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ORIGINAL ARTICLE

UV/TiO_2 PHOTOCATALYTIC OXIDATION OF COMMERCIAL PESTICIDE IN AQUEOUS SOLUTION

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ABSTRACT

Background: The use of pesticides massively in agriculture is considered among the causes of pollution most probable of surface and ground water. These organic compounds are very hazardous and harmful to health, as most are toxic and carcinogenic, even at low concentrations. For these reasons, in recent years, environmental regulations in many countries have become strict for the production of drinking water and wastewater treatment, in particular concerning pollution by pesticides. **Objective**: The removal of pesticides from contaminated water is an extremely complex problem due to the wide range of pesticides of multiple chemical structures and properties. Among the unconventional methods of treatment of polluted water, heterogeneous photocatalysis has been used for the treatment of several pollutants such as pesticides. The objective of this work is the evaluation of this method for the elimination of a pesticide under different operating conditions **Methods**: Photocatalytic degradation of aqueous commercial pesticide "Mythos" solution has been tested by using TiO_2 as a photocatalyst and a new batch reactor using low voltage UV lamps as an irradiation source. The influence of some parameters such as the presence of UV irradiation, aeration, pH, pesticide concentration and time of reaction was examined. The effect of some inorganic anions, such as Cl-, SO_4^{2-} and NO_3^-, commonly present in real effluents, on the photocatalytic degradation, was also studied. **Results**: The degradation rates were found to be strongly influenced by all the above parameters, except pH which has a moderate effect. It is found that the photocatalytic degradation process follows first-order reaction kinetics represented by the Langmuir-Hinshelwood mechanism. The presence of Cl- and SO_4^{2-} anions led to a slight decrease of the effectiveness of the photocatalytic degradation. However, the observed inhibitive effect on the degradation of the tested fungicide is shown to follow the order: $Cl^- < SO_4^{2-} < CO_3^{2-} < PO_4^{2-} < NO_3^-$. The aeration of the medium bulk enhanced the photodegradation rate of the pollutant. **Conclusion**: based on the results, the photocatalytic degradation using a low-voltage UV light could be a useful method for the removal, of wastewaters containing Mythos.

Keywords*: Photocatalysis, TiO_2, low-voltage UV, Mythos, kinetic model.*

1. INTRODUCTION

A diversity of organic pollutants especially pesticides are introduced into the water system from different sources such as industrial water discharges, agricultural runoff, and chemical spills [1,2]. Their toxicity, stability to natural decomposition, and persistence in the environment have been the cause of much concern to the societies and regulatory authorities around the world [3,4].

The control of organic pollutants in water is an important measure in environmental protection. Among many processes proposed and/or being developed for the destruction of the organic contaminants, biodegradation has received the greatest attention. However, many organic chemicals, especially those that are toxic or refractory, are not amendable to microbial degradation [5].

On recent years, novel methods for water and air purification have been developed including chemical, electrochemical and photochemical processes [6,7]. Indeed; photocatalytic degradation has been shown to be a promising technology for the treatment of water contaminated with organic and inorganic pollutants [8,9].

Heterogeneous photocatalysis involves the use of a semiconductor material which is excited by light with energy equal to or greater than the band gap. This leads to the formation of electron/hole pairs which react at the particle-water interface resulting in the degradation of chemical species by both oxidative and reductive pathways. Titanium dioxide (TiO_2) is the photocatalyst of choice for water decontamination treatment because it is not soluble under normal pH ranges found in natural water. In the other hand, it is photoactive, photostable, and relatively inexpensive. However, TiO_2 is a wide band gap semiconductor (E_{bg}= 3.2 eV for anatase) and can only be excited by UV light, meaning that only 5% of the solar spectrum can be utilised.

In our previous paper [10], we mentioned that during the adsorption phase, consider as the vital stage in photocatalytic degradation of organic pollutants on TiO_2 surface, the amount of pyrimethanil fixed by the particles of TiO_2, and depend on several parameters. The time needed to reach adsorption equilibrium increase with concentration, but not exceed 30 minutes. The uptake of pyrimethanil was found to increase with the increase of salt's concentration especially in the presence of NaCl and $NaHPO_4$.

In the present work, photocatalysis based on TiO_2 and a batch reactor using low voltage UV lamps as an irradiation source were applied to degrade the commercial solutions of pyrimethanil, named Mythos. The effects of the major system parameters (pH, initial concentrations of Mythos, concentration of ions and presence of O_2) on the degradation of this fungicide in presence of TiO_2 were studied.

2. MATERIALS AND METHODS

2.1. Chemicals

Pyrimethanil is an anilinopyrimidine, 4,6-dimethyl-N-phenyl-2-pyrimidinamine. Imazalil sulfate: 1-[2-(2,4-dichlorophenyl)-2-(2-propenyloxy)ethyl]-1H- imidazole sulfate. The commercial pyrimethanil used is named MYTHOS SC300 purchased from Bayer Corporation. The molecular structure of this pesticide is shown in Figure 1. All chemicals used in the experiments were of analytical pure grade and used without further purification. A stock pyrimethanil standard solution (100 mg in water) was prepared in doubly distilled water.

The photocatalysts used in this experiment were technical grade: Titanium (IV) oxide, anatase (purity > ≥99%), was purchased from Sigma-Aldrich and used as received, and TiO_2 Degussa P-25 (anatase/rutile, surface area 56 m^2 g^{-1}, nonporous).

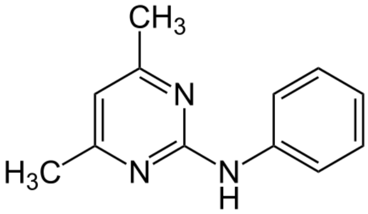

Figure 1: The figure presents the molecular structure of Pyrimethanil.

2.2. Photocatalytic Reactor: This photo-reactor, of a capacity of 1 L, has a cylindrical form made with Pyrex vessel. The cover of the photoreactor has several ports for sampling and for introducing oxygen into the reaction mixture. It's placed in the center of a stainless steel cylinder with 6 low-pressure UV lamps (8 watts, TUV G8T5, Philips), with maximum emission wavelength mainly around 365 nm, on axial position allowing that the intensity received by the effluent in the Pyrex reaction vessel is uniform. The photoreactor has placed in a closed box ($1m^3$). The ventilation of this enclosure is ensured by two fans.

2.3. Analysis and procedure: Absorbance was measured using UV-Vis spectrophotometer (Jasco V630). The photocatalytic experiments were performed using a volume of the solution of 1L containing the concentration 15 mg/L of Mythos in the presence suspension of 1 g/L TiO_2 powder. Before irradiation, the suspension was magnetically stirred in the dark for 60 min to reach adsorption–desorption equilibrium. During irradiation, a test of samples was withdrawn with the help of a syringe and filtered through syringe (45 μm) filters to remove TiO_2 particles before analysis. Total volume of the samples withdrawn from each experiment was less than 10% (by volume) of the reaction solution. The pH of the reaction solution was adjusted by adding H_2SO_4 or NaOH. Materials and Methods are written in this area. Describe in detail the technic used, the Name and the references of laboratory materials used should be cited.

3. RESULTS

3.1. Photolysis

As shown in Figure 2, the degradation of Mythos in aqueous solution by photolysis at 365 nm (without TiO_2) is very slow in comparison with photocatalytic degradation, as only 10% of the initial amount of the compound is degraded after 5 h of irradiation without TiO_2.

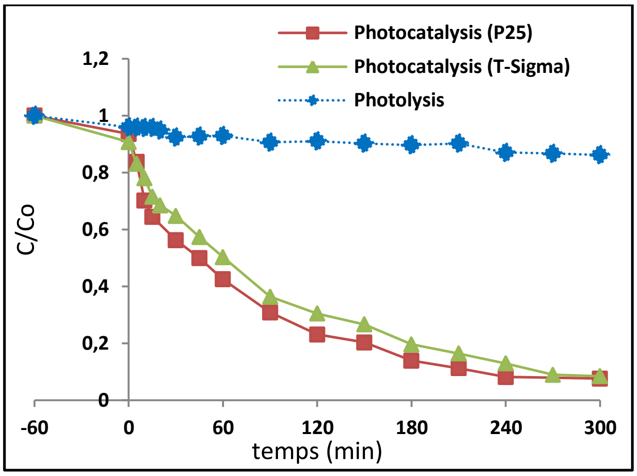

Figure 2: The figure presents the degradation kinetics of Mythos in the presence and the absence of TiO_2 P25 and TiO_2 Sigma, using UV/O_2 system ([Mythos] = 15 mg/L; TiO_2 = 1 g/L).

This fact could be attributed to a relatively low substrate adsorption at this wavelength and the quantum yield of photolysis. Conversely, in the presence of TiO_2 91% and 82% of initial concentration the Mythos was degraded, respectively for TiO_2 P-25 and TiO_2 anatase. Consequently, the decrease of the Mythos concentration in the presence of TiO_2 is mainly due to the photocatalytic degradation. Similar results were observed for photocatalysis of diuron, imidacloprid, formetanate and methomyl [10]. As the TiO_2 P-25 gives a slightly better result than the other form, it will be used for the rest of this work.

3.2. Effect of substrate initial's concentration

Figure 3 presents the evolution of illuminated suspensions containing different concentrations of Mythos in the presence 1 g/L of TiO_2, at free pH, as a function of time. The increase in the initial concentration in the range of 15-60 mg/L is traducing by a decreased of a reaction rate. Thus, the higher the initial concentration of the pesticide, the longer the time it takes to remove it. Similar trends have been observed for the photocatalytic degradation of triclopyr and daminozid [12], carbofuran [13], propham and tebuthioron [14].

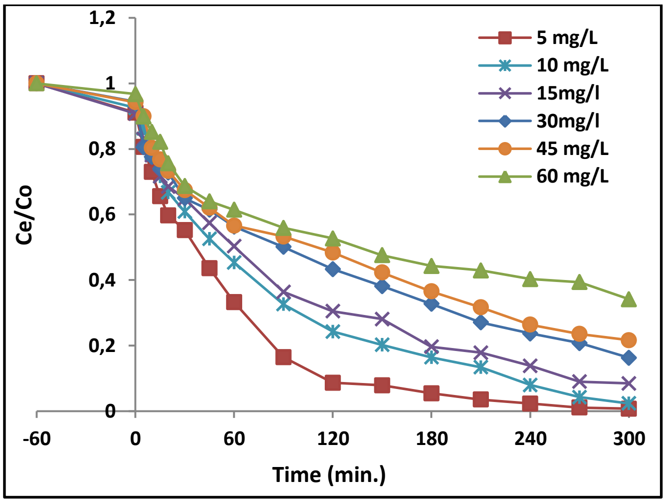

Figure 3: The figure presents the influence of the initial concentration on the photodegradation of Mythos in the presence of 1 g/L of TiO_2 P-25.

As indicated in several investigations, this trend maybe explains considering that as the concentration of the target pollutant increase, more molecules of the compound are adsorbed on the surface of the photocatalyst. Therefore, the reactive species ($OH^•$ and $O_2^•$) required for the degradation of the pollutant must increases. However, the formation of $OH^•$ and $O_2^•$ on the catalyst surface remains constant for a given light intensity, catalyst amount, and duration of irradiation. Hence, the available $OH^•$ radicals are inadequate for pollutant degradation at higher concentrations. Consequently, the pollutant degradation rate decreases as the concentration increases [15]. In addition, an increase in substrate concentration can lead to the generation of higher concentration of intermediates, which may adsorb on the surface of the catalyst. Slow diffusion of the generated intermediates from the catalyst surface can result in the deactivation of active sites on the photocatalyst and result in a reduction in the degradation rate. However, at low concentration, the number of catalytic sites will not be limiting factor and the rate of degradation will be proportional to the substrate concentration. Similar trends have been reported for the photocatalytic degradation of propachlor [14], acephate [16], 2,4-DNP [17], isoproturon [18], and diphenamid [19].

3.3. Kinetic study

A Linear relationship was found by plotting ln(C_e/C_o) versus irradiation time (t) (Figure 4) displayed that Mythos photocatalytic degradation followed the pseudo-first-order kinetic for the different concentrations studied. The pseudo-first-order rate constants (k_{app}) were determined from the slope value of the line upon linear regression according to "Eq. (1) ":

$$Ln\frac{C_e}{C_o} = K_{app}t \qquad (1)$$

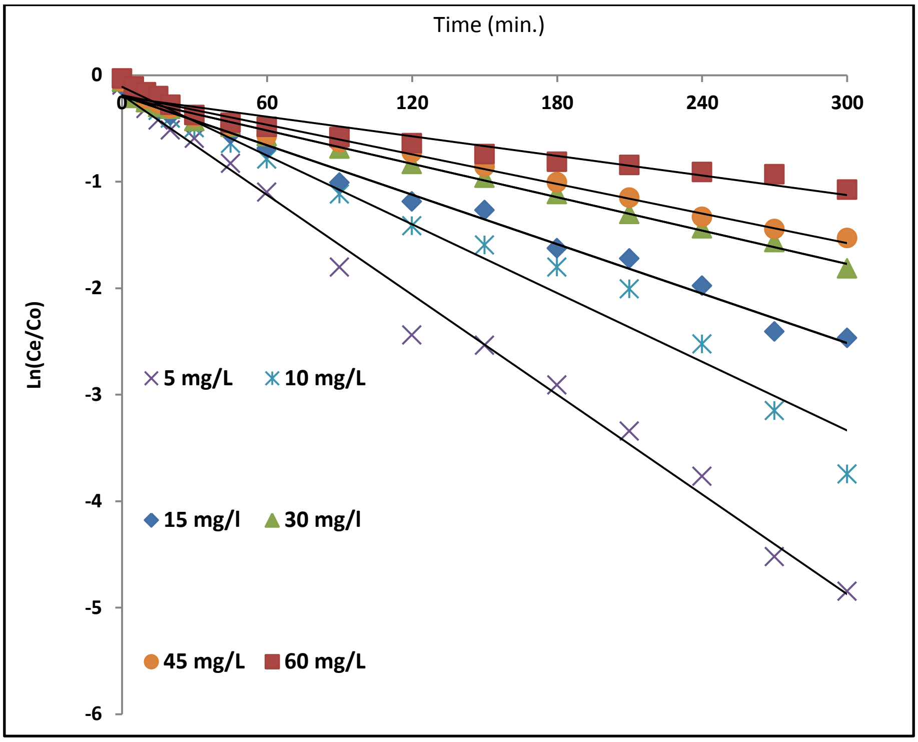

Figure 4: The figure presents the variation of ln(C_e/C_o) versus irradiation time for Mythos ([TiO_2] = 1g/L).

3.3.1. Analysis of degradation using the Langmuir-Hinshelwood (L-H) Kinetic Model

The Langmuir-Hinshelwood model has been used to describe the kinetics of the reaction [21, 22]. This model has been used extensively to describe experimental results in heterogeneous photocatalysis reactions [23, 24]. The rate of the photocatalytic reaction depends on the fraction of the sites covered by the Pyrimethanil and the k_{LH} a rate constant which depends on the concentration of $HO^{\bullet}$ produced.

Although some authors consider the Langmuir-Hinshelwood model not to be a sufficient process (Yu et al., 2003; Zhu et al., 2005), this model is widely used because it correlates the rate of degradation to the instantaneous concentration. The equation used is:

$$v_o = -\frac{dc}{dt} = KC = K_{LH}\theta = K_{LH}\frac{k_r C_o}{1+kC_o} \qquad (2)$$

The linear form of "Eq. (2)" is presented in "Eq. (3)"

$$\frac{1}{vo} = \frac{1}{k_{L-h}\,KCo} + \frac{1}{k_{L-H}} \qquad (3)$$

Where,

C_o: Mythos initial concentration (mg/L),
k_{L-H}: Adsorption equilibrium constant under irradiation (L/mg),
V_o: Substrate degradation reaction rate (mg/L.min),

T: irradiation time (min),
K_r: Specific rate constant for the oxidation of the organic compound (mg /L min).

The slop of $\frac{1}{\text{vo}}$ as a function of $\frac{1}{\text{Co}}$ is linear, and the calculated constants k_r and K_{L-H} were k_r= 0.0373 mg/L.min and K_{L-H}= 0.572 L/mg, respectively. Hence, the calculated values of K_{L-H} (0.572 L/mg) and K_{ads} (0.0422 L/mg L) were different. The difference obtained in this study can be attributed to the inexactitude of Langmuir and Langmuir–Hinshelwood alignments. Thus, the adsorption affinity of Mythos on the surface of TiO_2 can be reflected by the parameter K_{L-H}.

3.3.2. Half-life of the reaction

One of the most useful indications for evaluating the reaction rate of first order kinetics is the calculation of the half-life of the reaction. The integration of the Langmuir-Hinshelwood equation (L-H) over time gives "Eq. (4)":

$$t = \frac{1}{k_r K_{L-H}} Ln \frac{C}{C_o} - \frac{1}{k_r}(C - C_o) \qquad (4)$$

Where **C_0**: Initial pollutant concentration (mg/L),
C: Concentration of organic compound (mg/L),
t: Reaction time during substrate degradation (min).

The half-life of the reaction corresponds to the disappearance of half of the initial amount of the substrate. The theoretical reaction time $t_{1/2}$ is estimated by "Eq. (5)":

$$t_{\frac{1}{2}}\,(estimated) = \frac{0{,}5C_o}{k_r} + \frac{Ln2}{k_r K_{L-H}} \qquad (5)$$

On the other hand, for the apparent reaction constant reaction, the half-life ($t'_{1/2}$) is calculated according to "Eq. (6)":

$$t'_{\frac{1}{2}}\,(calculated) = \frac{Ln2}{k_{app}} \qquad (6)$$

The different values of $t_{1/2}$ (estimated) and $t'_{1/2}$ (calculated) obtained for Mythos for different initial (15 to 60 mg/L) in the presence of TiO_2 (1 g/L) are given in the Table below.

Table 1: Estimated and calculated half-life values for the various initial concentration of Mythos.

[Mythos] mg/L	$t'_{1/2}$cal. (min.)	$t_{1/2}$est. (min.)	$\Delta t_{1/2}$ (min.)
5	46.2	33.8	12.4
10	70.7	56.5	14.2
15	99.0	79.3	19.7
30	173.3	147.5	25.8
45	261.6	215.8	45.1
60	346.6	284.0	62.6

The graphical exploitation of the half-life time as a function of the initial concentration gives straight lines (Figure 5).

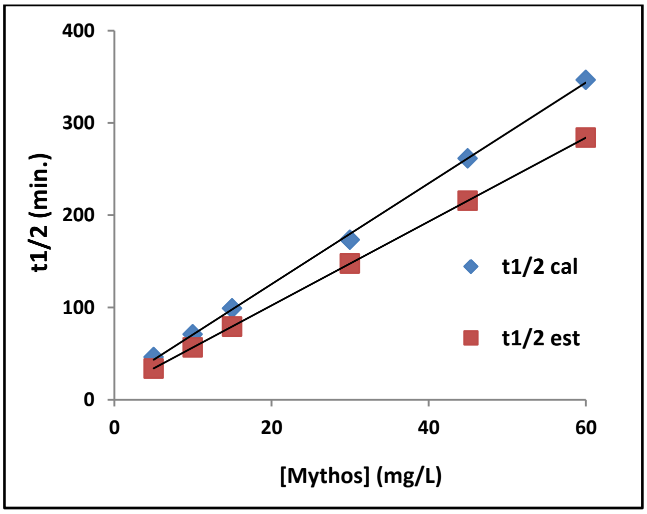

Figure 5: The figure presents the half-life time estimated ($t_{1/2}$ est) and calculated ($t_{1/2}$ cal) as a function of different concentrations of Mythos, [TiO_2 = 1 g /L].

These Figures show a difference between $t_{1/2}$ and $t'_{1/2}$, which has become significant with the increase in the initial concentration of the dye. This behaviour could be explained by the fact that the photodecomposition of the Mythos gives rise to intermediates, which could also be competitively adsorbed on the surface of TiO_2, resulting in the delay of half-life time.

3.4. Effect of pH

Many studies have indicated that the pH of a solution is an important parameter in the adsorption and photocatalytic degradation of organic compounds. This is because pH of the aqueous solution affects the surface charge of photocatalyst of an organic pollutant. Thus the electrostatic interaction between semiconductor surface, solvent molecules, substrate and charged radicals formed during photocatalytic oxidation [12,15,25].

In addition protonation and deprotonation of the organic pollutants can take place depending on the solution pH. Sometimes protonated products are more stable under UV-radiation than its main structures [27].

For this study, the effect of initial's pH of solution on the photodegradation of the pesticide is given in Figure 6. The analysis of the results show that the pH has a slight effect was observed under the conditions examined. At acid pH, a total degradation was observed after 240 minutes of irradiation, compared to 91% degraded for pH 6 and pH 9. Similar observation has also been made for the degradation rate of methamidophos was nearly independent of solution pH when Re-doped TiO_2 was used as the photocatalyst [28].

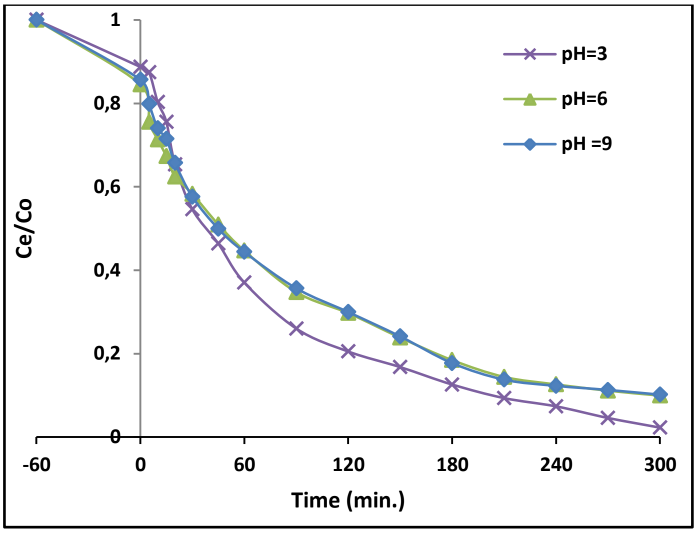

Figure 6: The figure presents the effect of pH effect on photocatalytic degradation of Mythos ([Mythos] = 15 mg/L, TiO_2=1g/L).

3.5. Effect of Inorganic ions

A number of studies demonstrated that some anions (phosphate, nitrate, sulfate, and chloride), found in natural or polluted waters, can affect the photocatalytic degradation rate of organic pollutants since they can be adsorbed onto the surface of TiO_2 [29, 30]. Thus, the presence of anions is reported to alter the ionic strength of the solution, which influences the catalytic activity and the photocatalytic degradation. Depending on the solution pH, they can also compete with the target pollutant for the active sites. The adsorption of water components can reduce the formation of $OH^{\bullet}$ radicals. Although hydroxyl radical scavenging by the anions bicarbonate, phosphate, nitrate, sulfate, and chloride resulted in corresponding anion radicals, they have lower oxidation potential. Consequently, all these reactions can influence the overall rate of photocatalytic oxidation.

In order to examine the influence of certain ions on the photocatalytic degradation of Mythos, experiments were carried out in the presence of 0.1 mol/L of the following ions: Cl^-, NO_3^-, PO_4^{2-}, and SO_4^{2-}. Figure 7 presents the results founded. The observed inhibitive effect on the degradation of the tested fungicide is shown to follow the order: $Cl^- < SO_4^{2-} < PO_4^{2-} < NO_3^-$. Negligible inhibition was observed for sulfate and chloride relative to the other ions. Similar trends have been observed for the photocatalytic degradation of 4-flurophenol and turbophos [31,32].

The detrimental effect of the tested anions may be attributed to their competition for the active sites on the TiO_2 surface and catalyst deactivation which subsequently, decrease the degradation rate. A major drawback from the high reactivity and non-selectivity of $OH^{\bullet}$ is that it also reacts with non-target compounds present in the background water. This results in a higher $OH^{\bullet}$ demand to accomplish the desired degree of degradation [30,32]. This inhibition may also be due to the reaction of positive holes and hydroxyl radicals with anions that behaved as radical (h+ and $OH^{\bullet}$) scavenger resulted in longer Mythos removal [32,33].

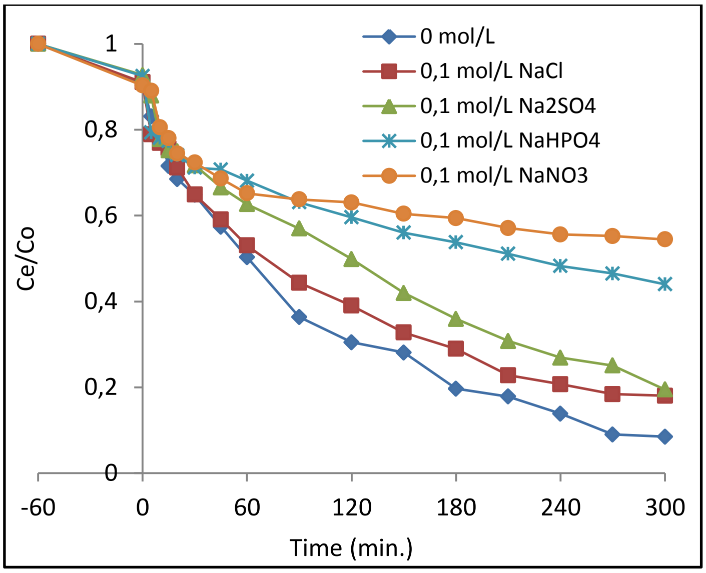

Figure 7: The figure presents the degradation of Mythos in the presence of 0.1 mol/L of different ions ([Mythos] = 15 mg/L, TiO_2 1g/L]).

3.6. Effect of medium bulk aeration

In the water purification by heterogeneous photocatalysis, the pollutants are usually organic. Their reaction in the presence of oxygen is:

$OH^{\bullet}$ + pollutants + O_2 $\longrightarrow$ CO_2 + H_2O + intermediate products [34].

Oxygen is essential for complete degradation and should not be in competition at the level of adsorption with other reactive species on the catalyst for this, we studied the effect of the aeration of the medium bulk on the photocatalytic degradation of pollutants. The results are shown in Figure 8:

We note that with aeration of the heterogeneous medium (TiO_2/UV), there is an acceleration of the decrease in the concentration of the pollutant. Other research has shown that the rates and efficiencies of photo-assisted degradation of organic substrates are reported as significantly improved in the presence of oxygen or by the addition of several oxidizing species such as peroxydisulfate or peroxides [35-37]. This acceleration may be related to inhibition of the recombination of electron-hole pair and also by the production of more radicals $OH^{\bullet}$ in the middle, so we conclude that oxygen (O_2) here plays the role of a catalyst but since it is not regenerated at the end of the reaction, it affects the performance of the degradation reaction by increasing, so we can conclude that reacts with the pollutant.

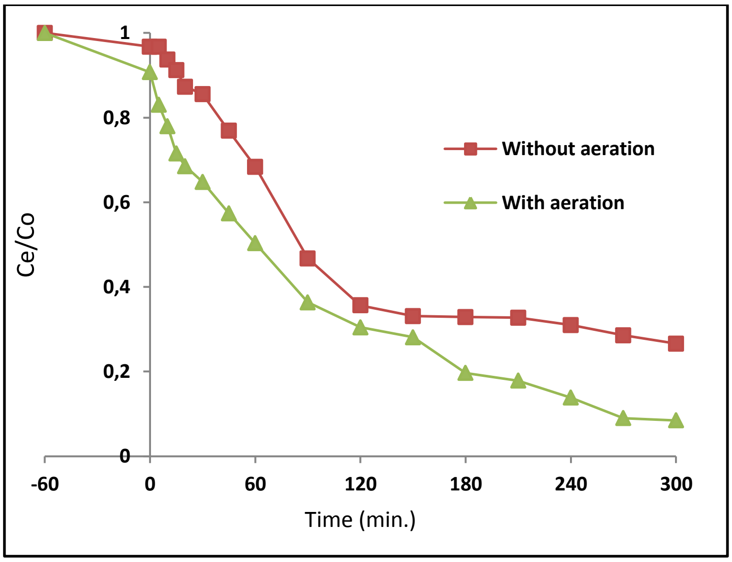

Figure 8: The figure presents the effect of aeration on the Mythos's photodegradation. ([Mythos] = 15 mg/L, TiO_2 = 1g/L).

5. CONCLUSION

Kinetics degradation of Mythos, a broad spectrum fungicide, often used to treat seeds, has been investigated in aqueous solution using UV light in the absence and in presence of slurry titanium dioxide (TiO_2). The degradation efficiency (%) of Mythos by using TiO_2 photocatalyst depends strongly on the concentration of this pesticide. Kinetic parameters were experimentally determined and an apparent first order kinetic was observed. The application of the Langmuir kinetic model allowed us to determine the constants related to this model. On the other hand, we also determine the half-life time. A difference between $t_{1/2}$ (estimated) and $t'_{1/2}$ (calculated) was found and become significant with the increase in the initial concentration of the pesticide. The initial pH of pesticide has no significant effect on photodegradation of this compound. In addition, the photodegradation efficiency of Mythos was slow in the presence of PO_4^{2-} and NO_3^-. The aeration of the medium bulk enhanced the photodegradation rate of the pollutant.

These results show that the photocatalytic process, based on TiO_2 and an economic UV light, are able to degrade the pesticide Mythos and may find application in the remediation of water contaminated with pesticide residues.

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