

## Factors influencing the photocatalytic degradation of Rhodamine B by TiO<sub>2</sub>-coated non-woven paper

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### Abstract

The photocatalytic degradation of Rhodamine B (RhB) has been investigated in aqueous solutions using TiO<sub>2</sub>-coated non-woven paper as photocatalyst. The experiments were carried out to investigate the factors that influence the RhB photocatalytic degradation, such as adsorption, initial concentration of dye solution, temperature, and some inorganic species commonly present in real wastewaters such as Cl<sup>-</sup>, NO<sub>3</sub><sup>-</sup>, SO<sub>4</sub><sup>2-</sup>, CH<sub>3</sub>COO<sup>-</sup> and HPO<sub>4</sub><sup>2-</sup>. The experimental results show that adsorption is an important parameter in controlling the apparent kinetic order of the degradation. The photocatalytic reaction is favoured by a high concentration in respect to Langmuir–Hinshelwood model. The photodegradation was temperature-dependent with a high degradation rate at high temperature. The presence of the Cl<sup>-</sup>, CH<sub>3</sub>COO<sup>-</sup> and HPO<sub>4</sub><sup>2-</sup> ions leads to the reduction of the effectiveness of the photodegradation. However, the presence of SO<sub>4</sub><sup>2-</sup> increases the rate of the degradation.

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**Keywords:** Photocatalytic degradation; Rhodamine B; TiO<sub>2</sub>; Non-woven paper; Inorganic salts

### 1. Introduction

Heterogeneous photocatalysis is an efficient technique to destroy organic pollutants in water [1–8]. This technique is based upon the use of UV-irradiated semiconductors (generally titania). When TiO<sub>2</sub> is irradiated with photons whose energy is equal to or greater than its band gap energy ( $E_G = 3.2$  eV) i.e., with  $\lambda = 390$  nm, electron-hole pairs are created. In aqueous system, holes react with H<sub>2</sub>O or OH<sup>-</sup> adsorbed at the surface of the semiconductor to produce OH<sup>•</sup> radicals which are the most oxidizing species in this process. On the other hand, electrons are trapped at surface sites and removed by reactions with adsorbed molecular O<sub>2</sub> to form superoxide anion radical O<sub>2</sub><sup>•-</sup> (or HO<sub>2</sub><sup>•</sup> at lower pH) [9].

For the degradation process two methods are favoured, suspended photocatalyst in aqueous media, and immobilized on support materials. In view of practical engineering, the immobilized photocatalyst should be preferred, to avoid downstream treatment (particle–fluid separation and/or photocatalyst recy-

cling) [10]. This has led to a major attempt to immobilize the photocatalyst on support including ceramic [11], glass fibre [12], glass, quartz and stainless steel [13], activated carbon [14], non-woven paper [15] and others. However, these efforts have not produced materials which meet all demands of photocatalytic activity.

The Rhodamine B is one of the most common xanthenes dyes for textile industry, it is famous for its good stability as dye laser materials, and it is also used as biological stain. The Rhodamine B is highly soluble in water and organic solvent, and its colour is fluorescent bluish-red. This compound is now banned from use in foods and cosmetics because it has been found to be potentially toxic and carcinogenic. So the photodegradation of RhB is important with regard to the purification of dye effluents.

Recent articles have shown that Rhodamine B can be destroyed in aqueous suspension using TiO<sub>2</sub> [16], TiO<sub>2</sub>-coated silica [17] and TiO<sub>2</sub>-coated silicone sealant [18]. In this work, the influence, on the photocatalytic degradation of Rhodamine B by immobilized TiO<sub>2</sub> on non-woven paper, of various parameters such as the initial RhB concentration, the previous adsorption in the dark, the temperature and some organic anions commonly present in real wastewaters was studied.

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## 2. Experimental

### 2.1. Materials

The immobilized photocatalyst used in this study was a commercial titania photocatalyst from Ahlstrom firm (France), it consists of PC500 Titania by Millennium inorganic chemicals (anatase: >99%, specific surface area 350–400 m<sup>2</sup> g<sup>-1</sup>, crystallites mean size = 5–10 nm). Titania PC500 was coated on non-woven paper (natural and synthetic fibres 254 μm of thickness) using an inorganic binder. The binder was an aqueous dispersion of colloidal SiO<sub>2</sub> (EP1069950B1 European patent). A specific surface area extender (zeolite UOP, 2000 m<sup>2</sup> g<sup>-1</sup>) was used to increase adsorption properties of the photocatalyst. The physical and chemical properties of the photocatalyst are shown in Table 1.

The Rhodamine B (purity, 99%) was purchased from Exciton (USA) and used as received. It consists on green crystals or reddish-violet powder, its molecular formula is C<sub>24</sub>H<sub>31</sub>ClN<sub>2</sub>O<sub>3</sub> (molecular weight: 479.02). The Rhodamine B structure is given in Fig. 1. All the other reagents used in this study were analytical grade. NaOAc (Rhône-Poulenc), Na<sub>2</sub>HPO<sub>4</sub> (BDH Chemicals), Na<sub>2</sub>SO<sub>4</sub> (Labosi), NaCl and NaNO<sub>3</sub> (Merck) salts were used as purchased.

### 2.2. Photocatalytic reactor

Experiments were carried out using a cylindrical batch reactor opened at air, 8 cm in diameter and 12 cm in working height, the water jacket has a diameter of 5 cm contain the UV-lamp and permits the water circulation (Fig. 2). The photoreactor was recovered inside with (11 cm × 25 cm) of the photocatalyst and was exposed to a luminous source composed of a HPK Philips UV-lamp (125 W), placed in axial position inside the water jacket. The reactor was initially loaded with 500 mL of RhB aqueous solution and maintained in low continuous stirring (100 rpm) by means of a magnetic stirrer.

### 2.3. Procedure and analysis

The adsorption experiments were carried out by immersing 11 cm<sup>2</sup> of the photocatalyst in 20 mL of RhB solutions during

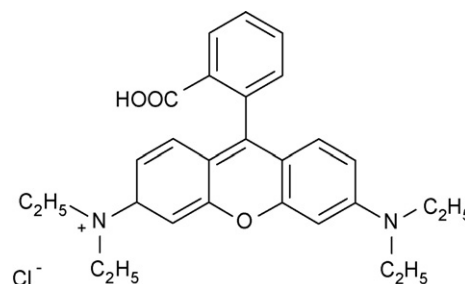


Fig. 1. Molecular structure of Rhodamine B.

1 h, the adsorption isotherms were obtained by different initial concentration (4–20 mg L<sup>-1</sup>) at 25 °C, and the effect of temperature was obtained by varying temperature from 25 to 40 °C with an initial concentration of 12 mg L<sup>-1</sup>. The quantity adsorbed was calculated by measuring the concentration of the solution before and after adsorption using the following equation:

$$q_e = \frac{(C_0 - C_e)}{S} \quad (1)$$

where  $q_e$  (mg m<sup>-2</sup>) is the amounts of RhB adsorbed per unit surface of the photocatalyst at adsorption equilibrium,  $C_0$  (mg L<sup>-1</sup>) is the initial RhB concentration,  $C_e$  (mg L<sup>-1</sup>) is the RhB concentration at equilibrium and  $S$  (m<sup>2</sup> L<sup>-1</sup>) is the ratio between the surface of the photocatalyst and the volume of the aqueous solution.

The photocatalytic degradation experiments were carried out by loading 500 mL of RhB solutions in the photocatalytic reactor. The effect of initial concentration was obtained with different initial concentrations (4–20 mg L<sup>-1</sup>) at 25 °C. The effect of temperature was obtained by studying the photocatalytic degradation at different solution temperatures (25–40 °C) with an initial solution concentration of 12 mg L<sup>-1</sup>. For the effect of the inorganic anions, the mass of each salts equivalent to 200 mg L<sup>-1</sup> of each anion was added to the initial solution of the dye. All solutions were irradiated after 1 h of previous adsorption.

The RhB aqueous solutions were filtered by Millipore membrane filter type 0.45 μm HA, and the concentrations were determined from UV–vis absorbance characteristic with the calibration curve method. A Jenway 6405 UV–visible spectrophotometer was used. The maximum adsorption wavelength ( $\lambda_{max}$ ) was 554 nm. The concentrations were calculated taking

Table 1  
Chemical and physical properties of the Ahlstrom photocatalyst (Ref: 1048)

Composition	
PC500	18 g m <sup>-2</sup>
SiO <sub>2</sub>	20 g m <sup>-2</sup>
UOP200	2 g m <sup>-2</sup>
Physical properties	
Mass per unit area	75 g m <sup>-2</sup>
Thickness	254 μm
Air permeability	2570 L m <sup>-2</sup> s <sup>-1</sup>
Tensile strength MD	1100 N m <sup>-1</sup>
CD	500 N m <sup>-1</sup>
Elongation MD	3%
CD	5%
Water drop	2 s

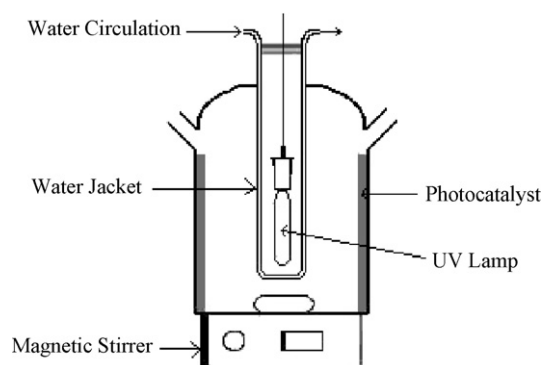


Fig. 2. Schematic diagram of the photocatalytic reactor.

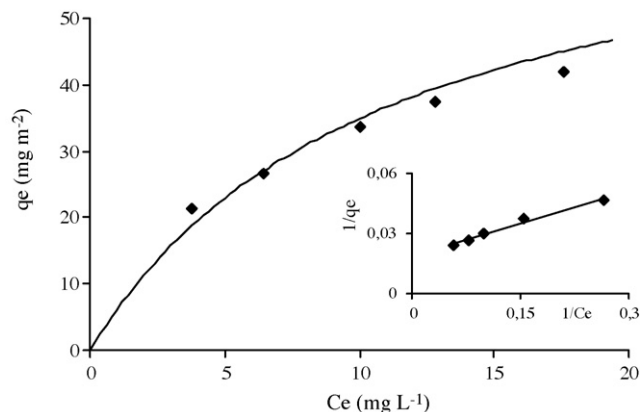


Fig. 3. Langmuir adsorption isotherm of RhB: quantity adsorbed as a function of equilibrium concentration. In the insert: transformation of Langmuir isotherm: reciprocal of the quantity adsorbed as a function of reciprocal of equilibrium concentrations.

into account the effect of inorganic salts on the calibration curve of RhB.

### 3. Results and discussion

#### 3.1. Adsorption study in the dark

It is likely that sorption of the dye is an important parameter in determining photocatalytic degradation rate. The adsorbed dye on the surface of the semiconductor particles acts as an electron donor, injecting electrons from its excited state to the conduction band of the semiconductor under UV irradiation. Adsorption tests in the dark were carried out in order to evaluate the equilibrium constants of the adsorption of the dye on the photocatalyst surface. Fig. 3 shows an isotherm of L-shape according to the classification of Giles et al. [19]. The L-shape of the isotherm means that there is no strong competition between the solvent and the adsorbate to occupy the adsorbent surface sites.

The experimental data were fitted to the Langmuir equation (Eq. (2)) to describe the adsorption of RhB on the surface of the photocatalyst:

$$\frac{q_e}{q_m} = \frac{K C_e}{1 + K C_e} \quad (2)$$

where  $q_m$  ( $\text{mg m}^{-2}$ ) is the maximal amount of the dye adsorbed per unit surface of the photocatalyst,  $q_e$  ( $\text{mg m}^{-2}$ ) is the amount of the dye adsorbed per unit surface of the photocatalyst at the equilibrium,  $K$  ( $\text{L mg}^{-1}$ ) is the Langmuir equilibrium constant and  $C_e$  ( $\text{mg L}^{-1}$ ) is the concentration of the dye in aqueous solution at the equilibrium.

The linear transformation of Eq. (2) can be expressed by the following equation:

$$\frac{1}{q_e} = \frac{1}{q_m} + \frac{1}{K q_m C_e} \quad (3)$$

The linear transforms are given in the insert in Fig. 3, the ordinate at the origin is equal to the reciprocal of  $q_m$ , whereas  $K$  can be calculated from the slope ( $1/q_m K$ ). From the data obtained,

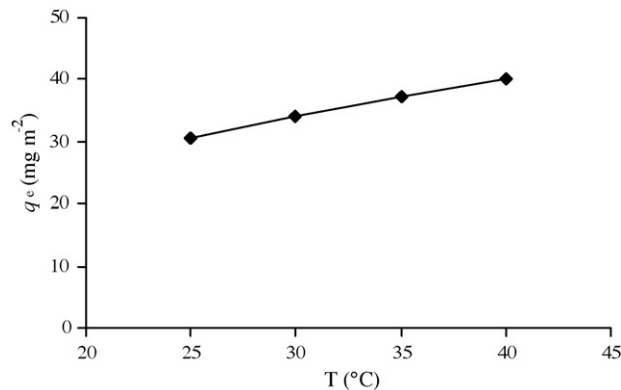


Fig. 4. Variation of RhB adsorbed on the photocatalyst as a function of solution temperature.

the maximal adsorption quantity and the Langmuir adsorption constant are, respectively,  $73 \text{ mg m}^{-2}$  and  $0.092 \text{ L mg}^{-1}$ .

Since real effluents to be treated can be at different temperatures (according to the seasons, the geographical sites, during the treatment itself, etc.), comparative experiments were performed by varying this parameter. Fig. 4 shows the variation of the RhB adsorbed on the photocatalyst as a function of temperature. An increase of the amount of RhB adsorbed is observed with rising the temperature. From these results, the enthalpy of adsorption ( $\Delta H$ ) has been calculated using the linear transformation of the following equation [20].

$$q_e = (q_e)_0 \exp \left[ - \left( \frac{\Delta H}{RT} \right) \right] \quad (4)$$

The linear transformation of this equation expressed by  $\ln(q_e)$  as function of  $1/T$  gives a straight line whose slope is equal to  $-\Delta H/R$ . From the data obtained, the enthalpy of adsorption calculated was  $13.78 \text{ kJ mol}^{-1}$ ; the positive sign of this energy indicates that the adsorption reaction was endothermic.

#### 3.2. Kinetic analysis as a function of RhB initial concentration

The kinetics of disappearance of RhB dye is represented in Fig. 5. It is evident that the photodegradation rate depends on the initial concentration of the dye. Since the lifetime of hydroxyl radicals is very short (only a few nanoseconds), they can only react at or near the location where they are formed. A high dye concentration logically enhances the probability of collision between organic matter and oxidizing species, leading to an increase in the decolouration rate.

According to numerous works [20–23], the influence of the initial concentration of the solute on the photocatalytic degradation rate of most organic compounds is described by pseudo-first order kinetics.

$$-\frac{dC}{dt} = k_{ap} C \quad (5)$$

where  $k_{ap}$  ( $\text{h}^{-1}$ ) is the apparent rate constant and is affected by dyes stuff concentration. Integration of that Eq. (5) will lead to

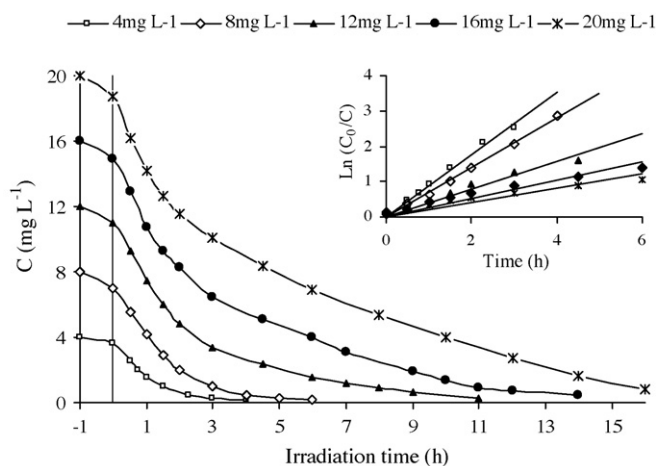


Fig. 5. Variation of RhB concentration versus time for different RhB initial concentration. In the insert: the plot of  $\ln(C_0/C)$  versus  $t$  with different initial concentration of RhB.

the expected relation:

$$\ln\left(\frac{C_0}{C}\right) = k_{ap}t \quad (6)$$

The plot of  $\ln(C_0/C)$  versus  $t$  with different initial concentration of RhB is shown in the insert in Fig. 5. The figure shows that the photocatalytic degradation follows the pseudo-first order kinetic in the case of initial concentrations of 4 and 8 mg L<sup>-1</sup>. However, for concentrations greater than 8 mg L<sup>-1</sup>, the photocatalytic degradation takes place in two stages, the first stage of the process follows pseudo-first order kinetic with respect to RhB concentrations and a second slower stage until the end of the process of the photocatalytic degradation. The length time of the first stage decreases with increasing the initial RhB concentration. This result can be explained by the competitive adsorption/degradation behaviour between RhB molecules and its degradation intermediates, which could be more significant with high concentration in solution.

The effect of initial RhB concentration on the initial rate of degradation is shown in Fig. 6. The figure indicates that rate of decolouration increases with increasing initial concentration of RhB which correspond to Langmuir–Hinshelwood adsorption model according to the following equation:

$$r_0 = k_{ap}C_0 = \frac{k_c K C_0}{1 + K C_0} \quad (7)$$

where  $r_0$  (mg L<sup>-1</sup> h<sup>-1</sup>) is the initial rate of the photocatalytic degradation,  $k_{ap}$  (h<sup>-1</sup>) is the apparent rate constant and  $k_c$  is a constant depending on the other factors influencing the process (UV intensity, temperature, ...).

A linear expression can be conventionally obtained by plotting the reciprocal initial rate constant against initial concentration:

$$\frac{1}{k_{ap}} = \frac{1}{k_c} C_0 + \frac{1}{k_c K} \quad (8)$$

The plot of  $1/k_{ap}$  against  $C_0$  as shown in the insert in Fig. 6 gives linear relationship between  $1/k_{ap}$  and  $C_0$ . From the values

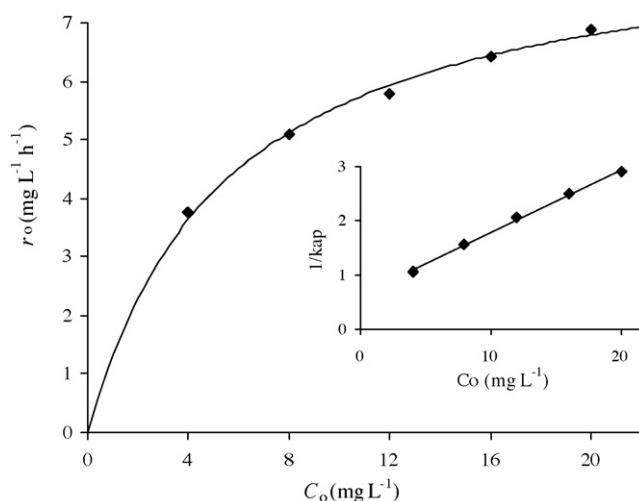


Fig. 6. Effect of initial RhB concentration on the initial rate of degradation. In insert: plot of reciprocal of pseudo-first order rate constant against initial concentration.

of the slope ( $1/k_c$ ) and the intercept ( $1/k_c K$ ),  $k_c$  and  $K$  values for the photocatalytic degradation of RhB were found to be, respectively, 8.68 mg L<sup>-1</sup> h<sup>-1</sup> and 0.18 L mg<sup>-1</sup>. The adsorption equilibrium constant  $K$  obtained from this linearization is different to the constant obtained from the adsorption isotherm. This difference can be due to the contribution of the specific surface area extender (zeolite) and the binder (SiO<sub>2</sub>) on the initial adsorption of the dye, so the adsorption isotherm represents both the adsorption on photoactive TiO<sub>2</sub> and on the support of the photocatalyst. However, the constant obtained from the kinetic of the photocatalytic degradation represents only the adsorption on the photoactive TiO<sub>2</sub>.

### 3.3. Effect of temperature

The kinetics of the photocatalytic degradation of Rhodamine B at different working temperature in the range of 25–40 °C has been carried out. The results are shown in Fig. 7. The figure indicates that, the kinetic of the photocatalytic degradation is

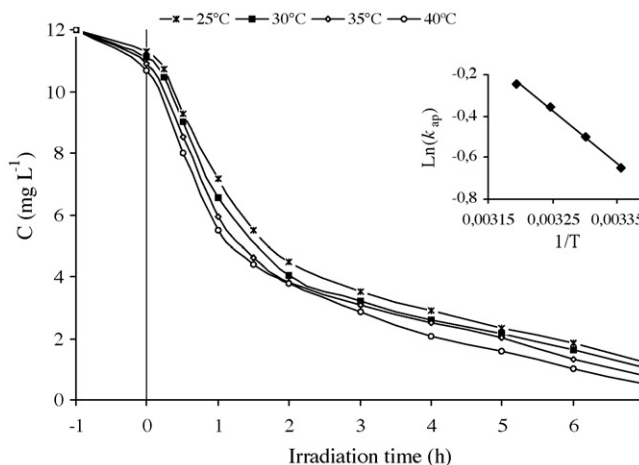


Fig. 7. Kinetic of photocatalytic degradation of RhB at different solution temperature. In insert: plot of  $\ln(k_{ap})$  versus  $1/T$ .

temperature-dependent, the rate of degradation increases with the increase of temperature. Increase in temperature increases the amount of Rhodamine B adsorbed on the surface of the photocatalyst previously shown and also helps the reaction to compete more efficiency with  $e^- - h^+$  recombination [24].

The apparent activation energy ( $E_a$ ) has been calculated from the Arrhenius equation

$$k_{ap} = k_{ap0} \exp \left[ - \left( \frac{E_a}{RT} \right) \right] \quad (9)$$

The linear transform  $\ln(k_{ap}) = f(1/T)$ , which is shown in the insert in Fig. 7, gives a straight line whose slope is equal to  $-E_a/R$ . From the data obtained, the apparent activation energy is 21.17 kJ mol $^{-1}$ . This apparent energy represents the total activation energy of both adsorption and photocatalytic degradation of RhB.

By considering the Langmuir–Hinshelwood model to describe the photocatalytic degradation process, the apparent rate constant can be expressed by:

$$k_{ap} = \frac{k_c K}{1 + K C} \quad (10)$$

At low concentration  $K C$  can be neglected with respect to 1 and one gets the simplified expression:

$$k_{ap} = k_c K \quad (11)$$

This equation can also be expressed as a function of temperature by applying the Arrhenius's law to  $k_c$  and the Van't Hoff one to  $K$ :

$$k_{ap} = k_{ap0} \exp \left[ - \left( \frac{E_a}{RT} \right) \right] = k_{c0} K_0 \exp \left[ - \left( \frac{E_t + \Delta H}{RT} \right) \right] \quad (12)$$

hence

$$E_a = E_t + \Delta H \quad (13)$$

where  $E_t$  represents the true activation energy of the photocatalytic degradation, it depends on the other parameter affecting the photodegradation process (light flux and oxygen concentration [24,25], ...), from this results the true activation energy is 7.39 kJ mol $^{-1}$ , this value is close to the true activation of Acid Blue 25 dye on Degussa P-25 obtained by Bouzaida et al. [20].

### 3.4. Effect of some anions

In the real industrial wastewaters, dyes are present with many organic and inorganic species which can affect the photocatalytic degradation. In this study, the effect of  $Cl^-$ ,  $NO_3^-$ ,  $SO_4^{2-}$ ,  $CH_3COO^-$  and  $HPO_4^{2-}$  was investigated. The effect of these anions on the apparent rate constant of the photocatalytic degradation is shown in Table 2. The table indicates that the presence of the  $Cl^-$ ,  $CH_3COO^-$  and  $HPO_4^{2-}$  ions leads to reduction of the effectiveness of the photodegradation, the presence of  $SO_4^{2-}$  increases the rate of the degradation; however, the presence of  $NO_3^-$  does not affect the degradation rate.

Previous studies have shown that chloride anions, which are used to promote exhaustion of the dye onto cotton in the textile industry, inhibit the photocatalytic degradation of organic

Table 2  
Effect of some anions on the pseudo-first order rate constant

	Anion					
	No anion	$NO_3^-$	$CH_3COO^-$	$Cl^-$	$SO_4^{2-}$	$HPO_4^{2-}$
$k_{ap}$ (h $^{-1}$ )	0.475	0.474	0.410	0.431	0.549	0.406

molecules [26–28]. This inhibition can be explained by both the competition of adsorption with RhB onto  $TiO_2$  surface and the scavenging of photo-produced  $OH^\bullet$  by  $Cl^-$  (Eq. (14)) [29].



The  $Cl^\bullet$  radicals are in principle also capable of oxidizing pollutants, but at lower rates than the  $OH^\bullet$  radicals, due to their lower oxidation power [30]. The generation of  $Cl^\bullet$  radicals leads also to the formation of chlorinated organic compounds [31], which are known as very harmful and persistent substances and therefore longer treatment periods are needed.

For  $HPO_4^{2-}$  ions, it's well known that they are strongly adsorbed on the surface of  $TiO_2$ , and therefore, they compete with the adsorption of RhB on the surface of the photocatalyst. They can also scavenge  $OH^\bullet$  to form the corresponding anion radicals  $HPO_4^{\bullet-}$ , which leads to the decrease of the oxidation process [32].

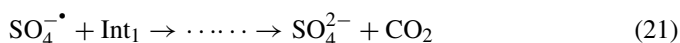
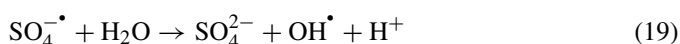
The reduction of the photocatalytic degradation in presence of  $CH_3COO^-$ , which is used to reduce the alkalinity of the effluent in dyeing section, can be explained by the “photo-kolbe” reaction (Eq. (15)) [33], which inhibits the formation  $OH^\bullet$  radicals via Eqs. (16) and (17) on the surface of the photocatalyst.



For  $SO_4^{2-}$  ions, they are adsorbed in the surface of  $TiO_2$  and consequently react with photo-induced holes ( $h^+$ ) [34]:



As S is the strong oxidizing agent, the sulfate radical can accelerate the reaction and participate in the degradation process according to the following reactions [35].



where  $Int_1$  symbolises the first reaction intermediates partially transformed until the total mineralization. The reaction Eq. (19) enhances the photocatalytic reaction rate by contribution to the  $OH^\bullet$  formation. Similar result was found by Wang et al. [36] for the photocatalytic degradation of *o*-methylbenzoic acid.

Concerning the  $NO_3^-$  ions, it has been shown that they are weakly adsorbed on the surface of  $TiO_2$  [37], and have on this fact, a little impact on the photocatalytic degradation. This result



is in agreement with those observed by Guillard et al. [28] and Lucas Vaz et al. [29].

#### 4. Conclusion

The photocatalytic degradation of Rhodamine B has been performed with immobilized TiO<sub>2</sub> photocatalyst on non-woven paper. The photocatalytic degradation can be explained in terms of Langmuir–Hinshelwood kinetic model. The photocatalytic degradation is directly related to the adsorbed quantities of the pollutant. The experimental results show that the rate of degradation is sensitive to the operational parameters. The photocatalytic degradation was temperature-dependent with apparent activation energy of RhB of 21.17 kJ mol<sup>-1</sup>. The presence of the Cl<sup>-</sup>, CH<sub>3</sub>COO<sup>-</sup> and HPO<sub>4</sub><sup>2-</sup> ions leads to reduction of the effectiveness of the photodegradation, the presence of SO<sub>4</sub><sup>2-</sup> increases the rate of the degradation; however, the presence of NO<sub>3</sub><sup>-</sup> does not affect the degradation rate.

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