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# Solar photocatalytic decolourization of Crystal violet using supported $TiO_2$: effect of some parameters and comparative efficiency

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## ABSTRACT

The present study focuses on the decolourization of crystal violet (CV) using $TiO_2$ supported on glass slides under natural sunlight (sunny days, June 2013). The commercial Degussa P25 $TiO_2$ was used as a photocatalyst in this study. The P25 $TiO_2$ deposition process was achieved using acetone as precursor. The effect of some process parameters (CV concentration, number of $TiO_2$ glass slides, pH and presence of metallic cations as interfering ions such as $Cu^{2+}$, $Fe^{2+}$, $Fe^{3+}$, $Mn^{2+}$ and $Zn^{2+}$) was investigated. It was found that the decolourization process follows a pseudo-first order with respect to the substrate concentration. The pH had a net effect on the decolourization process. The apparent rate constants increased proportionally to the number of $TiO_2$ glass slides. The results show that the presence of $Fe^{2+}$, $Mn^{2+}$ and $Cu^{2+}$ cations with CV decreases the decolourization rate by varying degrees (22.1–35.3%) while $Zn^{2+}$ has a negligible effect. However, the presence of $Fe^{3+}$ led to an acceleration of the CV decolourization. The P25 $TiO_2$-supported can be reused several times under the present working conditions. Finally, the photoactivity efficiency of P25 $TiO_2$-supported was compared to that of P25 $TiO_2$-dispersed under artificial UV (single lamp, 300–400 nm) and natural sunlight. Under the present experimental conditions, a complete decolourization was achieved less than 6 h of solar irradiation at natural pH and using two $TiO_2$ glass slides.

*Keywords:* $TiO_2$-supported; Photocatalysis; Decolourization; Crystal violet; Natural sunlight

## 1. Introduction

The scarceness of water resources poses a major challenge particularly with the increase of the world population. In this respect, the treatment of wastewaters has become a global strategy in order to decrease the dependency on natural water resources. Several technologies are available for wastewater treatment [1–3]. Of these technologies, the advanced oxidation processes (AOPs) are extensively investigated [4–6]. The AOPs can be used for the elimination of organic pollutants [7], inorganic pollutants [8] or both [9]. The AOPs include several techniques among which, the one relevant to this study, is titanium dioxide/UV light [10]. These techniques generally require the use of artificial UV sources which account for the most important part of costs. In view of the ever increasing demand of energy from both the developed and emerging economies, alternative energy sources are

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constantly sought [11,12]. The use of solar energy for the treatment of wastewaters has become an attractive option as it is a free and clean energy therefore economical [13–16].

The heterogeneous photocatalysis process used to degrade recalcitrant organic pollutants makes use of the powerful oxidizing $^{\cdot}$OH radical species generated in aqueous solutions containing a semiconductor under UV irradiation (Eqs. (1)–(3)) [17,18].

$$\mathrm{TiO_2} + hv \rightarrow \mathrm{TiO_2} + e^-_{cb} + h^+_{vb} \tag{1}$$

$$\mathrm{O_2} + e^-_{cb} \rightarrow \mathrm{O_2^{\cdot -}} \tag{2}$$

$$\mathrm{H_2O} + h^+_{vb} \rightarrow {}^{\cdot}\mathrm{OH} + \mathrm{H^+} \tag{3}$$

Among the different types of semiconductors used, $TiO_2$ is of special interest thanks to its efficient photoactivity and stability [19]. On the other hand, one of the drawbacks of the use of $TiO_2$ particles in suspension is the post-$TiO_2$ separation step and the fouling in circulating reactors. To overcome this limitation, a wide range of investigations for the photodegradation of organic pollutants using immobilized $TiO_2$ have abundantly been reported in the literature [20].

In the present work, the decolourization of crystal violet (CV) as a model molecule (Fig. 1) was investigated using Degussa P25 $TiO_2$-supported on glass slides under natural sunlight (Annaba, Algeria, June 2013). CV is often used to dye paper and as a component of navy blue and black inks for printing. It is also used in the textile industry for dyeing nylon, wool, cotton and silk, [21]. The effect of substrate concentration, number of $TiO_2$ slides, pH and presence of metallic cations was investigated. Furthermore, the reuse of the P25 $TiO_2$-supported under natural sunlight was tested. Finally, the photoactivity efficiency of P25 $TiO_2$-supported was compared to that of P25 $TiO_2$-dispersed under UV (300–400 nm) and natural sunlight.

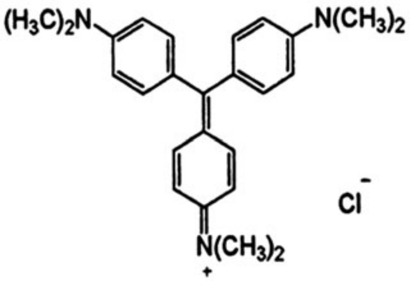

Fig. 1. Chemical structure of CV.

## 2. Materials and methods

### 2.1. Materials

P25 Titanium dioxide (Degussa AG Company) was used in this study. Its polycrystalline structure is composed of approximately 80% anatase and 20% rutile. It has a BET surface area of $50\,m^2/g$ and is approximately 21 nm as primary particles [22]. Acetone (Aldrich, 99.99%) was used for fixing P25 $TiO_2$ on glass slides. CV (MW = 408.979 g/mol, Fluka) of analytical grade was used without further treatment. Sample solutions of CV were prepared using double distilled water and protected from light. Adjustment of the pH solutions was achieved with $H_2SO_4$ or NaOH and monitored by a pH-meter (HANNA HI 9812-5). The intensity of radiations (UV lamp and sunlight) was measured at 365 nm using a VLX-3 W radiometer (Vilber Loumart, France). Sulfates salts of metallic cations ($Cu^{2+}$, $Fe^{2+}$, $Fe^{3+}$, $Mn^{2+}$ and $Zn^{2+}$) were used for the study of the effect of the interfering substances.

### 2.2. Preparation of P25 $TiO_2$-supported

The P25 $TiO_2$-supported used in this study was prepared by depositing P25 $TiO_2$ on glass slides (2.6 × 7.6 cm) according to a slightly modified method used by Bianchi et al. [23] and Tusnelda et al. [24]. Prior to their use, the glass slides were cleaned with double distilled water and acetone. A mixture of P25 $TiO_2$ and acetone (approximately 0.6 g $TiO_2$/30 mL acetone) was homogenized by mixing for 30 min then poured onto horizontally disposed glass slides and left until complete evaporation of the solvent. The slides were then dried overnight at 110°C to ensure strong fixation of $TiO_2$. This procedure enabled the formation of a thin layer of $TiO_2$, the mass of which on each glass slide was approximately 0.050 g.

## 3. Experimental procedure

The photocatalytic decolourization experiments of CV were performed using a static batch reactor (Fig. 2) consisting of 250 mL-Pyrex beakers open to air under natural sunlight (sunny days, June 2013) between 9:00 am and 3.00 pm at sea level (in front of the chemistry department without any obstacle. The solution of the substrate (250 mL) in the presence of P25 $TiO_2$ (dispersed or supported) was exposed to natural sunlight with constant stirring. The $TiO_2$ glass slides were kept in a slight inclination inside the beaker. The magnetic agitation had no effect on their positioning. As the slide width is relatively small (2.6 cm), the

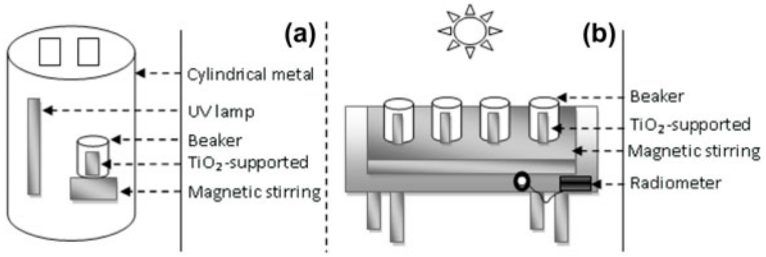

Fig. 2. Experimental set-up used for the decolourization of CV under UV (a) and sunlight (b) irradiations.

dimension of the beaker allows the allocation of a maximum four slides. UV-irradiation experiments were performed under the same conditions using a single TLD UV lamp emitting in the range 300–400 nm, with a maximum emission located at 365 nm and placed in a vertical position at 10 cm from the beaker. During the experiments, samples (4 mL) were collected at selected time intervals. In the case of P25 $TiO_2$-dispersed, the samples were filtered through 0.45 μm-pore size membrane filters (Millipore Corp.). The decolourization of CV (maximum absorption at $\lambda_{max}$= 590 nm) was monitored using a UV–vis spectrophotometer (JENWAY 6405). During the period of experimentation, the fluctuations of solar intensities were in the range 1.55–1.65 $mW/cm^2$. The extent of water evaporation during the solar photocatalysis experiment was in average of 6.0% in volume after 6 h of irradiation which is considered to be within the experimental errors. Due to the inherent non reproducibility of solar radiations and in order to minimize the experimental errors, the study of the effects of each parameter was performed simultaneously on a set of experiments along with a blank sample (without $TiO_2$) to evaluate the contribution of solar photolysis. In a separate trial, experiments were carried out in triplicate under the same experimental conditions in order to evaluate the error. All the results were reproducible within ±10% variation. The calibration curve was repeated between four and five times during the measurements which gave a determination ($R^2$) coefficient ≥ 0.9998.

## 4. Results and discussion

### 4.1. Effect of CV loading

The results of CV decolourization as function of the concentration are shown in Fig. 3. The results, presented as the normalized concentration ($C/C_0$) of CV, demonstrate that the increase of CV concentration leads to a decrease of the decolourization process. This may be attributed to: (i) saturation of $TiO_2$ surface at high concentration of CV, which hinders the penetration of sunlight onto the $TiO_2$ surface (screen effect); (ii) insufficient amount of $^{\cdot}OH$ radicals, as the production of these oxidizing species is constant for a given amount of the catalyst. The kinetics of the CV decolourization with respect to the concentration of the substrate is presented in Fig. 2 and is satisfactorily described by a pseudo-first-order reaction (Eq. (4)) according to Langmuir-Hinshelwood kinetic model.

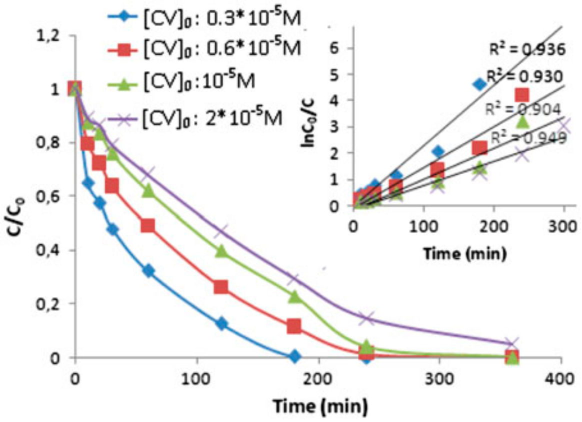

Fig. 3. Effect of initial CV concentration on the photocatalytic decolourization process using P25 $TiO_2$-supported under sunlight irradiation. Conditions: natural pH (6.3); number of $TiO_2$ glass slides: 2.

$$\ln\frac{C_t}{C_0} = -kt \tag{4}$$

where $C_0$ and $C_t$ represent the CV concentration before and after reaction (mol/L); $t$ is the irradiation time (min); and $k$ is the apparent rate constant ($min^{-1}$).

The $10^{-5}$ M value was chosen for the study of the subsequent parameters as it is the maximum concentration leading to a completed decolourization after 6 h.

### 4.2. Effect of number of P25 $TiO_2$-supported slides

Fig. 4 shows the dependence of the number of the P25 $TiO_2$-supported slides on the CV photocatalytic decolourization. From this figure, it can be noticed that, as anticipated, the process of CV decolourization increases with the number of slides impregnated in the solution. This enhancement is most probably due to the production of more oxidizing species when the number of slides increases leading to a faster decolourization process. The pseudo-first-order decolourization also applies under these conditions. The apparent rate constants increase proportionally to the number of slides used. The $k$ values were $0.575.10^{-2}\,min^{-1}$ ($R^2 = 0.990$), $0.903.10^{-2}\,min^{-1}$($R^2 = 0.948$), $1.591.10^{-2}\,min^{-1}$($R^2 = 0.998$) and $2.048.10^{-2}\,min^{-1}$($R^2 = 0.993$) for one, two, three and four slides, respectively. For the study of the effect of the subsequent parameters on the CV decolourization, two slides were used as they were enough to yield a total decolourization after 6 h.

### 4.3. Effect of pH

As pH is an important parameter for the decolourization reactions taking place on the photocatalyst surface [18], the photocatalytic decolourization of CV under sunlight was studied at different pH values in the range 2.5–8.7. The decolourization rates of CV as function of pH are presented in Fig. 5 along with their respective apparent rate constants ($k$) based on a pseudo-first-order reaction kinetics. From this figure, it is interesting to note that the apparent rate constants increase with the increase of the solution pH reflecting an increasing efficiency. The decolourization rates were at their maxima at pH 4.7 (after 6 h, $0.0139\,min^{-1}$), natural pH 6.3 (after 6 h, $0.015\,min^{-1}$), pH 7 (after 4 h, $0.0172\,min^{-1}$) and pH 8.7 (after 2 h, $0.0332\,min^{-1}$). However, at pH 2.5 the removal degree was 97.4% with a small apparent rate constant (after 6 h, $0.01\,min^{-1}$). This may be attributed to the surface ionization of photocatalyst particles [25]. At pH greater than $pH_{ZPC}$, the photocatalyst surface has a net negative charge due to the significant fraction of total surface sites present as $TiO^-$ leading to an increase in the adsorption and decolourization of CV molecules. Furthermore, as the pH increases, a more efficient generation of ·OH with increasing concentration of $OH^-$ is expected ($OH^- + h^+ \rightarrow {}^{\cdot}OH$). At pH less than $pH_{ZPC}$, the photocatalyst surface has more positive charge; hence, hindering the decolourization process. It is worth noting that a complete decolourization is achieved less than 5 h at natural pH (6.3). This represents a practical advantage despite the relatively long time for complete decolourization.

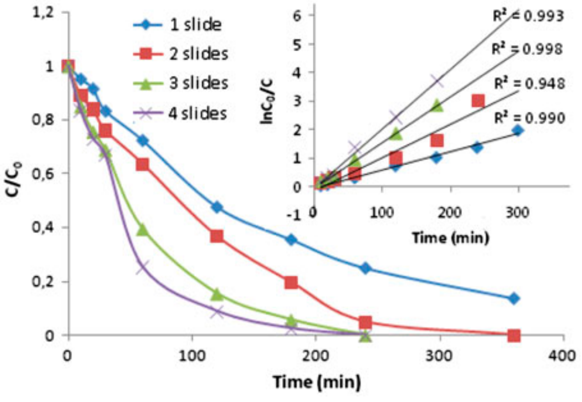

Fig. 4. Effect of number of slides of P25 $TiO_2$-supported on the CV photodecolourization under natural sunlight. Conditions: natural pH (6.3); [CV]: $10^{-5}$ mol/L.

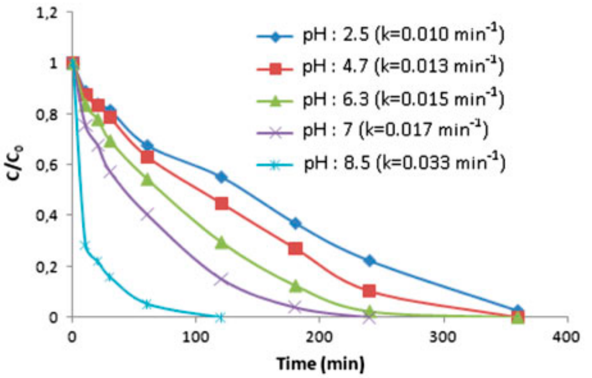

Fig. 5. Effect of pH on the CV decolourization process using P25 $TiO_2$-supported under natural sunlight irradiation. Conditions: [CV]: $10^{-5}$ mol/L; number of $TiO_2$ glass slides: 2.

### 4.4. Effect of the presence of metallic cations

Since industrial effluents may contain different types of metallic cations along the organic pollutants, it is therefore interesting to investigate their effects on the supported photocatalyst efficiency. Fig. 6 shows the effect of some metallic cations ([cation] = 50 mg/L) in the photocatalytic decolourization of CV under sunlight. From this figure, it can be noticed that the presence of $Cu^{2+}$, $Fe^{2+}$ and $Mn^{2+}$ inhibits the photocatalytic process. This negative effect could be accounted for by: (i) the addition of these cations decreasing the pH solution, which leads to a decrease of the decolourization rate as shown previously (section 4.3). (ii) These cations can be adsorbed on the $TiO_2$ surface hence blocking its active sites. (iii) The oxidation of

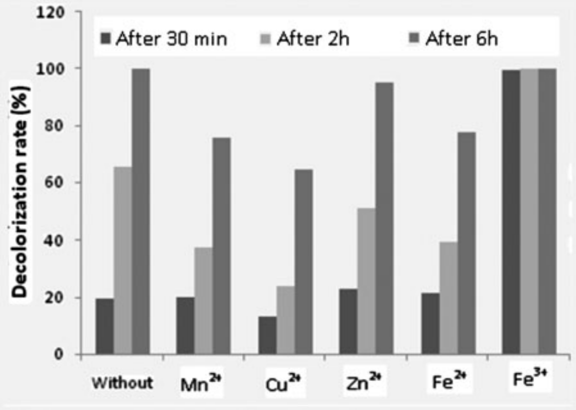

Fig. 6. Effect of metallic cations on the photocatalytic decolourization using P25 $TiO_2$-supported under natural sunlight irradiation. Conditions: [CV]: $10^{-5}$ mol/L; number of $TiO_2$ glass slides : 2, [cation] = 50 mg/L.

reduced metal cations by $^{\cdot}OH$ radicals or by photogenerated holes. On the other hand, $Zn^{2+}$ has a negligible negative effect estimated at 4.1% of the decolourization process after 6 h of irradiation. This may be due to the fact that $Zn^{2+}$ is at its maximum oxidation state resulting in its inability to inhibit the photocatalysis reaction [26]. Furthermore, the $Fe^{3+}$ strongly enhances the process leading to a decolourization rate of 100% just after 30 min. This increase is accounted for by the participation of $^{\cdot}OH$ radicals which are known to be generated by the solar photolysis of the $Fe(III)\text{-}OH^{2+}$ complex [27] formed in solution by excess of $Fe^{3+}$ yielding, in the present case, a solution pH of 3.7. In this reaction, $^{\cdot}OH$ radicals are generated and $Fe^{3+}$ is reduced to $Fe^{2+}$ when the light of wavelength from 300 to 400 nm is irradiated [27,28] (Eq. (5)).

$$Fe^{3+} + H_2O \xrightarrow{hv} Fe^{2+} + H^+ + {}^{\cdot}OH \qquad (5)$$

### 4.5. Photocatalyst reuse

It is known that the main drawback of the photocatalytic processes which use dispersed photocatalysts and artificial UV radiation is the relatively high cost of the UV lamp energy together with the difficulty step of recuperation of the photocatalysts. Consequently, it is interesting to reduce the cost of this process by making reuse of the supported photocatalyst and use of natural sunlight. In this study, the reuse of the photocatalyst was tested under the same working conditions for 4 d in a row. Prior to their reuse, the slides of the P25 $TiO_2$-supportedwere washed with double distilled water then dried for 2 h at 110˚C. The results (Fig. 7) show that it is possible to reuse the P25 $TiO_2$-supported without any significant decrease of the removal degree of CV for at least the first three uses, the apparent constant rates being equal to 0.0213, 0.0116 and 0.0101 $min^{-1}$ for the first, second and third uses, respectively. However, the slight decreasing observed in the fourth use (84.5%; $k$ = 0.0077 $min^{-1}$) may be due to the adsorption of byproducts of the CV decolourization on the surface of the photocatalyst together with some loss of $TiO_2$ particles. However, considering the simple method of deposition of $TiO_2$, this result indicates a relatively good stability after four usages of the $TiO_2$.

### 4.6. Comparative efficiency of CV decolourization

Fig. 8 illustrates the decolourization process using P25 $TiO_2$-supported and P25 $TiO_2$-dispersed irradiated by UV and natural sunlight. The results show that at natural pH (6.3) the P25 $TiO_2$-dispersed is more efficient in both cases (under UV and natural sunlight) than the P25 $TiO_2$-supported. This is due to the large surface area of catalyst available for the reaction between the substrate and P25 $TiO_2$-dispersed. Furthermore, it can be noticed that the decolourization processes using P25 $TiO_2$-dispersed and P25 $TiO_2$-supported are more significant and faster under natural sunlight than under UV-lamp. This can be due to the relatively high intensity of sunlight received on the earth surface on these summer days and is characterized by an important flux of UV photons. As an indication, the intensity of sunlight measured at $\lambda$ = 365 nm, during the days of experimentation, was in average 1.6 $mW/cm^2$ while that of the UV lamp was around 0.3 $mW/cm^2$, measured at a distance of 10 cm

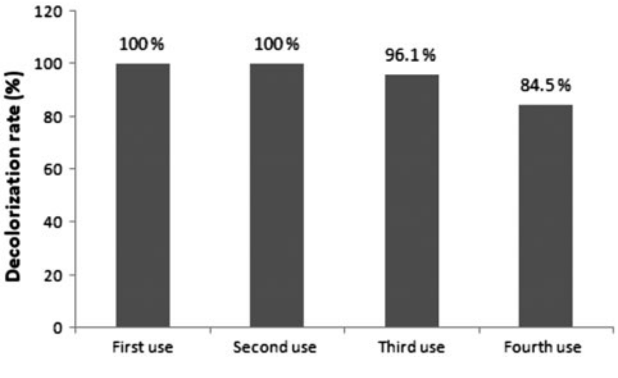

Fig. 7. Reuse of the P25 $TiO_2$-supported for the decolourization of CV under natural sunlight. Conditions: [CV]: $10^{-5}$ mol/L; natural pH (6.3); number of $TiO_2$glass slides: 2; time: 6 h.

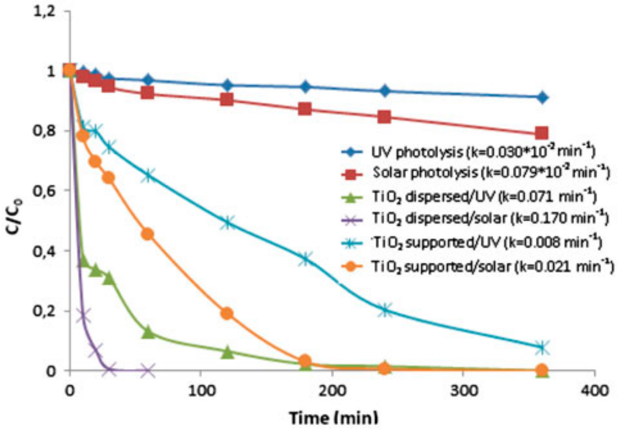

Fig. 8. Photocatalytic decolourization of CV using P25 $TiO_2$-supported and dispersed under UV and sunlight irradiations. Conditions: [CV]: $10^{-5}$ mol/L; natural pH (6.3); [P25 $TiO_2$-dispersed]: 0.5 g/L; number of glass slides: 2.

from the lamp. Consequently, a higher $TiO_2$ photoactivity is expected. Moreover, the contribution of the solar photolysis of CV is relatively important which may be related to the polychromatic nature of the sunlight. The extent of decolourization, due to photolysis, was in average equal to 20%. Although the P25 $TiO_2$-dispersed is more efficient than the P25 $TiO_2$-supported, it is worth recalling the main drawback associated with the use of the former, namely the post filtration step, the tedious recuperation of photocatalyst. Besides, the quantity of P25 $TiO_2$-dispersed (0.5 g/L) is relatively larger than the P25 $TiO_2$-supported (0.05 g/slide corresponding to approximately 0.4 g/L of $TiO_2$). Taking into account these considerations, the use of P25 $TiO_2$-supported in the CV photocatalytic decolourization under natural sunlight confirms the relatively higher efficiency that offers this method for wastewater treatment.

## 5. Conclusion

The use of $TiO_2$-supported on glass slides as photocatalyst has been proven to be effective for the complete CV decolourization under natural sunlight irradiation. The decolourization process increases with the decrease in the initial concentration of substrate and increase in the number of $TiO_2$ glass slides. The pH has a net effect on the process kinetics but yields a complete CV decolourization in the pH range investigated. The study of the effect of the metallic cations shows that their presence with the CV has a net negative effect in the case of $Cu^{2+}$, $Fe^{2+}$, $Mn^{2+}$ and to a lesser extent in the case of $Zn^{2+}$. However, $Fe^{3+}$ contributes positively to the decolourization process. Finally, the results show that the P25 $TiO_2$-supported can be reused four times without any significant decrease in its activity. The P25 $TiO_2$-supported presents numerous economical advantages such as relatively small quantity of $TiO_2$ used, absence of filtration and possibility of its reuse. Another major economical advantage is the use of free and clean energy of the sun for the decolourization of organic dyes, particularly in Algeria where the average sunshine duration is about 2,500 h per year which can reach 3,900 h per year in the south of the country [29]. Consequently, further investigations are required at the plant scale for industrial effluent treatment.

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