdrawn after an appropriate illumination time and centrifuged at 8,000 rpm to settle the suspended solids. The clear supernatant was then analyzed in a UV-vis

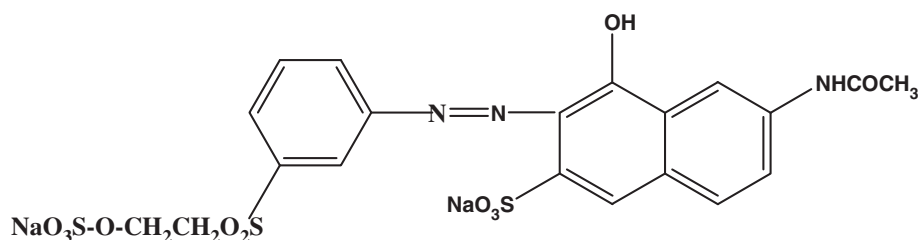


Figure 1 Structure of BGY.

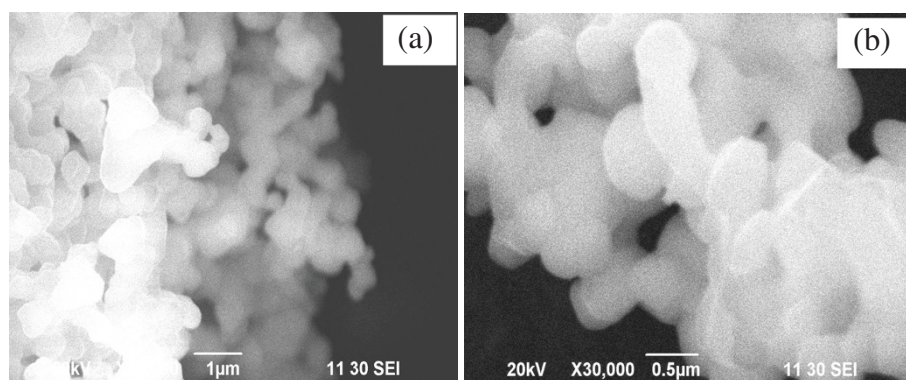


Figure 2 SEM images of the as-prepared nanocomposites of ZnO/TiO₂ with a ratio of 1:1. 600°C (a) and 900°C (b).

spectrophotometer (UV-1800, Shimadzu, Kyoto, Japan) at $\lambda_{\max} = 417$ nm, absorption maximum of BGY. The decolorization efficiency (D) was observed in terms of change in intensity of the dye before and after the solar irradiation. The D was calculated as $D (\%) = \{[(\text{dye})_0 - (\text{dye})]/(\text{dye})_0\} \times 100$, where $(\text{dye})_0$ was the equilibrium concentration of BGY after adsorption-desorption equilibrium, and (dye) was the concentration of BGY at solar irradiation time ' t '.

Results and discussion

ZnO/TiO₂ photocatalysts

Figure 2a, b shows the SEM images of ZnO/TiO₂ nanocomposites prepared by the thermal method at 600°C and 900°C, respectively. The average particle size of the composites is about 500 nm. The SEM images show the uniform sizes of the nanocomposite particles prepared at two different temperatures. Differentiation between ZnO and TiO₂ in the nanocomposites was not possible by SEM owing to the similar electron density of Zn and Ti. To get more insight into the crystal phases of the constituents of the prepared nanocomposites, XRD patterns of the samples were recorded after annealing them at different temperatures.

The phase composition of the prepared photocatalysts has been identified by XRD analysis (Figure 3). The composites are all crystalline and have the diffraction peaks at $2\theta = 31.778^\circ, 34.428^\circ, 36.258^\circ, 47.548^\circ, 56.608^\circ, 62.868^\circ, 66.388^\circ, 67.968^\circ, \text{ and } 69.108^\circ$, marked by their miller indices $((1\ 0\ 0), (0\ 0\ 2), (1\ 0\ 1), (1\ 0\ 2), (1\ 1\ 0), (1\ 0\ 3), (2\ 0\ 0), (1\ 1\ 2), \text{ and } (2\ 0\ 1))$ corresponding to ZnO (JCPDS no. 36-1451), indicating that the phase of ZnO was wurtzite (Figure 3a). In addition, peaks at $2\theta = 25.4^\circ, 38.1^\circ, 48.1^\circ, 54.8^\circ, 62.5^\circ, 75.1^\circ$ and at $2\theta = 27.4^\circ, 36.1^\circ, 41.2^\circ, 56.6^\circ, 69.1^\circ, \text{ and } 69.9^\circ$ correspond to anatase and rutile phases of TiO₂, respectively, were observed in the sample prepared at 600°C (Figure 3b). The influence of increase in temperature on the phase transformation in the photocatalysts is shown in Figure 3c. At 900°C,

surface reaction occurred between ZnO and TiO₂, resulting in the formation of Zn₂TiO₄ (Figure 3c).

Effect of molar ratios of ZnO/TiO₂ on the photocatalytic activity of the nanocomposites

In order to find out the effect of molar ratios of ZnO/TiO₂ on the photocatalytic activity of the as-prepared nanocomposites, a set of parallel experiments were done with samples 1 to 5. Figure 4 shows the effect of the variation of ZnO/TiO₂ molar ratios on the decolorization of BGY. The results indicate that all the nanocomposites (samples 2 to 4) show higher photocatalytic activity than the pure ZnO or TiO₂ (samples 1 and 5), and samples 2 and 3 with the ZnO/TiO₂ molar ratios of 3:1 and 1:1, respectively, show better photocatalytic activity with the decolorization efficiency about 98%.

In the ZnO/TiO₂ nanocomposites, after irradiation by sunlight, electron transfer occurs from ZnO to TiO₂, and simultaneously, hole transfer takes place from TiO₂

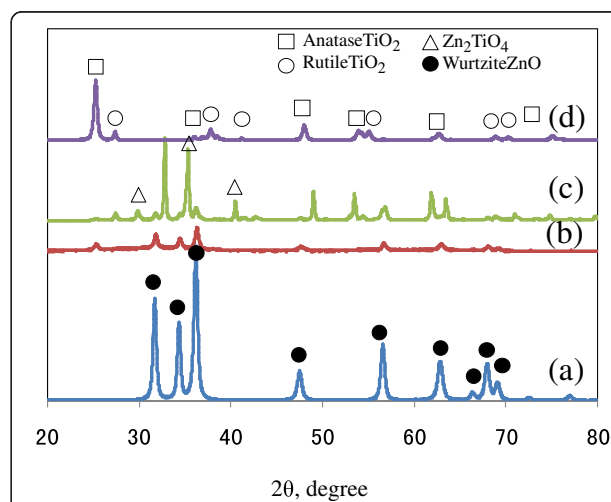


Figure 3 XRD patterns. ZnO (a), ZnO/TiO₂ nanocomposite (ZnO: TiO₂ = 1:1) annealed at 600°C (b), ZnO-TiO₂ annealed at 900°C (c), and pure TiO₂ (d).

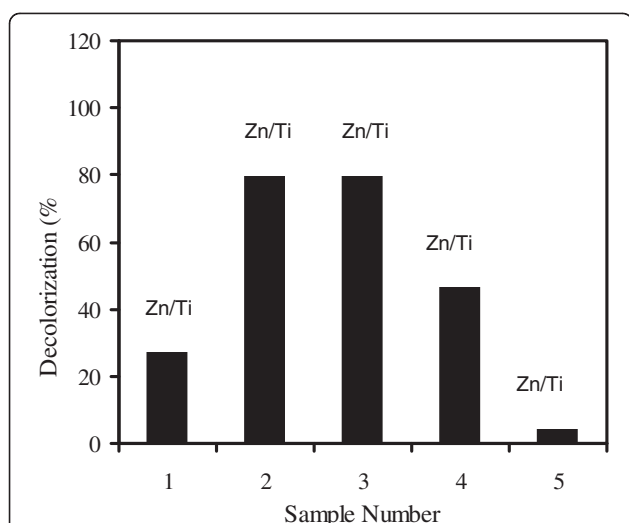


Figure 4 Effect of molar ratios of ZnO/TiO₂ (Zn:Ti) on the decolorization of BGY. BGY = 20 mg/L, pH 7.5; irradiation time = 2 h.

to ZnO [23,24]. This causes efficient charge separation, increases the life time of charge carriers, and enhances the efficiency of interfacial charge transfer to the adsorbed substrates. The combined effect of all of these is the ultimate increase of the photocatalytic activity of the ZnO/TiO₂ composite. Furthermore, the higher photocatalytic activity can be related to the amount of ZnO, which has to be sufficient to trap the photogenerated holes. On the other hand, lower decolorization efficiency was observed for sample 4. As for sample 4, the concentration of photogenerated holes trapped by ZnO was lower because of the insufficient amount of ZnO compared to that in samples 2 and 3. Accordingly, sample 2 (ZnO/TiO₂ = 3:1) has been used as a photocatalyst throughout the experiments.

As observed in the present work, Zn₂TiO₄ has also been reported to be generated by the incorporated Zn reacting with the Ti-rich phase on the SrTiO₃ surface [25]. This has led to the decrease of the surface defect concentration of SrTiO₃ and consequently increased the photocatalytic activity of Zn-doped SrTiO₃. While preparing TiO₂-capped ZnO nanoparticles, the formation of Zn₂TiO₄ phase at higher calcination temperature has been noticed [26]. The nanoparticles with TiO₂ cap as Zn₂TiO₄ exhibited higher photodecolorization yield of methylene blue under UV light (3 mW/cm²) and faster kinetics in comparison to the uncoated ZnO and P25-Degussa TiO₂ nanoparticles. It is to be noted that the band gap energy of Zn₂TiO₄ is approximately 3.10 eV [27].

Zn₂TiO₄ is known to show high separation efficiency of photogenerated carriers [28]. This characteristic of Zn₂TiO₄ and the capability of low energy photons in generating some holes in ZnO are likely to be important factors in the enhancement of decolorization of BGY by

sunlight catalyzed by ZnO/TiO₂ composite [29], where the amount of ZnO is sufficient in the mixture, and the annealing temperature is about 900°C.

Probable mechanism of sunlight decolorization of BGY in the presence of ZnO/TiO₂

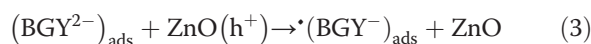
Decolorization of BGY is the result of surface reactions initiated by its adsorption on the solid matrices, forming (BGY²⁻)_{ads}. It gets excited by solar irradiation:



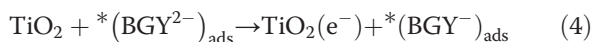
The pH of the suspension environment is likely to make ZnO as the most efficient adsorbent. Favorable hole concentration in ZnO, ZnO(h⁺), produces [•]OH radicals, the most efficient agents for BGY degradation:



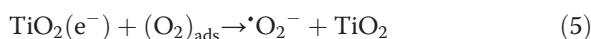
ZnO(h⁺) also produces dye radicals:



[•](BGY⁻)_{ads} is also produced by injecting an electron by [•](BGY²⁻)_{ads} in the conduction band of TiO₂:



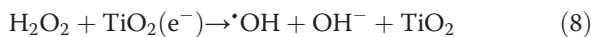
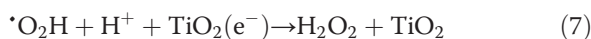
TiO₂(e⁻) in its turn initiate host of processes, such as



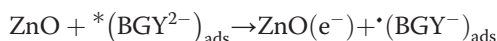
[•]O₂⁻ can then generate far more reactive species [•]O₂H (or [•]OOH) in a favorable environment:



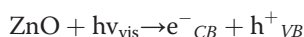
[•]O₂H may initiate the formation of [•]OH:



BGY decolorization is initiated by the attack of [•]OH on its -N = N- linkage and fragmenting the dye molecule. In the case of ZnO alone, sample 1, solar radiation not only causes reaction (4) to take place

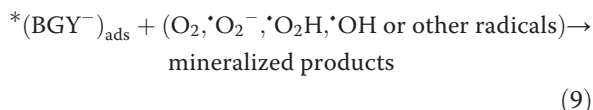


but also may induce the formation of both types of charge carriers



On the other hand only reaction (4) is likely to be possible by solar irradiation when TiO₂ is used alone, sample 5. Accordingly, ZnO is a more efficient photocatalyst than TiO₂ when sunlight is the light source.

Decolorization of BGY is accompanied by its mineralization:



The mineralized products are N_2 , CO_2 , H_2O , Na^+ , NO_3^- , and SO_4^{2-} .

Effect of photocatalyst loading on decolorization

In order to avoid the use of excess catalyst, it is desirable to find out an optimum catalyst loading for efficient decolorization. A series of experiments was carried out by varying the amount of catalyst from 0 to 12 g/L with a dye concentration of 20 mg/L, solution pH of about 7, and solar irradiation time of 2 h. The decolorization efficiency of BGY for various photocatalyst loadings is illustrated in Figure 5. The results show that an increase in the catalyst loading from 1 to 6 g/L increases the dye decolorization sharply from about 59% to about 98%. This is due to the fact that an increase in catalyst loading increases the active sites on the surface of the catalyst, which in turn, increases the adsorption of number of BGY^{2-} species and, hence, the proportion of the excited species by the incoming photons. Therefore, the rate of decolorization increases with higher catalyst loading. With the increase of the catalyst loading from 6 to 8 g/L, the decolorization rate is almost constant, suggesting that an optimal level for catalyst effectiveness has been attained for this amount of BGY (20 mg/L). Further increase of the catalyst loading beyond 8 g/L resulted in a decrease in decolorization rate of the dye. This phenomenon may be due to the hindrance and blocking of light penetration caused by the excessive amount of

the photocatalyst particles [30]. On the other hand, at high concentrations of catalysts, particle aggregation is significant, reducing thereby the active sites on the catalyst surface. Therefore, the decolorization rate also reduces. So, the optimal range of catalyst is 6 to 8 g/L with dye concentration of 20 mg/L. Since the maximum decolorization of BGY is observed with 6 g/L of ZnO-TiO_2 photocatalyst, the other experiments were performed using this amount of the composite. It is to be noted that no significant decolorization of BGY under solar irradiation was observed in the absence of the photocatalyst, indicating that the dye was resistant to self photolysis in aqueous solution under sunlight.

Effect of pH on the decolorization

The pH of the BGY solution is an important parameter for metal oxides that act as efficient photocatalysts because the environment pH influences the surface charge of the photocatalysts [31-33]. Figure 6 shows that the decolorization efficiency of BGY increases with the increasing solution pH from 6 to 7.5 (maximum decolorization efficiency, 98%, at pH about 7). A further increase of pH beyond 7.5 results in the reduction of decolorization rate. The pH effect on the photodecolorization of the dye in the presence of ZnO-TiO_2 can be explained on the basis of the zero point charge (pH_{zpc}) of the nanocomposites. The pH_{zpc} of pristine ZnO is approximately 8.5 and that for TiO_2 (rutile) is about 5.3; therefore, about 7 could be the resultant pH_{zpc} of the nanocomposite. Thus, the surface of the catalyst becomes positively charged at lower pH, but at higher pH, it becomes negatively charged. However, the photocorrosion of ZnO [32] may take place at lower pH (< 6.0). All these considerations make pH of the composite suspension around 7 an ideal environment for optimum solar decolorization of BGY.

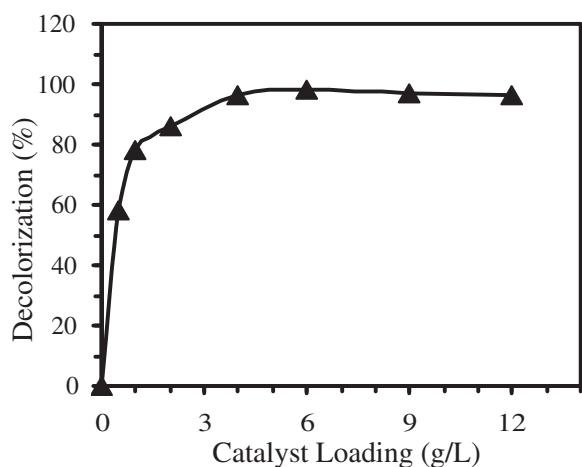


Figure 5 Effect of catalyst loading on decolorization of BGY. BGY = 20 mg/L, pH approximately 7; illumination time = 2 h.

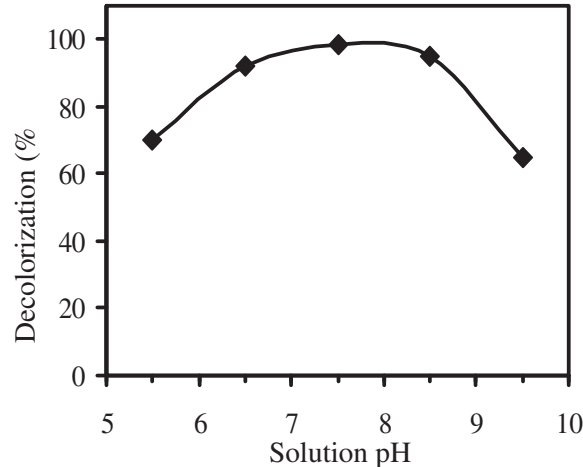


Figure 6 Effect of initial solution pH on decolorization of BGY. BGY = 20 mg/L, catalyst loading = 6 g/L, illumination time = 2 h.

Effect of initial dye concentration on the decolorization

Figure 7 shows the effect of initial dye concentration on the decolorization of BGY. The results suggest that the decolorization efficiency of BGY decreases with increasing initial concentration of the dye. Similar results have been obtained in other studies on photodecolorization of dyes including BGY [5-7,34-36]. As the initial concentration of a dye increases, the color of dye solution becomes deeper which results in the reduction of penetration of light to the surface of the catalyst, decreasing the number of excited dye molecules.

At low initial dye concentration, the rate expression is given by the equation below:

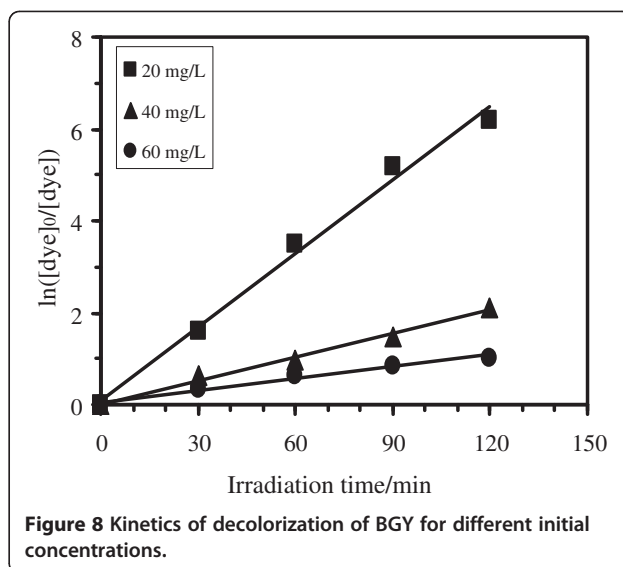
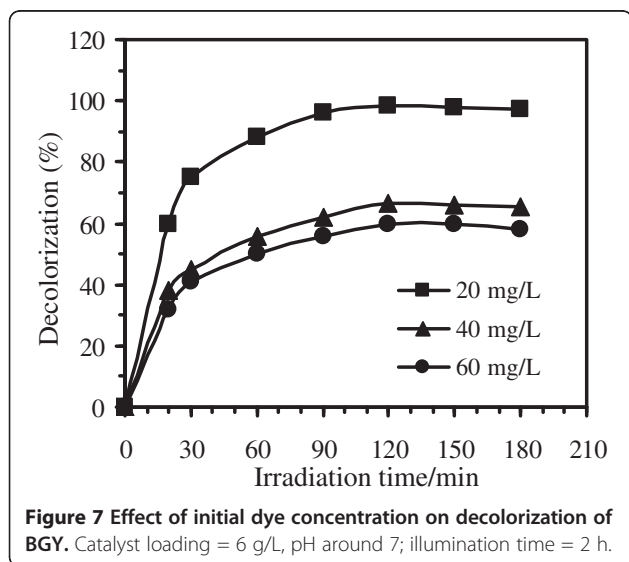
$$-\frac{d[\text{dye}]}{dt} = k_{app}[\text{dye}].$$

Integration of the above equation yields as follows:

$$\ln[(\text{dye})_0/(\text{dye})] = k_{app}t$$

where k_{app} is the pseudo-first-order rate constant. $(\text{dye})_0$ is the equilibrium concentration of the BGY solution after adsorption, and it is taken as the concentration of dye solution at solar irradiation time ' $t = 0$ ' for kinetic analysis. (dye) is the concentration of BGY at solar irradiation time t , and t is the solar irradiation time (min).

A plot of $\ln[(\text{dye})_0/(\text{dye})]$ versus t for the BGY photodecolorization is shown in Figure 8. When the initial dye concentration changed from 20 to 60 mg/L, the correlation constant (R^2) for each of the fitted lines are 0.9907, 0.9915, and 0.9842, respectively corresponding to the calculated pseudo-first-order rate constants 0.0533, 0.0169, and 0.0086/min. The decrease in k_{app} can be explained in terms of the less availability of photons as the color of the solution gets more intense [37] and the



consequent decrease in the number of excited BGY molecules on catalyst surface.

Effect of the additives on the decolorization

In dyeing process, some additives such as surfactants, anti-static agents, salts, etc. are often used. Therefore, wastewaters from dyeing/textile industries contain considerable amount of surfactants, antistatic agent, or salts. Here, sodium dodecyl sulfate (SDS) as anionic surfactant and Triton X-100 (BDH Chemicals Ltd.) as non-ionic surfactant were used as additives to observe their effects on the decolorization efficiency of BGY. Three groups of controlled experiments were carried out under the following conditions (BGY = 20 mg/L; catalyst loading = 6 g/L; pH around 7; irradiation time = 2 h): (1) dye solution without any additives, (2) dye solution with 10^{-4} M SDS, and (3) dye solution with 10^{-4} M Triton X-100 (BDH Chemicals Ltd.). The results showed that the addition of SDS inhibited decolorization efficiency of BGY; on the other hand, no significant change in decolorization efficiency was observed upon the addition of Triton X-100 (data not shown, BDH Chemicals Ltd.). The retarding effect by SDS can be explained by considering a competitive adsorption of the anionic dye and anionic surfactant ions on the positively charged surface of the photocatalyst.

Photodecolorization of wastewater

The nanocomposite containing 3:1 molar ratio of ZnO/TiO₂ was used to decolorize wastewater of a textile factory under the following conditions: catalyst loading = 6 g/L; pH about 7, and solar irradiation time = 2 h. The textile factory used BGY and Orange G(an anionic dye) for dyeing, and about 80% photodecolorization was achieved (data not shown). Lower decolorization of the wastewater by the ZnO-TiO₂ nanocomposite may be due to the presence of

other materials such as surfactants, antistatic agents, salts, etc. which inhibits the decolorization efficiency of the ZnO-TiO₂ nanocomposites.

Conclusions

The feasibility of photocatalytic decolorization of BGY and wastewater of a textile factory, using ZnO-TiO₂ nanocomposite as photocatalyst in the form of suspension by irradiation with solar light, has been investigated. The experimental results show that the ZnO-TiO₂ nanocomposites with ZnO/TiO₂ molar ratios of 3:1 and 1:1 were the most efficient photocatalysts. The optimal conditions for about 98% decolorization of BGY are as follows: catalyst loading = 6 g/L, BGY = 20 mg/L, solution pH around 7, and solar irradiation time = 2 h. The addition of additives, e.g., sodium dodecyl sulfate (SDS), salts, etc. is found to inhibit the photocatalytic decolorization of BGY. The photocatalytic decolorization of the dye with ZnO-TiO₂ nanocomposites obeyed pseudo-first-order kinetics. Finally, it is concluded that the nanocomposites can be used as photocatalyst for the remediation of industrial wastewater containing dyes.

Competing interests

The authors declare that they have no competing interests.

Authors' contributions

MTS is an undergraduate student, and his work is the basis of the paper. MAH and AJM are the supervisor and co-supervisor, respectively. NMB and IMI have helped with the instrumental analysis and final preparation of the paper. All the authors read and approved the final manuscript.

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