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Photocatalytic Degradation of Alizarin Red S and Bismarck Brown R Using TiO_2 Photocatalyst

Research Article

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Abstract

TiO_2 photocatalyst was prepared by the sol-gel method at a temperature of 300˚C and characterized by SEM with EDS and FTIR. The photocatalytic degradation of Alizarin Red S (ARS) and Bismarck Brown R (BBR) was carried out using prepared TiO_2 photocatalyst irradiated with 16 W UV light source. The effect of various parameters, i.e., photocatalyst concentration, dye concentration, and pH of the solution on the percentage of degradation of selected dyes has been examined. The kinetic analysis of photodegradation of ARS and under different initial concentration followed the Langmuir-Hinshelwood model. TiO_2 can be an excellent photocatalyst candidate for the degradation of the selected dyes under UV light irradiation.

Keywords: TiO_2 nanoparticles; sol-gel method; Alizarin Red S; Bismarck Brown R; Langmuir-Hinshelwood model

Introduction

Textile, paint, leather, detergent, paper, agrochemical and other industrial dyes constitute one of the largest groups of organic compounds that represent an increasing environmental risk. Over 7×10^5 tons and above 10,000 different types of dyes and pigments are produced worldwide yearly. About 10-20% of the total world production of dyes is lost during dyeing process and is accordingly released in the textile wastewaters. The release of wastewaters that contain high concentration of dyes is a major trouble for the industry as well as a threat to the environment [1,2]. Dyes are extensively used in the textile industry, particularly those involved in finishing processes are among the major cause of water pollution. Due to the high concentration of organics in the effluents and the higher stability of modern synthetic dyes, these are not easily treated by chemical and biological methods and are released in wastewater. This is mainly due to the fact that dyestuffs are designed to be resistant to chemical loss and light induced fading. In addition, the stability of their molecular structure also makes them resistant to aerobic microbial degradation [3].

Several physicochemical and biological methods currently exist for the treatment of dye industrial wastewaters. Physical methods basically do not degrade the pollutants but only transfer them from the liquid phase to the solid or another liquid phase, thereby causing secondary pollution [4]. Biological methods are more environmental friendly and easier to implement, but they have a major drawback of production of sludge and when the volume to treat is huge, recycling is necessary [5]. Furthermore, many of the dyes belong mostly to the non-biodegradable and unmanageable type of water pollutants [1,6]. Thus, there is a great challenge towards the treatment of textile effluents.

Therefore, new methods for the treatment of textile effluents, which should be cheaper and more efficient are being explored. Recently, the development of advanced oxidation processes in wastewater treatment research seems to complete these criteria. Heterogeneous

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photocatalysis which employs only suitable semiconductor and light source appeared as a promising destructive technology leading to the complete removal of most of the pollutants [3,7,8]. Semiconductor materials employed in photoconversion systems present a moderately wide energy gap between the conduction band (CB) and the valence band (VB), this separation being known as the band-gap energy (E_{gap}). The absorption of energy of certain wavelengths by a semiconductor promotes electron transfer from the VB to the CB, leaving vacancies in the VB called holes. The photogenerated electron/hole (e^-/h^+) pair promotes the reduction and oxidation of species adsorbed at the surface of the semiconductor and induces oxidative degradation of species in solution through radical reactions [9,10].

Photocatalytic reaction using a semiconductor such as Titanium dioxide as photocatalyst is one of the advanced cost effective, clean and green technology which has attracted considerable interest for the termination of toxic organic and inorganic pollutants from wastewater [2,11]. The ability of this technique to completely degrade organics into water and carbon dioxide, without generating any harmful byproducts has popularized its role as a waste water purifier [2-12]. Titanium dioxide which is a metal oxide semiconductor is the most favored material in this technique, since it is chemically and biologically inactive, cheap, photostable and very photoactive, although it has a large energy gap (Eg) (3.2 eV) and only absorbs in the UV region.

Photocatalytic processes occur according to the following proposed mechanism: Irradiation of TiO_2 particles with photons of energy equal to greater than the band gap energy ($h\nu > Eg=3.2$ eV; $\lambda < 390$ nm) results in the promotion of an electron from VB to the CB of the particle, The outcome of this process is the region of positive charge in the VB, termed as hole (h^+) and a free electron (e^-) in the CB (Eq. (1)):

$$TiO_2 + h\nu \rightarrow TiO_2 (h^+ + e^-) \quad (1)$$

The photo generated holes move around to the particle surface to react with the surface bound hydroxyl groups (OH^-) and water molecules (H_2O) to form hydroxyl ($OH^\bullet$) radical (Eqs. (2) and (3)).

$$h^+ + OH^- \rightarrow H^+ + OH^\bullet \quad (2)$$

$$h^+ + H_2O \rightarrow H^+ + OH^\bullet \quad (3)$$

The photo generated electrons react with adsorbed molecular O_2, reducing it to superoxide radical anion $O_2^{-\bullet}$ (Eq. (4)):

$$O_2 + e^- \rightarrow O_2^{-\bullet} \quad (4)$$

In the absence of an electron acceptor, most likely electron-hole recombination occurs. The presence of oxygen prevents recombination by catching electrons through the formation of superoxides ions, thereby protecting electron neutrality within the TiO_2 molecule. The final product of the reduction is commonly hydroxyl radicals ($OH^\bullet$). The hydroxyl radicals are known to be powerful oxidizing agents. During photocatalytic reactions, they can react with organic compounds and bacterial species adsorbed very close to the semiconductor surface, resulting in complete degradation into small inorganic species (Eqs. (5) and (6))

$$OH^\bullet + RH \rightarrow H_2O + R^\bullet \quad (5)$$

$$R^\bullet + O_2 \rightarrow ROO^\bullet \rightarrow CO_2 \quad (6)$$

Dye stuffs from residual textile treatment water have been treated for the removal by this photodegradation technique [2, 13-18].

Alizarin Red S (ARS) and Bismarck Brown R (BBR) which are model compounds selected for this study, as they are water-soluble dyes and are used extensively as a coloring agent for fibers, leather, etc. [19-21]. The chemical structure of selected dyes ARS and BBR are shown in Figure 1. The wide use of this dye in industry and its water soluble nature maximize its chances of being present as a contaminant in industrial effluents. This study was conducted with the aim of preparing photocatalyst TiO_2 nanoparticles by sol-gel method and examine the photodegradation kinetics of ARS and BBR. Degradation experiments were carried out with UV light/TiO_2 at different illumination time, TiO_2 doses, initial dye concentrations, and initial pH values in a photo reactor "Heber" photo reactor.

Materials and methods

Materials

Tetrabutyl titanate, ethanol Absolute AR (99.9%) and glacial acetic acid were purchased from Sigma Aldrich, India, were used for the preparation of TiO_2. Alizarin red S and Bismarck brown R of commercial grade were purchased from Nice chemicals Pvt. Ltd, Cochin, India. Hydrochloric acid and sodium hydroxide were also obtained from Sigma Aldrich India. All solutions and reaction mixtures were prepared in MilliQ water.

Preparation of TiO_2 catalyst

The preparation of TiO_2 nanophotocatalysts were carried out by sol-gel method. In this method, 20 mL tetrabutyl titanate and 4 mL acetic acid were added into 26 mL of absolute ethanol under continuous stirring condition to obtain solution A. 8 mL deionized water, 12 mL absolute ethanol and 12 mL acetic acid were mixed together to obtain solution B. Then, solution B was added drop wise into solution A under stirring. The obtained solution was sealed and stirring was continued for another 30 min at room temperature. The

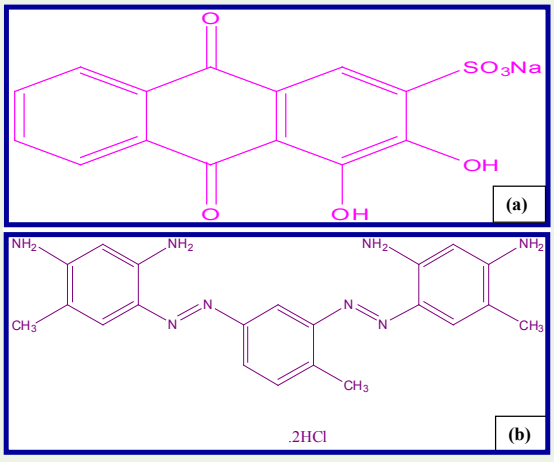

Figure 1: Chemical structures of selected dyes (a) ARS and (b) BBR.

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resultant gel was aged at room temperature for 24 h and dried in an oven at 100°C for 36 h. After grinding, the gel was heat-treated in a furnace at 300°C for 3 h. and white crystalline TiO_2 nanoparticles were obtained at the end of the process [22].

Characterization

The synthesized photocatalyst was characterized using scanning electron microscopy (SEM) with Energy Dispersive Spectrometry (EDS) analysis (Vega3Tescan, Brucker (Germany). Infrared (IR) studies of TiO_2 were performed on a JASCO-460 plus FTIR spectrometer. UV-visible spectra of selected dyes were recorded using Perkin Elmer Lambda 35 (USA).

Photodegradation of dyes under UV light in batch reactor

The photocatalytic degradation of ARS and BBR was performed in aqueous medium in a batch reactor. A cylindrical Pyrex glass photochemical reactor of 30 cm × 6 cm (height × diameter), provided with water circulation arrangement to maintain the temperature in the range 25-30°C, was used in all the experiments. The photodegradation was carried out using "Heber" photo reactor (16W, λ-max 251nm mercury lamp) placed inside a Pyrex glass jacket, thermostated by water circulation, and immersed in the dye solution contained in the batch reactor. The catalyst was maintained in suspension by stirring. Previously, the ARS and BBR solutions (50 ml, 50 mgL^{-1}) containing 100 mg of photocatalyst were magnetically stirred in the presence of light for 60 min. Aliquots were withdrawn at specific time intervals and analyzed after filtration to remove the catalyst. The variation of the ARS and BBR dye concentrations as a function of irradiation time was determined using a UV-Visible spectrometer "Perkin Elmer Lambda 35" (USA) and the absorbance was measured at 517 nm and 465 nm against blank respectively

Analytical methods

The concentration of the dyes ARS and BBR in the reaction mixture at different reaction time was monitored by UV spectrophotometry. The photodegradation efficiency of ARS and BBR was defined as follows:

$$\% = (C_0 - C_f) / C_0 \, x \, 100 \qquad (7)$$

Where C_0 is the initial concentration of dyes and C_f is the concentration of dyes at certain reaction time t (min).

Results and Discussion

Characterization of prepared TiO_2 nanoparticles

SEM micrograph of prepared TiO_2 nanoparticles (Figure 2) shows that the material is formed by an agglomeration of nearly spherical particles having an average diameter of 60 nm. EDS analysis was used to characterize the elemental composition of the TiO_2 nanoparticles. A typical EDS pattern of TiO_2 nanoparticles is shown in Figure 3. Figure 4 shows the IR spectra of prepared TiO_2 nanoparticles. The broad band observed at 3431 cm^{-1} is assigned to the asymmetrical and symmetrical stretching vibrations of hydroxyl group (-OH) and the band at 1627 cm^{-1} is corresponds to deformative vibration of Ti-OH stretching modes. This band is an evidence of the adsorbed water on the TiO_2 surface [23,24]. The band at 1627 cm^{-1} is assigned

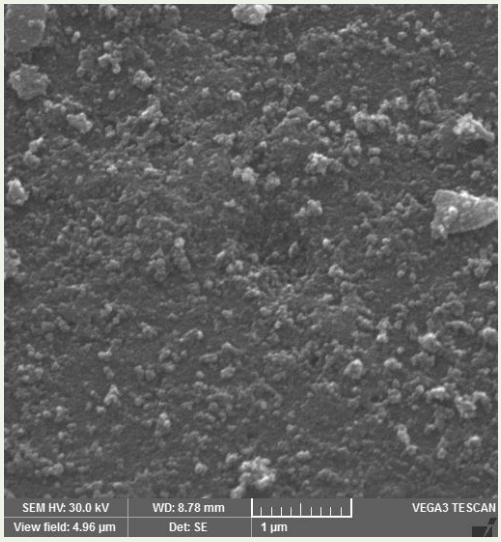

Figure 2: SEM micrographs of synthesized TiO_2 nanoparticles.

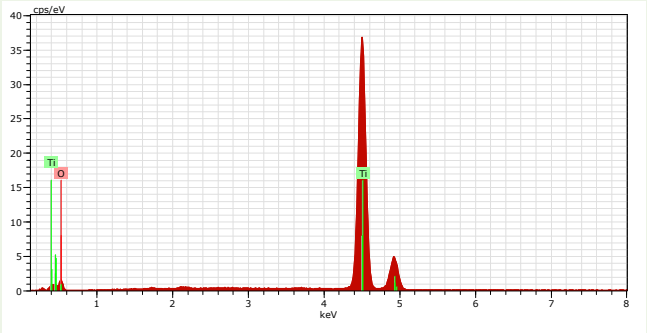

Figure 3: EDS pattern of synthesized TiO_2 nanoparticles.

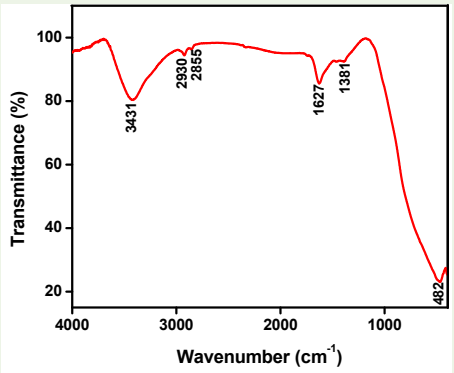

Figure 4: FTIR spectrum of synthesized TiO_2 nanoparticles.

to C=C bond which comes from the butyl groups after the sample was heat-treated at 300°C. The bands at 2930, 2855 cm^{-1} and 1381 cm^{-1} correspond to the stretching and bending modes of C-H bonds which come from the residual butyl group in the prepared TiO_2. The band at 482 cm^{-1} corresponds to the Ti-O bending mode of TiO_2 [22].

UV-Visible Spectral Studies

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Figure 5 shows the ultraviolet-visible (UV-Vis) spectrum of selected dyes ARS and BBR giving peaks at 517 nm and 465 nm against the blank respectively. The variation of ARS and BBR dye concentrations as a function of irradiation time was determined using a UV-Visible spectrometer.

Effect of illumination time

The relationship between the photodegradation efficiency of ARS and BBR and the illumination time was investigated by fixing the amount of TiO_2 as 100 mg. The result is shown in Figure 6. From the figure it can be seen that the photodegradation efficiency increased with the increase in the illumination time. The photodegradation of ARS and BBR increased from 19.2% to 94.1% and 10.0% to 91.4% respectively, when the illumination time increased from 10 to 80 min. The $OH^{\bullet}$ is widely accepted as primary oxidants in heterogeneous photocatalysis. The oxidizing power of the $OH^{\bullet}$ radicals is strong enough to completely oxidize dyes adsorbed on the surface of TiO_2 into CO_2, H_2O and other mineral acids as reported [25-27]. As seen in the Figure 6 when the illumination time was longer than 60 min, only small enhancement of photodegradation efficiency was observed. The probable reason is that, a large number of small organic molecules are produced by photodegradation with the increase of the irradiation time and these small organic molecules adsorb on the surface of TiO_2, resulting in the decreased formation of $OH^{\bullet}$ radicals that attack the dyes, and therefore increase in illumination time does not lead to greater photodegradation efficiency of ARS and BBR.

Effect of catalyst dose

The effect of photocatalyst dose on the degradation efficiency of ARS and BBR was investigated under UV light employing different dosages of TiO_2 nanoparticles varying from 25 mg to 150 mg at a fixed dye concentration of 10 mgL^{-1}. The degradation efficiency of catalyst load on ARS and BBR is illustrated in Figure 7, which reveals that the efficiency increased greatly by increasing catalyst loading from 25 mg to 100 mg and thereafter the rate of degradation remains almost constant. Maximum degradation was observed with 100 mg dose of TiO_2. Similar results were obtained earlier by other workers [2,28]. The increase in the amount of catalyst increases the number of active sites on the TiO_2 surface that in turn increases the number of $OH^{\bullet}$ and $O_2^{-\bullet}$ radicals [2,29]. Optimum catalyst loading found to dependent on initial solute concentration. At the same time with high dose of photocatalyst turbidity of the suspension increases, there will be a decrease in the penetration of UV light and hence photoactivated volume of suspension decreases [2,30]. Thus it can be concluded that a higher dose of catalyst may not be useful both in view of aggregation as well as reduced irradiation field due to light scattering. Therefore, catalyst dose of 100 mg was fixed for further studies.

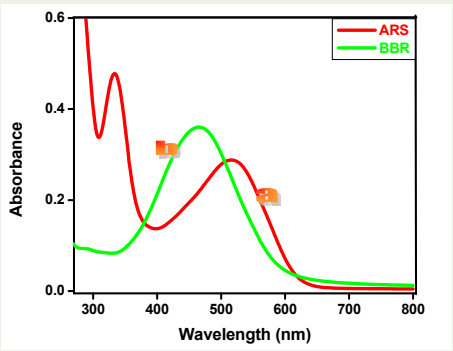

Figure 5: UV-vis spectra of the selected dyes (a) ARS and (b) BBR.

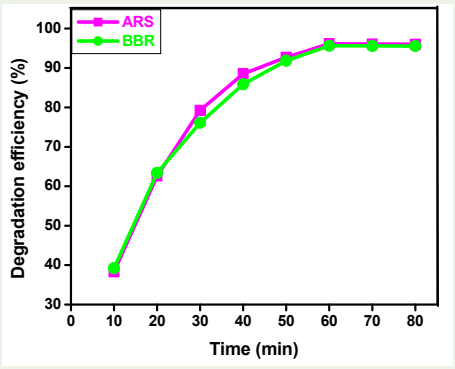

Figure 6: Effect of illumination time on the degradation of ARS and BBR dyes.

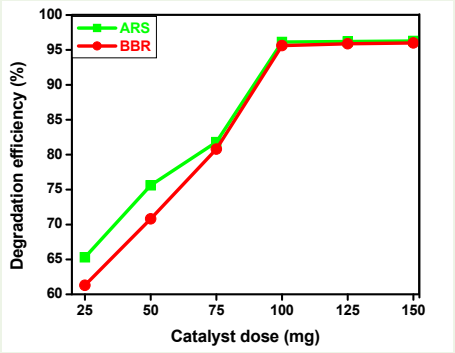

Figure 7: Effect of TiO_2 dose on the degradation of ARS and BBR dyes.

Effect of dye concentration

By varying the initial concentration from 10 to 50 mgL^{-1} at constant catalyst load (100 mg), its effect on the degradation rate was determined, and the results are shown in Figure 8. As seen in the figure, degradation efficiency of ARS and BBR decreased when increase the dyes concentration was increased. This negative result can be explained as follows; as the dye concentration is increased, the equilibrium adsorption of dye on the catalyst surface active sites increases; hence competitive adsorption of OH^- on the same sites decreases, meaning a lower formation rate of $OH^{\bullet}$ radical, which is the principal oxidant necessary for a high degradation efficiency. On the other hand, considering the Beer-Lambert law, as the initial dye concentration increases, the path length of photons entering the solution decreases, resulting in lower photon adsorption on catalyst particles and, consequently, a lower photodegradation rate.

Effect of pH

An important parameter of photocatalytic reactions on the nano TiO_2 surfaces is pH of the solution, since it states the surface charge

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properties of the photocatalyst. The photodegradation efficiency of both ARS and BBR dyes was studied at different pH range from 3 to 11. The photodegradation efficiency of selected dyes as a function of reaction pH is shown in Figure 9. Photodegradation efficiency both of the dyes decreased with the increase in reaction pH and the highest efficiency was observed at pH 3. The explanation of pH effect on the photocatalytic process is very difficult because of its multiple roles, electrostatic interactions between the photocatalyst surface, solvent molecules, substrate and charged radicals formed during the reaction process [19,31]. The ionization state of the surface of the photocatalyst can be protonated and deprotonated under acidic and alkaline conditions respectively, as shown in the following equations,

$$TiOH + H^+ \rightarrow TiOH_2^+ \quad (8)$$

$$TiOH + OH^- \rightarrow TiO^- + H_2O \quad (9)$$

The zero point charge (pzc) of the TiO_2 is widely reported at pH~6 [29]. Thus, TiO_2 surface will remain positively charged in acidic medium (pH < 6.25) and negatively charged in alkaline medium (pH > 6.25).

Higher uptakes of ARS obtained at lower pH values may be due to electrostatic attractions between ARS and more protonated adsorption sites available. A lower adsorption at higher pH values may be due to the abundance of OH^- ions and because of ionic repulsion between the negatively charged surface and the anionic ARS molecules [19]. The better efficiency for the degradation of BBR at lower pH may be due to the fact that the lone pairs of electrons present on the nitrogen atoms of amino groups can be protonated and deprotonated under acidic and basic conditions. Actually the photocatalytic oxidation seems to be favored in the structural orientation of the molecule, when it is protonated under lower pH values [31].

Kinetic analysis

Photocatalytic degradation of ARS and BBR was performed in varying initial concentrations (10, 20, 30, 40 and 50 mgL^{-1}) for kinetic analysis. Heterogeneous photocatalysis process is very complicated, involving the function of many factors and even their mutual effect [32]. Figure 10 demonstrates the relationship between different initial concentrations of both ARS and BBR and time. It was found that photodegradation of ARS and BBR followed the pseudo first-order kinetic expression as can be seen from Figure 11. Table 1 shows the pseudo-first-order rate constant k_{obs}, and half-life $t_{1/2}$ for photocatalytic degradation of ARS and BBR at different initial concentrations.

The influence of initial concentration of ARS and BBR on the photocatalytic degradation rate is described by pseudo-first order kinetic model for heterogeneous photocatalysis involving Langmuir-Hinshelwood expression [32-34]. The Langmuir-Hinshelwood model for dye degradation can be written as follows:

$$r = -\frac{d[Dye]}{dt} = k\frac{K_{Dye}[Dye]}{1 + K_{Dye}[Dye]_0} = k_{obs}[Dye] \quad (10)$$

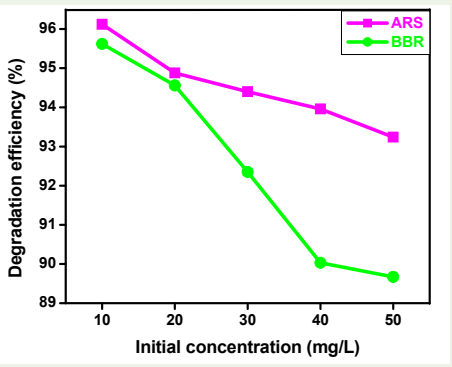

Figure 8: Effect of initial concentration on the degradation of ARS and BBR dyes.

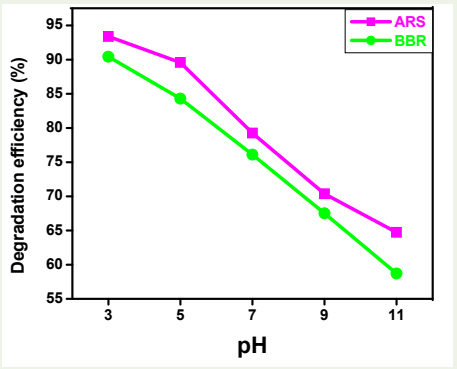

Figure 9: Effect of pH on the degradation of ARS and BBR dyes.

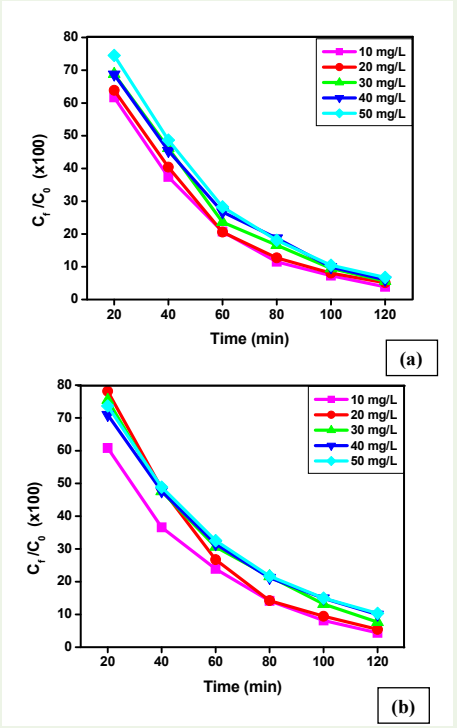

Figure 10: The relationship between different initial concentrations of (a) ARS and (b) BBR and time (pH 7.00, TiO_2=100 mg).

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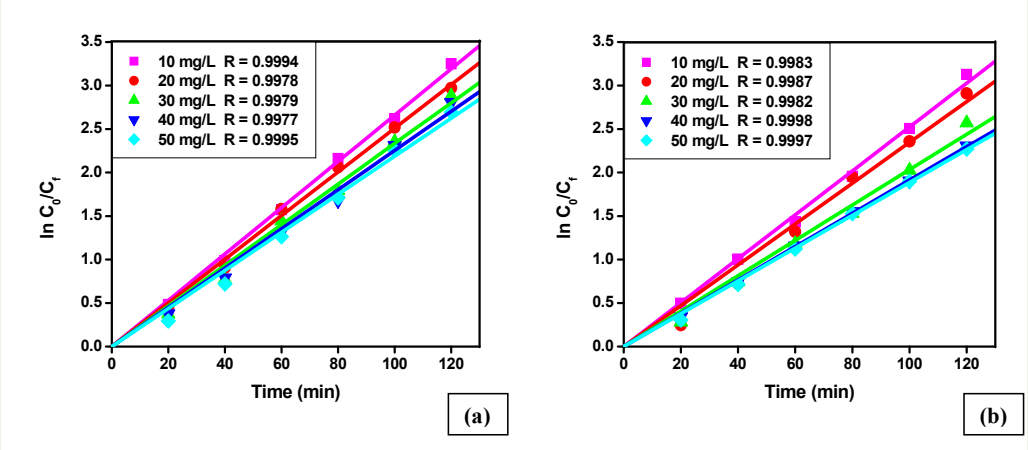

Figure 11: The relationship between ln C_0/C_f and time at different initial concentrations of (a) ARS and (b) BBR.

Table 1: The values of the k_{obs} and half-lives of ARS and BBR under different initial concentrations.

Initial concentration of dyes (mgL^{-1})	k_{obs} (min^{-1})		$t_{1/2}$ (min)		R	
	ARS	BBR	ARS	BBR	ARS	BBR
10	0.0266	0.0252	25.5844	13.2942	0.9994	0.9983
20	0.0251	0.0235	27.9933	14.2884	0.9978	0.9987
30	0.0234	0.0203	28.8610	16.1756	0.9979	0.9982
40	0.0225	0.0191	29.6371	18.0392	0.9977	0.9998
50	0.0219	0.0189	30.8795	18.3173	0.9995	0.9997

Table 2: values of the k and K_{Dye} of ARS and BBR.

Dyes	k (mgL^{-1} min^{-1})	K_{Dye} (Lmg^{-1})	R
ARS	4.8144	0.0058	0.9887
BBR	2.7463	0.01	0.9682

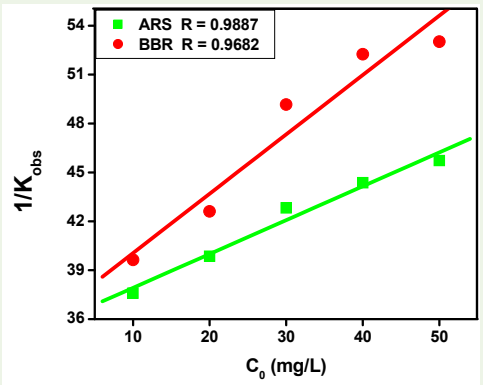

Figure 12: The relationship between $1/k_{obs}$ and different initial concentrations of ARS and BBR.

$$\frac{1}{k_{obs}} = \frac{1}{kK_{Dye}} + \frac{[Dye]_0}{k} \quad (11)$$

where $[Dye]^0$ is the initial concentration of dyes (mgL^{-1}), K_{Dye} the Langmuir–Hinshelwood adsorption equilibrium constant (Lmg^{-1}), k the rate constant of surface reaction ($mgL^{-1}min^{-1}$), and k_{obs} pseudo-first-order rate constant. According to Eq. 11, $1/k_{obs}$ holds a straight line relation with the parameter $[Dye]^0$. When initial concentrations were plotted versus $1/k_{obs}$, the rate constant of surface reaction and the adsorption equilibrium constant were calculated (Table 2) to be k = 4.8144 mgL^{-1} min^{-1} for ARS and 2.7463$mgL^{-1}min^{-1}$ for BBR and K_{Dye} = 0.0058 Lmg^{-1} and 0.01 Lmg^{-1} for ARS and BBR respectively (Figure 12). The regression coefficient R was 0.9887 and 0.9682 for ARS and BBR respectively, which suggest the photodegradation of both dyes by the TiO_2 fits the Langmuir-Hinshelwood kinetic expression well.

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Conclusions

TiO_2 photocatalyst can be efficiently applied for the degradation of Alizarin Red S and Bismarck Brown R dyes. Following conclusions are drawn from the results of the present study. The photocatalytic degradation process increased to some extend with increase in catalyst dose but decreased with increase in dye concentration. The maximum degradation was observed with 100 mg dose of TiO_2. Photodegradation efficiency decreased with increase in reaction pH and the highest efficiency was observed at pH 3. The percentage of photodegradation of ARS was 94.1% and BBR was 91.4% when the solution was irradiated by the 16 W mercury lamp for 60 min. The kinetic analysis of photodegradation of ARS and BBR under different initial concentrations followed the Langmuir–Hinshelwood model. The rate constant of surface reaction and the adsorption equilibrium constants were calculated to be k = 4.8144 mgL^{-1} min^{-1} for ARS and 2.7463$mgL^{-1}min^{-1}$ for BBR and K_{Dye} = 0.0058 Lmg^{-1} and 0.01 Lmg^{-1} for ARS and BBR respectively. Prepared TiO_2 can be an excellent photocatalyst candidate for the degradation of the organic contaminants under UV light irradiation.

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