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Research Article

Remediation by heliophotocatalysis of water contaminated by sulfamethoxazole

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Abstract : The présence of sulfamethoxazole in surface waters constitutes an environmental problem. The objective of this work was to determine the effectiveness of photocatalysis by sunlight in the elimination of sulfamethoxazole. Powdered titanium dioxide was fixed on clay balls by calcining in an oven at 400°C. Water solutions spiked with sulfamethoxazole were prepared. Photolysis and photocatalysis tests were carried out by exposing the reactors to solar irradiation. The results showed that the degradation rate was 94.95% after 300 minutes of solar irradiation in the presence of 60 g of clay balls containing TiO_2. While the yield of direct solar photolysis was about 5%. This study was an alternative to the depollution of water contaminated by sulfamethoxazole and proves that such water can indeed be rehabilitated by solar photocatalysis. This process constitutes, from an economic point of view, a significant advantage compared to conventional processes for the treatment of water contaminated by persistent organic pollutants.

Keywords : *Heliophotocatalysis ; Sulfamethoxazole, titanium dioxide, ultra pure water.*

1.INTRODUCTION

Sulfamethoxazole (SMX) is one of the most active antibiotic compounds used in sulfa groups. After use, SMX and its metabolites are excreted in urine and feces via wastewater treatment plants (WTPs) and end up in surface waters [1][2][3]. Previous studies show that SMX is removed at a low rate in wastewater treatment plants and is often found at a high frequency in surface waters [4].

As a compound of the sulfonamide group, SMX has antimicrobial properties that could promote antibiotic resistance in the environnement [5][6]. Therefore, cheap and effective processing techniques to decompose or transform this compound into non-toxic compounds are needed before releasing it into the environment.

Different techniques have been proposed to eliminate pharmaceutical residues in WWTPs, including biological, physical and chemical processes. Advanced oxidation processes (AOP) have proven to be increasingly effective and most promising for the removal of these compounds in wastewater treatment plants.

Clay-based hybrid photocatalysts have given rise to a rich database for their tailored microstructures, characterizations and environment-related applications. Undoubtedly, the control and preparation of new clay-based photocatalysts will continue to see many breakthroughs in the field of solar technologies [7]. The objective of this study is to determine the effectiveness of photocatalysis by sunlight in the elimination of sulfamethoxazole. Specifically, these are :

1. Determine the kinetics of the solar photolysis of sulfamethoxazole by clay not supported by TiO_2.
2. Determine the kinetics of degradation of sulfamethoxazole by clay supported by TiO_2 by following the influence of certain parameters such as the effect of TiO_2 ; of the initial SMX concentration and the pH of the medium.

2. MATERIALS AND METHODS

2.1. Material : The clay used comes from the underground of the city of Daloa. It was taken after drilling a water well approximately 4 m deep. The technical equipment used consists of chemicals, glassware and measuring and analysis devices. The analysis grade ethanol and acetone were 99.99% pure. Acetonitrile is HPLC grade with 99.99% purity. Formic acid for the acidification of mobile phases is 99.99% pure. The catalyst is titanium dioxide (TiO_2) with a purity of 99.95%. Orthophosphoric acid (H_3PO_4) and sodium hydroxide (NaOH) are more than 95% pure. The 99.99% sulfamethoxazole standards were manufactured by Sigma Aldrich.

The glassware used, all made of Pyrex, is made up of beakers, mini-reactors (50 mL test tubes) and 1000 mL volumetric flasks which were used to prepare the solution, a micropipette (10-100 µL), and vials. This glassware, washed and rinsed successively with tap water, demineralized water then distilled water, was carefully rinsed and calcined in a muffle furnace to destroy the forms of organic matter.

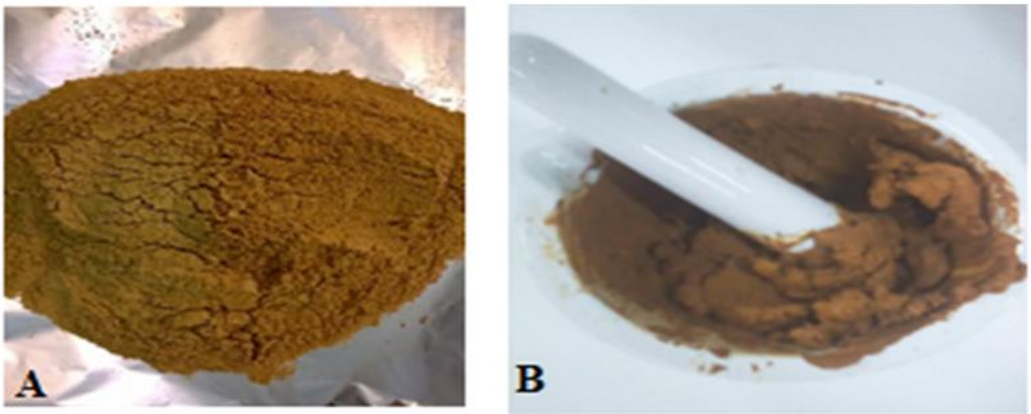
Apparatus : A Denver Instrument S-602 model balance was used for weighing the beads for the physical characterization of the beads and for the adsorption and photocatalysis tests. A RETSCH brand sieve made up of 7 sieves was used to split the clay into powder. The diameters of this sieve are between 45 µm and 2 nm. A MEMMERT type oven from NEO-TECH SA (Belgium) was used to dry the adsorbents and sterilize the glassware. A NABERTHERM brand oven was used for the calcination of the materials. A high-performance liquid chromatography system (Shimadzu Prominence) coupled with a UV detector (SPD-20A) was used for the identification and quantification of Sulfamethoxazole. This chromatograph is controlled using software called LabSolutions.

2.2. Methods : Preparation of solutions : We prepared a solution.7000 mg. L^{-1} by dissolving 0.35 g of sulfamethoxazole in 50 mL of distilled water. This stock solution was stored in the refrigerator. From this mother solution, daughter solutions were prepared and injected into the chromatograph for all experimental activities. Chromatographic analysis conditions : The chromatographic analysis conditions of sulfamethoxazole are reported in Table I.

Table I : Chromatographic analysis conditions for Sulfamethoxazole.

Stationnary phase	**LiChrospher Ø 4/4,6mm 100 RP-18**
Mobile phase	Eau/Acetonitrile (50% v/v) (Acide formique 0,1 %)
Elution method	Gradient LPGE (Low pressure gradient system)
UV Detection (nm)	230 nm ou 278 nm (pH libre = 6.5)
Throughput	1 mL.min^{-1}
Retention time	10.507 min
Pressure	9.5 Mpa
Injection volume	10 µL^{-1}
Limit of quantification	10 µg.L^{-1}

- **Bead development protocol** : the development of clay beads with or without TiO_2 was carried out according to each case [8].
- **Clay balls :** The clay taken was ground in a porcelain mortar (A). The powder obtained is sieved on a sieve of six sieves whose diameters were between 2 mm and 0.45 µm. To make the balls, 40 mL of ultra-pure water are added to 100 g of clay powder to obtain a homogeneous paste (B). Beads of approximately 0.3 cm in diameter were made. These beads were then dried at 105°C for 24 hours and then baked at 550°C in the oven to make them resistant in water (C) and also remove all forms of residual organic matter (Figure 1).



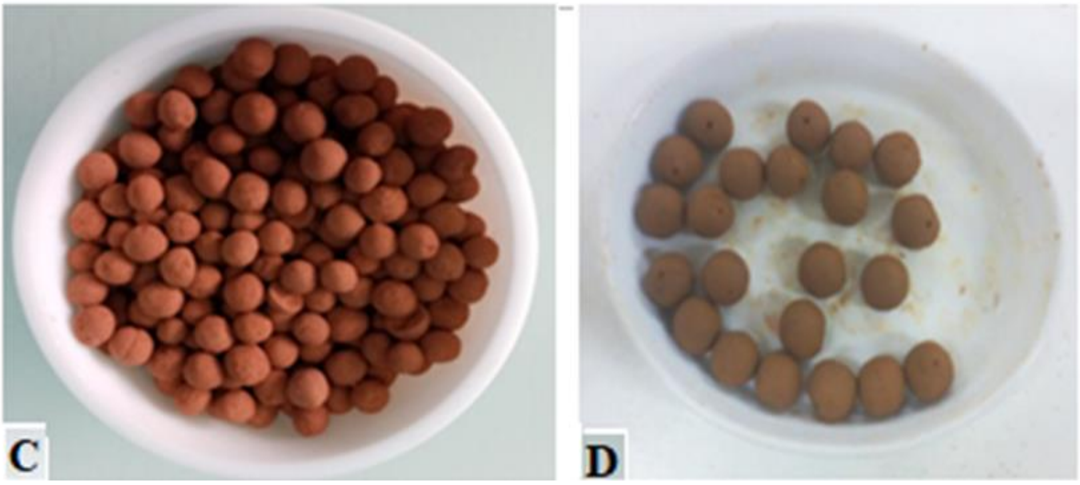


Figure 1 : Steps in the manufacture of calcined clay

Clay balls coated with TiO_2 : The clay balls were soaked in an ethanolic solution of 20 $g.L^{-1}$ of TiO_2 for three days. Then, the beads are calcined in the oven at 550 °C for 2 hours on a temperature ramp of 10 °C.min-1. This ramp promotes fixation of a large quantity of TiO_2. The beads were rinsed with ultra pure water to remove TiO_2 residues which had not been well fixed. This experiment was repeated three times. Figure 2 shows the clay beads coated with TiO_2.

Figure 2 : Clay balls coated with TiO_2

- **Adsorption tests of the beads in the dark** : Adsorption experiments were carried out in the dark both with the beads covered with TiO_2 and with those not covered with TiO_2. The duration of the test is 300 minutes.
- **Photolysis** : Under solar irradiation in the presence of beads not covered with TiO_2, samples are taken successively with Pasteur pipettes at regular time intervals during the irradiation (30 min ; 60 min ; 120 min ; 180 min ; 240 min ; 300 min) and analyzed by high performance liquid chromatograph.
- **Photocatalysis experimental device** : The experimental device is composed of four 50 ml test tubes corresponding to the different times of the experiments. These reactors were exposed to the sun as shown in Figure 3.

Figure 3 : Reactors arranged under solar radiation

- **Experimental protocol for photocatalytic tests :** The reactors containing water doped with Sulfamethoxazole solutions were placed under natural solar radiation. Samples are taken successively with pasteur pipettes at regular time intervals during irradiation (30 ; 60 ; 120 ; 180 ; 240 and 300 minutes) and analyzed by high-performance liquid chromatograph.

3. RESULTS AND DISCUSSION

3.1. Chromatograph calibration: From the surfaces obtained as a function of the concentrations of the sulfamethoxazole standards, the calibration curve shown in Figure 4 was plotted. The line obtained is a linear line with equation y = 34.83x. the correlation coefficient $R^2 = 0.99$. This value shows that there is a very good correlation between the concentrations of sulfamethoxazole standards and the peak areas obtained by liquid chromatography. This straight line made it possible to calculate the residual concentrations of sulfamethoxazole during the photocatalytic experiments.

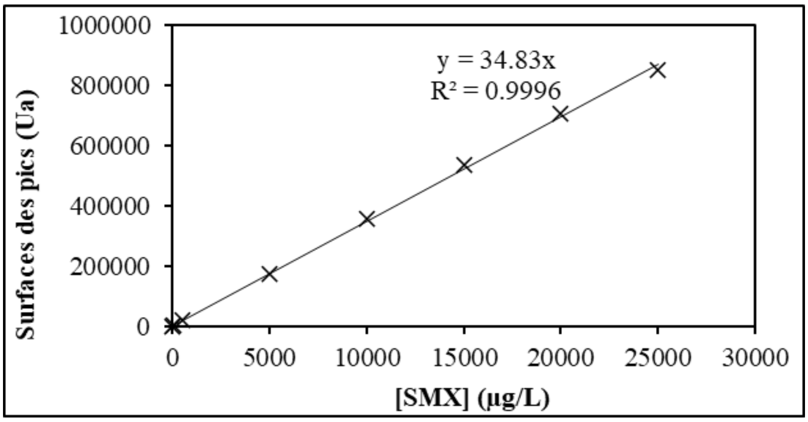


Figure 4 : Sulfamethoxazole calibration curve.

3.2. Sulfamethoxazole adsorption test in the dark : Absorption experiments were performed on beads without TiO_2 and beads coated with TiO_2. Figure 5 presents the adsorption kinetics of sulfamethoxazole under our experimental conditions. The two adsorption tests were carried out to evaluate the adsorption capacity of this antibiotic on the beads soaked in TiO_2 or not. The results indicated that sulfamethoxazole is not removed by adsorption tests. These results showed that sulfamethoxazole does not adsorb to the surface of clay materials.

Furthermore, solar photolysis experiments in the presence of beads not covered with TiO_2 also showed that sulfamethoxazole is only eliminated by this process to approximately 5.07%. This result would explain the presence of this sulfonamide in an aqueous environment, especially since it is very stable in environmental matrices. This low elimination rate was due to the fact that the irradiation of the experiment was at a wavelength of 360 nm while the absorption spectrum of SMX is mainly in the range of 240 -310 nm [9] [10].

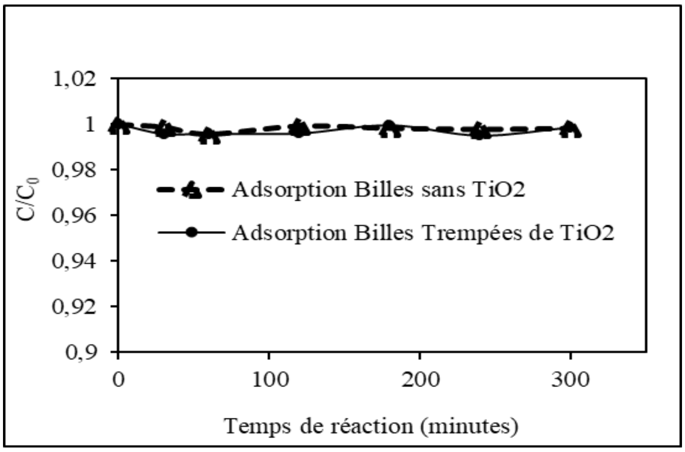


Figure 5 : Adsorption tests in the presence of simple clay and soaked with TiO_2, $V = 50$ ml, $C_o = 500$ µg/L, $T = 25$ °C, pH = 6

3.3. Solar photolysis of sulfamethoxazole : The kinetics of degradation of sulfamethoxazole by direct solar photolysis is illustrated in Figure 6. The results obtained during photolysis showed a slight slope. This slope reflects a degradation of sulfamethoxazole. The degradation rate of sulfamethoxazole was 5.07%.

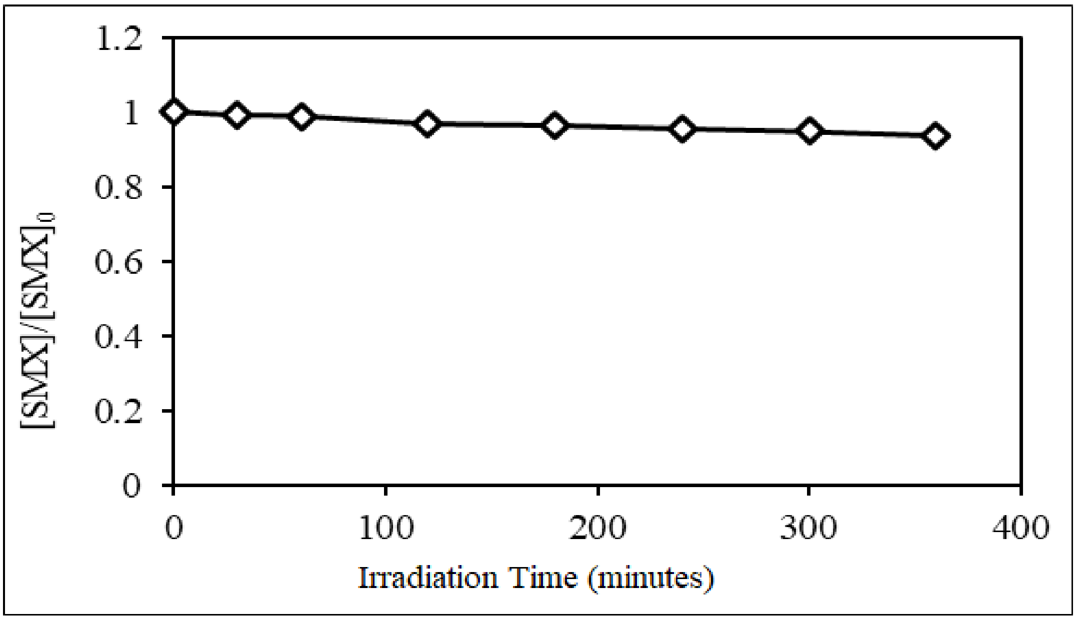


Figure 6 : Kinetics of SMX degradation under solar irradiation with clay beads without TiO_2, pH = 5.4 ; [SMX] = 5 mg/L ; mass of clay balls without TiO_2 = 60 g.

3.4. Solar photocatalysis of sulfamethoxazole : Figure 7 presents the degradation kinetics of sulfamethoxazole by solar photocatalysis. There is a break in slope, consequently, a rapid decrease in the initial concentration of sulfamethoxazole. The results indicated a disappearance of sulfamethoxazole of 94.95% after 300 minutes.

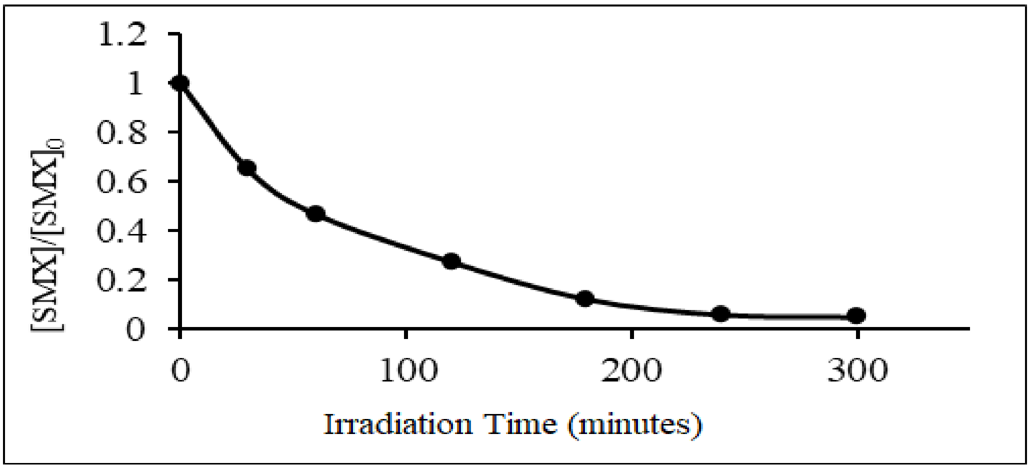


Figure 7 : Kinetics of SMX degradation under solar irradiation with TiO2 coated clay beads, [SMX] = 500 µg/L, pH = 5.4; Ball-TiO_2 mass = 50 g.

Photocatalytic activity of TiO_2 immobilized on the clay beads. This photocatalytic activity would result in the presence of hydroxyl radicals generated in the presence of sunlight. Since the photocatalysis reaction occurs on the photon-activated TiO_2 surface, understanding the reaction steps involving photodegradation of organic materials is indispensable in formulating the kinetic expression for heterogeneous photocatalysis [11] [12].

3.5. Comparative study of the kinetics of solar photolysis and solar photocatalysis : Figure 8 represents the degradation kinetics obtained by solar photolysis using clay on the one hand and on the other hand by the UV/TiO_2 system using clay (photocatalysis) of sulfamethoxazole, under solar irradiation.

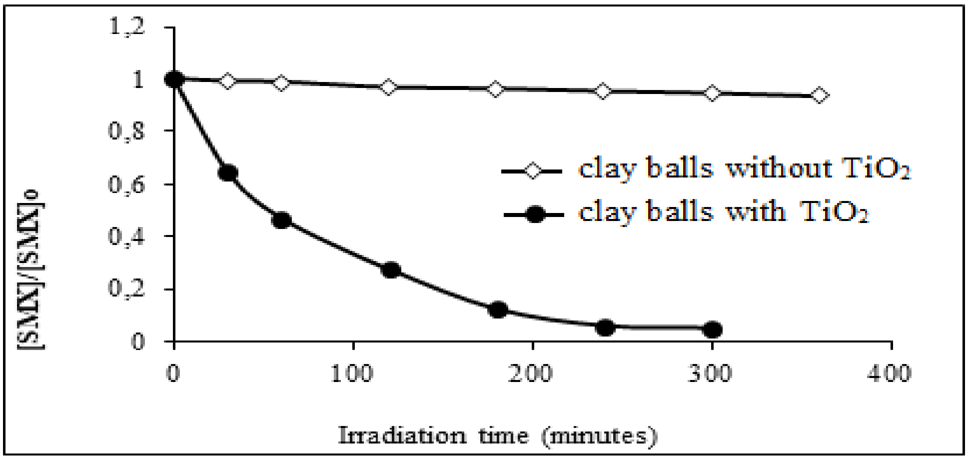


Figure 8 : Degradation kinetics by solar photolysis (using clay) and by the UV/TiO_2 system (photocatalysis) of sulfamethoxazole (using clay).

Kinetic parameters, such as apparent first-order rate constants (k) and half-life times ($t_{\frac{1}{2}}$), have been reported in Table II. The results showed that the straight line equations ln ($\frac{C_t}{C_0}$) = kt , are represented by linear lines with a correlation coefficient R² which is of the order of 0.9.

Tableau II : Kinetic parameters of sulfamethoxazole degradation by solar photolysis and photocatalysis

Experiments carried out	**Degradation rate (%)**	**Correlation coefficient (R^2)**	**Speed constants k_{app} (min^{-1})**	**Half-life time $t_{1/2}$ (min)**
Solar photolysis (balls without TiO_2)	5.07	0.97	2.10^{-4}	3465.73
Solar photocatalysis (beads with TiO_2)	94.95	0.98	$1.1.10^{-2}$	63.59

3.6. Influence of operating parameters

3.6.1. Influence of the initial concentration of the pollutant : The effect of initial concentration was studied for sulfamethoxazole concentrations of 500 ; 1000 and 2000 µg/L. The degradation kinetics were presented in Figure 9. The results showed higher degradation rates of 90.95% in our working conditions after 5 hours of solar irradiation respectively for 500 ; 1000 and 2000 µg/L of sulfamethoxazole. The rate of degradation decreases as the concentration of sulfamethoxazole increases

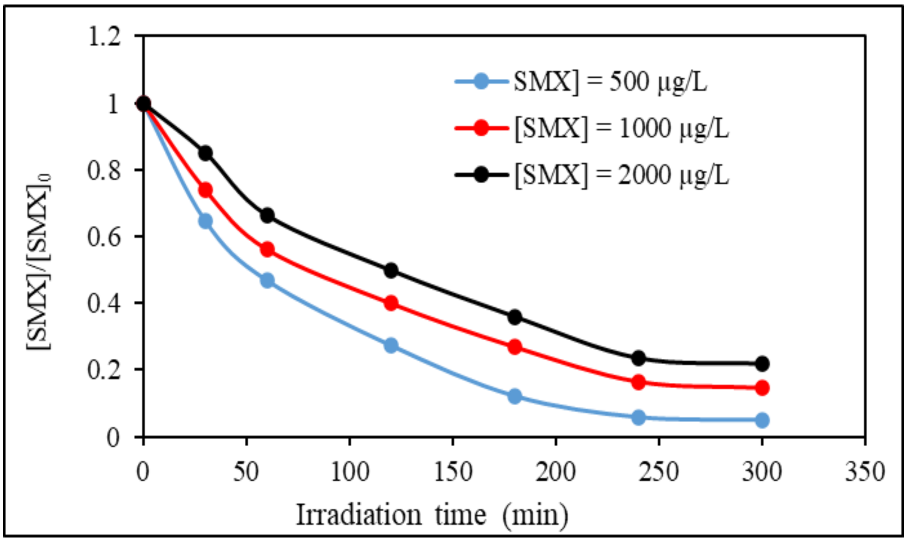


Figure 9 : Influence of the initial concentration on the kinetics of solar photocatalysis of sulfamethoxazole, pH = 5.4; Ball-TiO2 mass = 50 g.

Varying the initial concentration of Sulfamethoxazole showed an increase in the degradation rate at 500 µg. L^{-1}. The degradation rate obtained is 94.95%. Beyond this concentration, the results indicated a slight decrease in the degradation rate of 85.30% and 78.23% respectively for 1000 and 2000 µg. L^{-1}. This reduction in the degradation rate could be explained by saturation of the active sites of TiO_2 with Sulfamethoxazole.

Indeed, Zhou *and al.* [13] conducted photodegradation experiments with a variable initial concentration of sulfamethoxazole (ranging from 1 mg. L^{-1} to 8 mg. L^{-1}). They reported that as the concentration of SMX increased, the degradation rate decreased. This could be explained by the fact that at a higher concentration of sulfamethoxazole, the available light becomes limited [14]. This means that with the same amount of light, the amount of sulfamethoxazole molecules entering the excited state decreases, resulting in a lower rate of photodegradation.

Figure 10 expresses the variation of ln (Ct/Co) = kt as a function of irradiation time. The curve is a straight line with correlation coefficients greater than 0.98. The constant rate of degradation of sulfamethoxazole gradually decreases from Kapp = $1.1.10^{-2}$ min^{-1} to $5.5.10^{-3}$ min^{-1}, i.e. towards significantly lower values, when the initial concentration increases respectively by 500 to 2000 µg/L

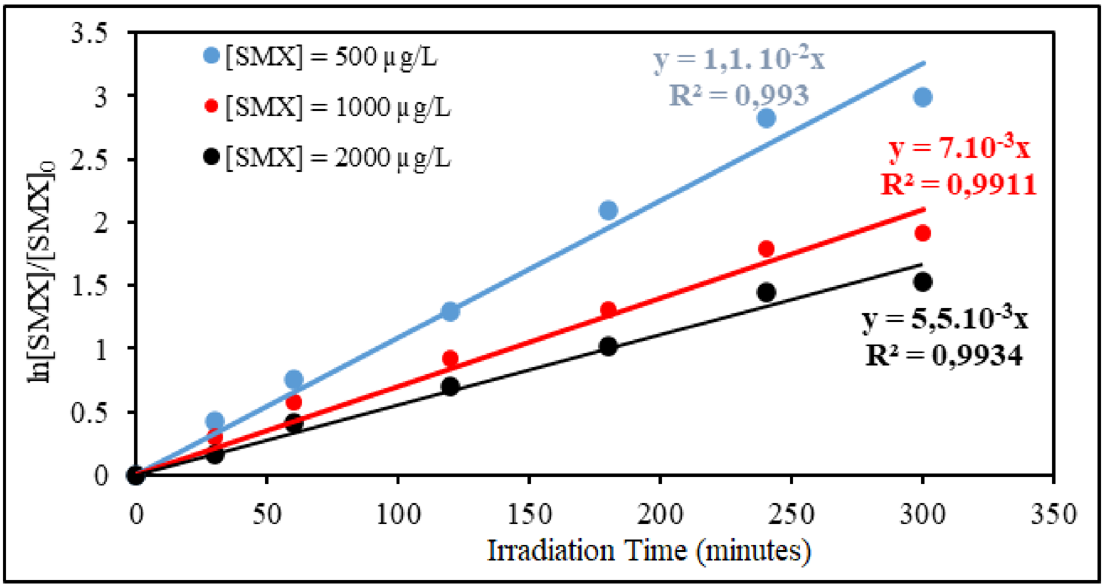


Figure 10 : Rate lines of sulfamethoxazole degradation : Effect of sulfamethoxazole concentration, pH = 5.4, mass of TiO_2 - beads = 50 g.

Les constantes cinétiques apparentes (k_{app}), les temps de demi-vie ($t_{1/2}$) et les coefficients de corrélation(R^2) sont mentionnés dans le tableau III. Les temps de demi-vie de cet antibiotique sont de 63,60 ; 99,02 ; 126,02 minutes respectivement pour 500 ;1000 et 2000 µg. L^{-1} ces résultats montrent les temps au bout desquels les concentrations de sulfamethoxazole ont été réduite de 50 % au cours de la durée des expériences. Plus précisément, il faut remarquer que la concentration de 500 µg. L^{-1} permet une rapide dégradation du sulfamethoxazole dans ces conditions expérimentales.

Tableau III : Kinetic parameters of sulfamethoxazole degradation by solar photocatalysis for different concentrations.

[SMX] µg/L	**degradation (%)**	**Correlation coefficient R^2**	**Kinetic constant $k_{app}(min^{-1})$**	**$t_{1/2}$ (min)**
500	95.01	0.993	$1.09.10^{-2}$	63.59
1000	85.3	0.99	7.10^{-3}	99.02
2000	78.23	0.99	$5.5.10^{-3}$	126.02

3.6.2. Influence of pH : Figure 11 illustrates the degradation kinetics of SMX as a function of pH. The results showed that the degradation kinetics are rapid at pH = 5.4, under slightly acidic conditions. But, under very acidic conditions, pH = 3 and very basic, pH = 10, the degradation rate decreases. The degradation rates were 87.56% ; 95.00% ; 91.87% and 74% respectively for pH values = 3 ; 5.4 ; 8 and 10.

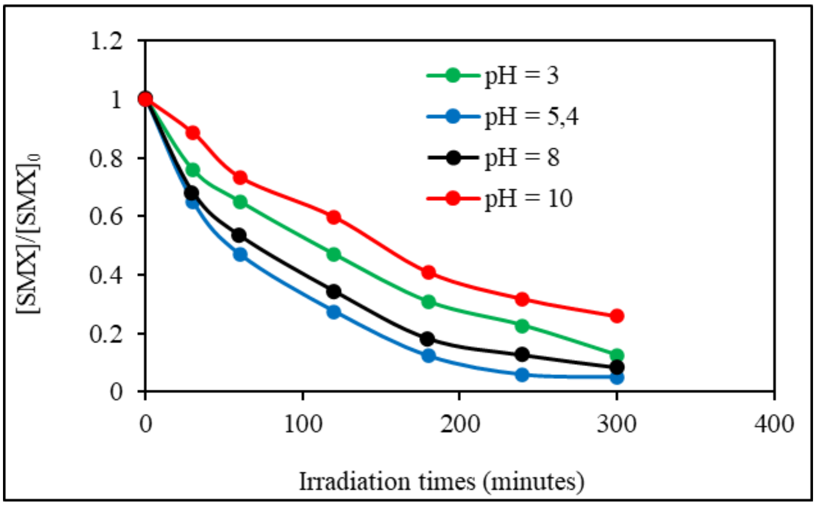


Figure 11 : Influence of pH on the kinetics of solar photocatalysis of sulfamethoxazole ; mass of TiO_2 balls = 50 g

Figure 12 shows the right-hand equations of the effect of pH on the degradation kinetics of sulfamethoxazole. The speed constants are $4.7.10^{-3}$ min^{-1} ; $6.6.10^{-3}$ min^{-1} ; $8.8.10^{-3}$ min^{-1} and $1.1.10^{-2}$ min$^{-1.}$ The rate of degradation decreases in very acidic and basic environments.

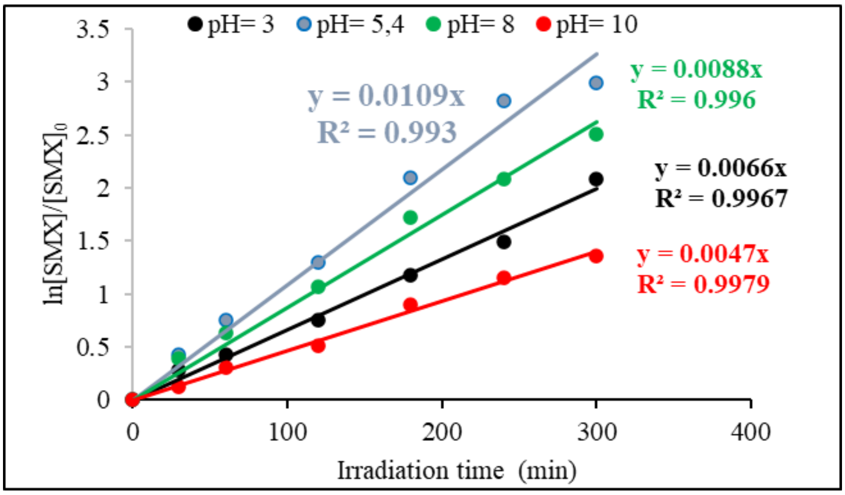


Figure 12 : Rate line of sulfamethoxazole degradation kinetics under solar irradiation.

The apparent kinetic constants (Kapp), half-life times (t1/2) and correlation coefficients (R^2) are mentioned in Table IV. The effect of pH made it possible to show that the effectiveness of this process occurs at pH 5.4 and pH 8. The rate constants were $1.1.10^{-2}$ min^{-1} and $8.8.10^{-3}$ min$^{. -1}$. The result indicates that the elimination of SMX is more favorable under slightly acidic conditions than under neutral or alkaline and highly acidic conditions. This phenomenon could be explained by the surface electric charge of the photocatalyst and the pH-dependent speciation of sulfamethoxazole. The main component of the catalyst is an anatase form whose point of zero charge (PZC) value was 6.1 [15].

If the pH was lower than the PZC point, the surface of the material would be positively charged, and on the other hand, the surface charge would be negative if the pH was higher than the PZC point. Additionally, sulfamethoxazole has two pKas which were 1.85 and 5.6 [16][17]. Sulfamethoxazole exists mainly in neutral form in the pH range from pKa1 to pKa2, mainly in protonated form at pH below pKa1 and mainly in negative charge form at pH above pKa2 [16][18]. Therefore, this alloues the adsorption of SMX on the catalyst surface and promoted the contact between the sulfamethoxazole molecule and the OH• radical. In contrast, in more acidic/alkaline media, the sulfamethoxazole form and the catalyst have the same charge, which reduced the adsorption of sulfamethoxazole onto the catalyst due to the repulsive force. As a result, the adsorption on the catalyst surface was reduced, which led to the reduction of degradation efficiency. Previous studies reported that the negative charge form was the most stable and had the lowest photochemical reactivity [19]. This could explain why sulfamethoxazole at a more alkaline pH has a lower degradation efficiency than under acidic conditions. In addition, more acidic or alkaline solutions are favorable to the formation of OH• radicals on the surface of TiO_2 [20]. The results showed good agreement with previous studies [10][21].

Table IV: Kinetic parameters for the degradation of SMX by solar photocatalysis for different pH values.

pH	**Percentage of degradation (%)**	**Correlation coefficient R^2**	**Apparent kinetic constant k_{app}(min^{-1})**	**$t_{1/2}$ (min)**
3	87.56	0.99	$6.6.10^{-3}$	105.022
5.4	94.95	0.98	$1.1.10^{-2}$	63.60
8	91.87	0.95	$8.80.10^{-3}$	78.76
10	74.21	0.99	$0.00470.10^{-3}$	147.50

CONCLUSION

Sulfamethoxazole can be efficiently degraded photocatalytically under solar irradiation. Indeed, the comparative study between direct solar photolysis, with clay beads, and solar photocatalysis in the presence of TiO_2 supported by clay beads was studied. The results showed a clear difference in degradation rates which are 5.07% with the clay balls and 94.95% in the presence of TiO_2 supported by the clay balls.

In order to determine the optimal conditions of the degradation process, the study of the influence of certain parameters such as the initial concentration of sulfamethoxazole and the initial pH of the reaction medium was carried out. The results showed that for a pH = 5.4 and a sulfamethoxazole concentration equal to 500 µg/L, the maximum degradation rate obtained was 94.95%.

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