

PHOTO-CATALYTIC DEGRADATION OF BIEBRICH SCARLET DYE USING TiO₂ NANOPARTICLES

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ABSTRACT

Photo-Catalytic Oxidation (PCO) is one of the efficient methods for degradation of the various hazardous organic pollutants in water. The photo-catalytic degradation of diazo dye Biebrich Scarlet (BS) in the wastewaters was studied. The degradation efficiency of the (BS) dye was monitored by UV-Vis spectrophotometer at the visible maximum absorption wavelengths ($\lambda_{max} = 507$). The degradation of dyes depends on several parameters such as pH, catalyst concentration, substrate concentration, H₂O₂ and existing some salts in water. The electron scavenger employed was inorganic oxidant such as H₂O₂. Photo-catalytic activity of TiO₂ was examined by focusing on its enhancement by electron acceptors (S₂O₇²⁻ or S₂O₈²⁻) in the photo-catalytic decomposition of the (BS) dye. The experimental data were analyzed by the Langmuir and Freundlich adsorption models. Equilibrium data fitted very well with the langmuir model.

INTRODUCTION

Adsorption methods are used as high quality treatment processes for the removal of dissolved organic pollutants, such as dyes, from industrial wastewater. The textile, pulp and paper industries are reported to utilize large quantities of a number of dyes, these pollutant may be found in wastewaters of many industries generating significant amounts of colored wastewaters, toxic and even carcinogenic, posing serious hazard to aquatic living organisms the presence of dyes and pigments in water causes considerable damage to microorganisms, aquatic and human life. Azo dyes, are the largest group of synthetic dyes, consists up to 70% of the commercial dyes produced by industries. In general, they are compounds with functional group R-N=N-R', in which R and R' can be either substituted aromatic rings or alkyl groups. Traditional physical methods such as: adsorption (Nasuha, et al., 2010), coagulation, ion flotation (Shakir, et al., 2010), and sedimentation (Zodi, et al., 2010) have been widely applied for purification of wastewaters from these dyes. But, these procedures have some limitations such as producing a secondary waste product which needs to be processed further. Oxidation processes have been also developed for removal of dyes from wastewaters.

It was proved that traditional procedures cannot be effective for degradation of synthetic textile dyes because of the chemical stability of these pollutants. Further more, the stability of azo dyes renders them resistant to biological and even chemical degradation. Numerous studies, have been devoted to use of Photo-Catalytic Oxidation (PCO) for the removal of azo dyes in aqueous solution, particularly, because it is very effective method for this propose. This methodology would normally use a strong oxidizing species such radicals produced in situ, which causes a sequence of reactions thereafter to break down the macromolecules into smaller and less harmful substances (Rauf, et al., 2011; Jiang, et al., 2015), have investigated visible-light photo catalytic degradation of crystal violet using BiO_xCl_y/BiO_mI_n composites. (Huang, et al., 2014), have studied photocatalytic activity of visible-light-responsive photocatalysts BiO_xCl_y/BiO_mBr_n by controlled hydrothermal method. Recently, (Lee, et al., 2014), reported photo catalytic degradation of ethyl violet dye mediated by TiO₂ under an anaerobic condition. (Das, et al., 2014), were investigated oxidation of Rhodamine B in aqueous medium using acid-activated such as MnO₂ as catalysts. (Lee, et al., 2012), were comprehensively studied the degradation efficiency of ethyl violet by HPLC-PDA-ESI-MS and GC-MS techniques.

(Fan, et al.,2011), investigated mechanistic pathways differences between P25-TiO₂ and Pt-TiO₂ mediated crystal violet photodegradation. (Mai, et al., 2010), studied fabrication of porous TiO₂ film on Ti foil by hydrothermal process and its photo catalytic efficiency and mechanisms with ethyl violet dye. The aim of the present work is to study the photo-catalytic degradation of water-soluble Biebrich Scarlet (BS) dye. In this research, a series of experiments have been performed to optimize factors influencing on the photo-catalytic degradation such as: Effect of (BS) dye concentration, effect of TiO₂ loading and effect of pH. In addition, real wastewaters normally contain a lot of salts, such as LiCO₃, NaCO₃, LiCl, MgCl₂, KCl, NaCl, K(NO₃) and Na(NO₃) hence the influence of these salts were examined at the same experimental conditions. Also to improve the photo-catalytic degradation process, an appropriate amount of S₂O₇²⁻ and S₂O₈²⁻ were used as electron acceptors. Equilibrium data well fitted with the langmuir model.

MATERIALS AND METHODS

Materials

Hydrogen peroxide (30% w/w) was obtained from Merck (Darmstadt, Germany). Titanium dioxide P-25 (99.5%, average particle size 30 nm and specific surface of 50 m²/g) from Degussa Corporation. Deionized water was used throughout this work. The salts Li₂CO₃, Na₂CO₃, LiCl, MgCl₂, KCl, NaCl, KNO₃ and Na₂NO₃ were purchased from Fluka (Buchs, Switzerland) and used without further purification. The pH value of solution adjusted using dilute HCl and KOH solutions. Hydrochloric acid was chosen because its effect on the adsorption surface properties of the TiO₂ is negligible. Azo dye, water-

soluble Biebrich Scarlet (BS) dye was prepared from Aldrich (Milwaukee, USA, C.I. number for Biebrich scarletdye is 26905).

Instrumentation

The photo-catalytic reaction was carried out in an inner irradiation type reactor. The photo-catalyst powder was dispersed by magnetic stirrer and after its photocatalytic degradation separated by filter paper. A 300 W high-pressure mercury lamp from Oriel Corporation, Model 8500 (Cheltenham, England) was used as UV-Vis light source (ca. 1800 μW/cm²). The photo degradation process of (BS) dye was monitored using UV-Vis spectrophotometer (Cecil 5000 with model 550) at the maximum absorption wavelengths (λ_{max} =507 nm). The cell was made with Quartz (3 mL content) that it was put it in close space (20 cm) in the front of the UV-Vis lamp.

RESULTS AND DISCUSSION

Kinetics of Photo Degradation

The photo-catalyst was added to dye solution and the suspension was irradiated under UV-Vis light. The photo-catalyst powder was separated after photo-catalytic degradation by filter paper. Then absorption spectra of the dye solutions were recorded and de-colorization process was monitored in terms of change in intensity at λ_{max} of the corresponding dye (i.e., at 507 nm). Fig. 1 presents UV-Vis spectra of the solution before and after 40 min dye degradation by UV-Vis irradiation, in this work was tried to increase degradation efficiency. According to the results of (Shu, et al.,2004), the photo-oxidation reaction is pseudo-first-order with respect to azo dye concentration. This commonly occurs when the pollutant is very dilute in solutions. The $\ln A/A_0$ is

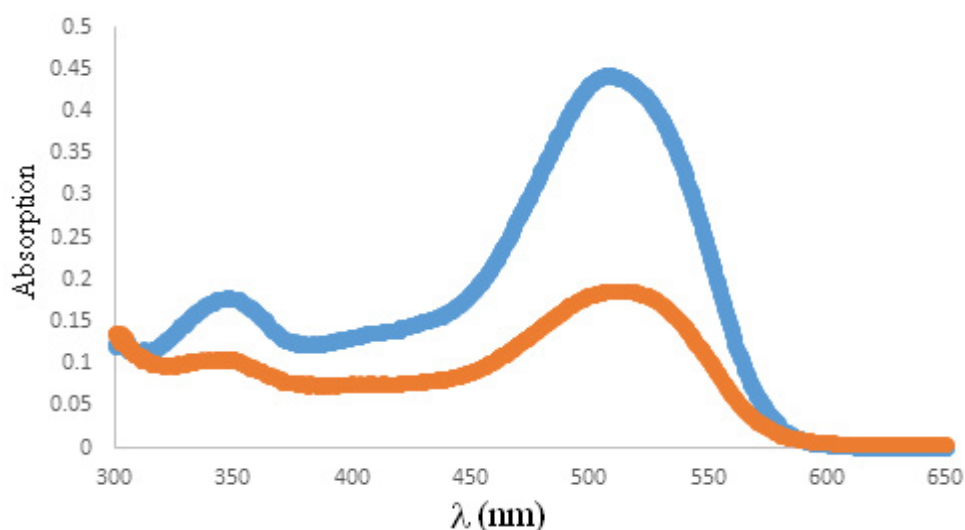


Fig. 1. UV-Vis spectrum of the dye before (upper trace) and after (lower trace) irradiation of the solution.

linearly increased as function of irradiation time for different concentration of BS dye (Equation 1). Fig. 2, shows $\ln A/A_0$ against of exposed time. That means this process is pseudo first order rate constants for different concentrations of dyes. The photo-degradation and photo-bleaching process of BS dye was studied by varying the BS dye concentrations from 1×10^{-5} M to 1.0×10^{-4} M. Where, A_0 and A are the absorbance values of the dye solution before and after irradiation respectively. The degree of decolourization (τ) or %degradation of day was studied by measuring intensity of UV-Vis spectra at the maximum absorption wavelength (Rauf, et al.,2005) (Equation 2).

$$\ln A/A_0 = kt \quad (1)$$

$$\tau = [1 - A/A_0] \times 100 \quad (2)$$

Factors Influencing on the Photo-Catalytic Degradation

The oxidation reaction and efficiency of the photo-catalytic system are highly dependent on a number of experimental parameters that govern the photo-degradation of organic molecule (Chong, et al.,2010). The kinetic of the de-colourization of dye depends on variable factors such as initial dye concentration, TiO₂ loading, pH values, H₂O₂ concentration and the kind of salt present in the solution.

Effect of (BS) Dye Concentration

The initial concentration of dye in a given photo-catalytic reaction is an important case which needs to be taken into account. The influence of dye concentration on the removal of BS dye is presented in Fig. 3. As expected by increasing the initial concentration of dye the degradation efficiency is decreased (i.e. %degradation is ca. 60%

at low concentration while is down to 10% in higher concentration). This is due to increase in initial dye concentration and adsorption sites on the surface area of the TiO₂. Clearly, at low concentration more surface area is available for interaction of dye to the surface of the TiO₂, of course at higher concentration of dye there is not enough surface area for all of dye molecules to access the surface and consequently %degradation is expected to be reduced.

Effect of TiO₂ Loading

Dye degradation is also can be influenced by the amount of sorbent. Increasing in catalyst amount in fact increases the number of active site on the photo-catalyst surface, as a result causing an increase in the number of OH radicals and consequently the photo-degradation is enhanced. It is pertinent to point out that in higher amount of catalyst the solution becomes rather turbid and consequently causes blocking UV radiation and hence reducing degradation processing (Sun, et al.,2008). In this experiment, at the first the solutions were irradiated with sun light for a given time (i.e., 180 min) but with different amounts of TiO₂. Thereafter, solutions were filtered and analyzed by Uv-Vis spectrophotometer for obtaining absorptions values at the wavelength of maximum absorbance ($\lambda_{max} = 507$ nm), results of this experiment are presented in Fig. 4. The extent of sunlight photolysis is highly dependent on UV absorption profiles of the molecule, the surrounding medium, and the emission spectrum of sunlight. Because the energy to break chemical bonds in these kinds of molecules usually ranges from 70 to 120 kcal mol⁻¹, corresponding to light at wavelengths of 250–400 nm, therefore the spectral radiance of sunlight becomes important in the photo degradation of the molecule. Loading TiO₂ can cause to increase

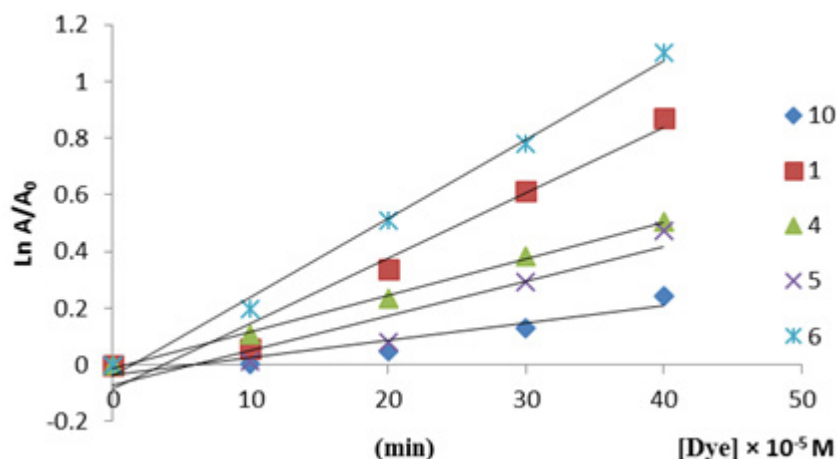


Fig. 2. Kinetics of photo-degradation of BS dye at different initial concentrations. Each solutions was exposed by UV-Vis for 40 min.

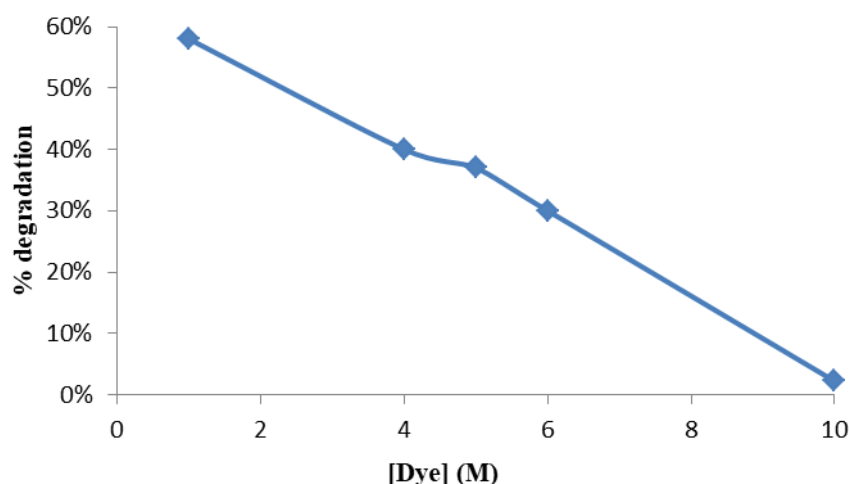


Fig. 3. Plot of %degradation of (BS) dye in various initial dye concentrations.

sunlight photolysis through coupling TiO_2 with the molecule in question (i.e. (BS) dye) and leads to more providing radical species and consequently enhancing degradation of dye. This accelerated degradation can be ascribed to larger surface area of nano particles of TiO_2 . As Fig. 4 shows, increasing catalyst loading causes higher degradation efficiency, i.e., absorption values of the solutions (after photo degradation) is decreased.

Effect of pH

The surface charge of the photo-catalyst and dye can be controlled by pH. To study the effect of pH on photo-degradation, several experiments were performed at 1.0×10^{-5} M dye concentration with different pH values, all solutions were exposed by UV-Vis radiation in the presence of 0.05 g/L TiO_2 and the same temperature. As Fig. 5 shows % degradation of dye is varied as function of pH. Results specified that the highest % degradation is occurred in high acidic solution (i.e. pH 1) and it decreases significantly in neutral and alkaline media. Photo-degradation efficiency of dyes is affected by the pH of the solution in the presence of TiO_2 . The charge surface of TiO_2 particle can be varied in different pH of solution and accordingly it varies the potentials of catalytic reactions. As a result, the adsorption of dye on the surface is altered thereby causing a change in the reaction conditions. Under acidic or alkaline conditions the surface of titania can be protonated or deprotonated respectively according to the following reactions (Equation 3 and 4).



Therefore titania surface would be positively charged

in acidic medium and in contrast is negatively charged in alkaline medium. Titanium dioxide acts as oxidizing active at lower pH because in acidic medium TiO_2 behaves as Lewis acid and on the other hand the anionic dye is aromatic compound with π electron conjugation. As a result, dye molecules (BS) can be efficiently adsorbed on the positively charged catalyst surface. It is pertinent to point out that at lower pH, reduction by electrons in conduction level is important factor for degradation (Soutsas, et al.,2010).. Experiments in this work show a little increasing of photodegradation of dye in higher pH, probably in this condition OH radicals can be easier produced by oxidizing hydroxide group available on TiO_2 surface, (Galindo, et al.,2000), or competitive adsorption with hydroxyl groups (Lachheb, et al.,2002).

Effect of H_2O_2 Concentration

Fig. 6 shows degradation efficiency of BS dye under UV-Vis irradiation at pH 2.0 in the presence of different peroxide concentrations. Clearly degradation process is increased with increasing peroxide concentrations. The degradation of dye can be occurred due to OH generated upon photolysis of H_2O_2 (Equation 5 and 6) (Baxendale, et al.,1957). Therefore it is expected by increasing concentration of H_2O_2 , more OH radicals would be formed, and consequently the degradation process is enhanced. It is noticeable that in high concentration of H_2O_2 the number of OH radicals decrease due to reaction of OH with excess of H_2O_2 and (Equation 7) Fig. 6 Shows %degradation of dye in different peroxide concentrations.



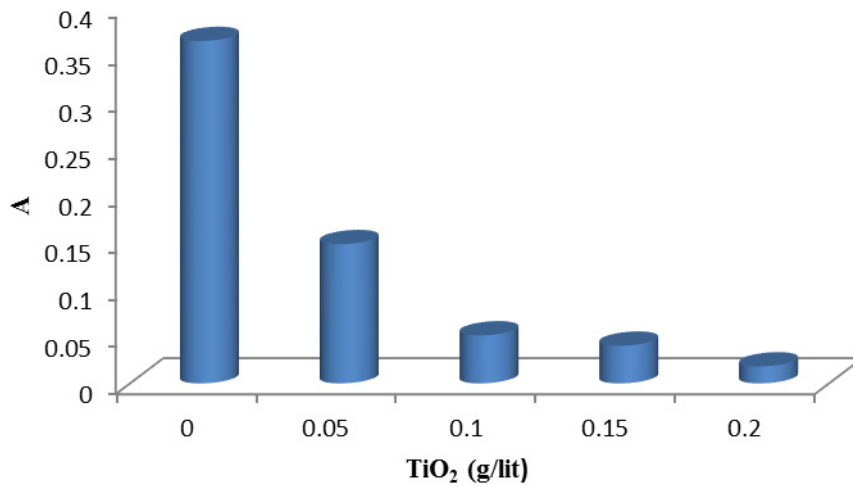


Fig. 4. The absorbents of (BS) dye (in $\lambda_{\max}=507\text{ nm}$) in different TiO₂ concentrations (g/L), solutions were exposed by sun light for 180 min.

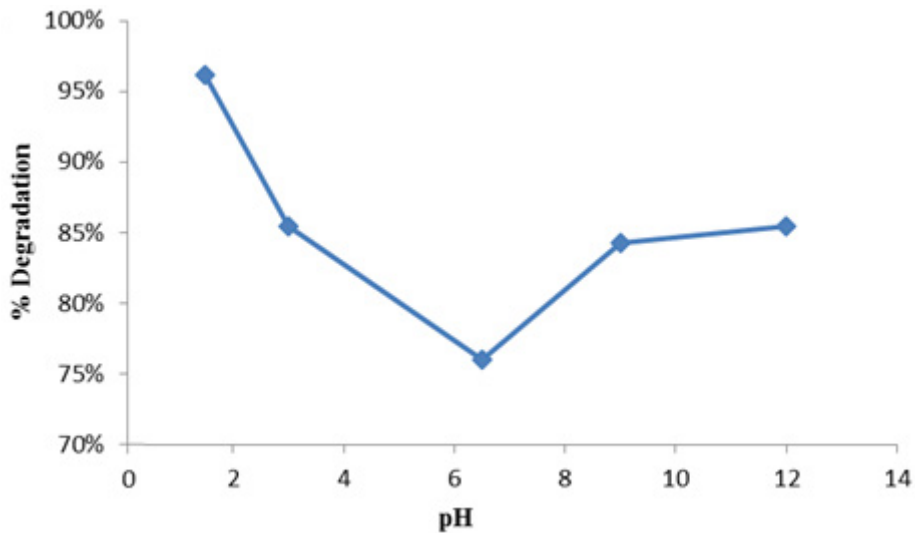


Fig. 5. %degradation of (BS) dye in variable pH. in presence of 0.05 g/L TiO₂.

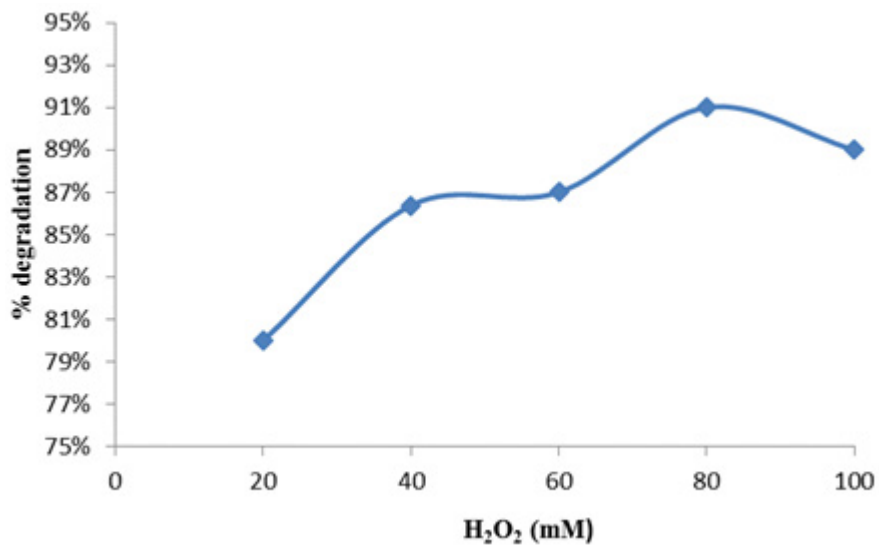
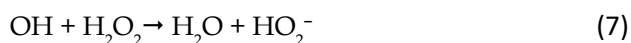


Fig. 6. Plot of %degradation of (BS) dye in different concentration of H₂O₂. Dye concentration= 1×10^{-5} , time of irradiation=8 min and pH=2.0 in present of TiO₂ =0.05 g/L.



Optimum Condition for Dye Degradation

Fig. 7 illustrates %degradation of (BS) dye in three different circumstances i.e., (i) only dye, (ii) in the presence of H_2O_2 and (iii) H_2O_2 plus TiO_2 . Results specified that %degradation of (BS) dye is occurred highly efficient in the presence of H_2O_2 plus TiO_2 . Experiments were performed to find the optimum conditions Results revealed that the optimum conditions as follows, $[\text{H}_2\text{O}_2]=80 \text{ mM}$, $\text{TiO}_2=0.2 \text{ g/L}$ and $\text{pH}=2$.

Adsorption Isotherms

The adsorption isotherm is based on the assumption that every adsorption site is equivalent and independent of whether or not adjacent sites are occupied. Isotherms show the relationship between dye concentration in solution and the amount of dye adsorbed on a the sorbent at a constant temperature. Two common adsorption isotherm models namely Langmuir and Freundlich models are used to investigate the adsorption behavior.

(i) **Langmuir adsorption isotherm** describes quantitatively the formation of a monolayer adsorbate on the outer surface of the adsorbent, and after that no further adsorption takes place. Thereby, the Langmuir represents the equilibrium distribution of metal ions between the solid and liquid phases . The Langmuir isotherm is valid for monolayer adsorption onto a surface containing a finite number of identical sites. The model assumes uniform energies of adsorption onto the surface and no transmigration of adsorbate in the plane of the

surface. Based upon the

$$q_e = \frac{q_m K_L C_e}{1 + K_L C_e} \quad (8)$$

Where, C_e the equilibrium concentration of adsorbate (mg/L), q_e the amount of dye adsorbed per gram of the adsorbent at equilibrium (mg/g), q_m the maximum amount of adsorption dye(i.e., monolayer coverage capacity (mg/g), K_L = Langmuir isotherm constant (L/mg).

Equation 8 can be express as following equation:

$$1/q_e = \frac{1}{q_m} + \frac{1}{q_m K_L C_e} \quad (9)$$

The values of q_m and K_L are calculated from the slope and intercept of the Langmuir plot respectively.

ii) **Freundlich adsorption isotherm** commonly used to describe the adsorption characteristics for the heterogeneous surface. These data often fit the empirical equation proposed by Freundlich:

$$q_e = K_F C_e^{1/n} \quad (10)$$

equilibrium (mg/g). Equation 10 can be written as follows (i.e., Linearizing equation, equation 10):

$$\log Q_{eq} = \log K_f + 1/n \log C_e \quad (11)$$

Where K_f is Freundlich isotherm constant (mg/g), n =adsorption intensity; C_e the equilibrium concentration of adsorbate (mg/L); Q_e the amount of dye adsorbed per gram of the adsorbent at at equilibrium (mg/g). the slope $1/n$, ranging between 0 and 1, is a measure of adsorption intensity or surface heterogeneity, becoming more heterogeneous as its value gets closer to zero. A value for $1/n$ below one indicates a normal Freundlich isotherm while

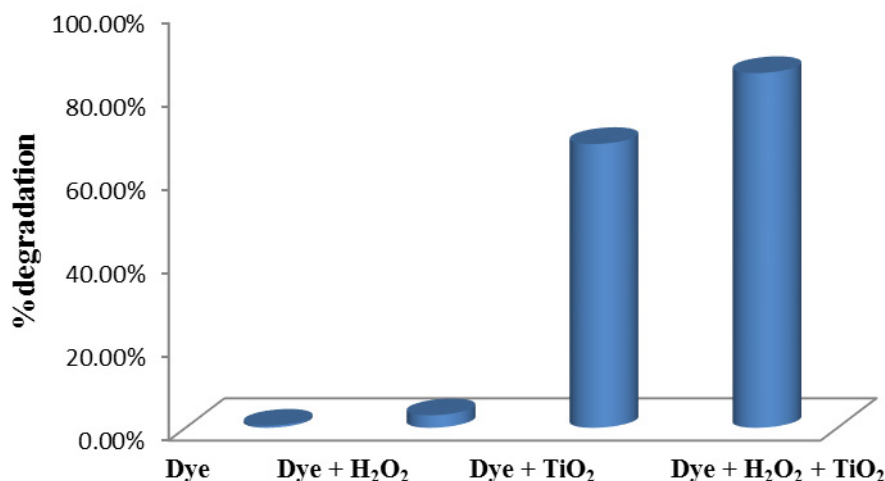


Fig. 7. %degradation of (BS) in different circumstances. $\text{H}_2\text{O}_2]=80 \text{ mM}$, $\text{TiO}_2=0.2 \text{ g/L}$ and $\text{pH}=2$.

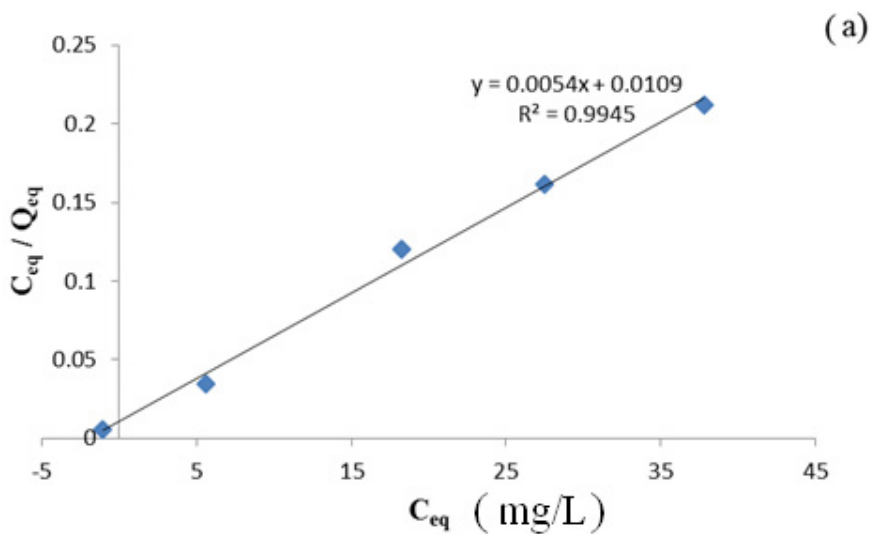


Fig. 8(a). Fit of the linearization to BS dye adsorption isotherms on TiO₂ nano-particles, Langmuir,

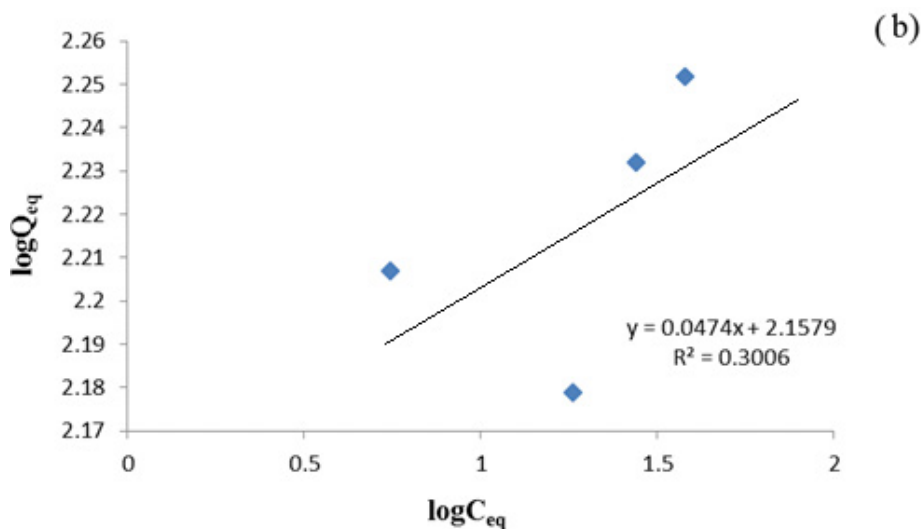


Fig. 8(b). Fit of the linearization to BS dye adsorption isotherms on TiO₂ nano-particles Freundlich.

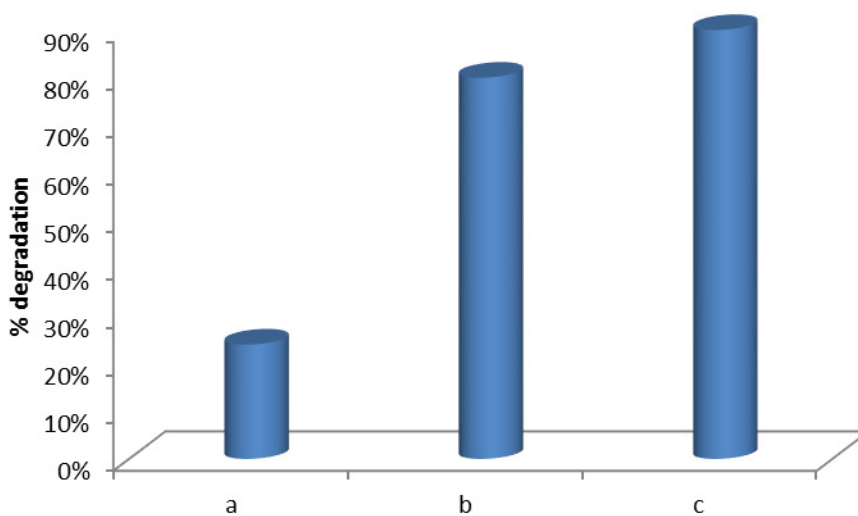


Fig. 9. The effect of electron acceptors in photocatalytic activity of TiO₂. (a) in present only TiO₂=0.12 g/litL, (b) in present TiO₂=0.12 g/L and S₂O₇⁻² = 1 × 10⁻² M. (c) in present TiO₂=0.12 g/L and S₂O₈⁻² =1 × 10⁻² M. All solutions were irradiated with UV light for 5 min at room temperature.

1/n above one specifies cooperative adsorption. It is pertinent to point out that Kf and n are parameters characteristic of the sorbent-sorbate system, which must be determined by data fitting and whereas linear regression is generally used to determine the parameters of kinetic and isotherm models.

Plot of (C_{eq}/Q_{eq}) versus C_{eq} Fig. 8a gives a straight line with a correlation coefficient $R^2=0.9945$. The Q_{max} (178.57 mg/g) and K_L (1.037 L/mg) values were calculated from the slope and intercept, respectively. The value of the calculated adsorption equilibrium constant, K_L of TiO_2 is considerable that specifies significant adsorption capacity of the TiO_2 nanoparticles. The plot of $(\log Q_{eq})$ versus $(\log C_{eq})$ Fig. 8b gives a straight line with a correlation coefficient $R^2=0.3006$. As a result it can be concluded that the behavior of (BS) dye on TiO_2 nanoparticles is good agreement with the Langmuir model ($R^2=0.9945$).

Effect of Electron Acceptors in Dye Photo-Degradation

Using inorganic oxidants, such as $S_2O_7^{2-}$ and $S_2O_8^{2-}$ in TiO_2 system increase the quantum efficiencies either by inhibiting electron-hole pair recombination through scavenging conduction band electron at the surface of TiO_2 or by offering additional oxygen atom as an electron acceptor to form the superoxide radical ion (Merabet, et al., 2009). The effect of electron acceptors in photo-catalytic activity of TiO_2 was performed by exposing the dye solutions with UV-Vis light for three different samples, (i) in present only TiO_2 (ii) in present TiO_2 and $S_2O_7^{2-}$ and (iii) in present of TiO_2 and $S_2O_8^{2-}$. All experiments were conducted under the same circumstances (e.g., 5 min irradiation at room temperature) with concentrations of 0.12 g/L TiO_2 , 1×10^{-2} M of $S_2O_7^{2-}$ and $S_2O_8^{2-}$ M. Samples were analyzed with the same spectral condition and results are presented in Fig. 9. Results specify that the degradation of dye is significantly improved in the present of the oxidant reagent. Comparison between $S_2O_7^{2-}$ and $S_2O_8^{2-}$ also reveals that the latter compound affects more efficient than that the former r.

CONCLUSION

The photo-degradation of the (BS) dye in the present of TiO_2 was carried out by radiation of UV light at room temperature. Experimental conditions were studied to find the best situation of degradation of Biberichscarlet (BS). Results were specified % degradation depend on different experimental conditions, such as hydrogen peroxide concentration and pH values. Results revealed increasing H_2O_2 concentration leads to a enhance % degradation.

Other various parameters such as TiO_2 loading and present of some electron acceptors and common salts in wastewater, were also examined. Results indicated that the degradation of dye is also highly depended to the oxidant reagent such as $S_2O_7^{2-}$ or $S_2O_8^{2-}$. The equilibrium data have been analyzed using Langmuir and Freundlich isotherms models and the characteristic parameters for each isotherm have been determined. According to the correlation coefficients R^2 , the Langmuir isotherm has been fitted better for the adsorption of (BS) on TiO_2 .

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