Monitoring the removal of a mixture of emerging pharmaceutical contaminants assisted by a fixed TiO_2 support in a photocatalytic process

Andres Perez-Gonzalez[1], Veronica Pinos-Velez[2], and Piercosimo Tripaldi[3]

***Abstract* — This study evaluated the efficacy of removing estradiol, sulfamethoxazole, and paracetamol from water using titanium dioxide (TiO_2). A layer of TiO_2 was fixed onto ceramic tiles and exposed to a solution of the pharmaceuticals under 254 nm ultraviolet light. The TiO_2 exhibited a surface area of 2.78 m^2/g with Methylene Blue, which favors the adsorption and subsequent degradation of the pharmaceuticals. After 60 minutes of exposure, the removal of estradiol was 69.43%, paracetamol 50.84%, and sulfamethoxazole 24.80%. Additionally, it was determined that the degradation kinetics of each drug varied, with some fitting first-order models (estradiol and paracetamol) and others fitting second-order models (sulfamethoxazole). The time in minutes required for the drug concentration to decrease to one-tenth of its original concentration was 107 for estradiol, 190 minutes for paracetamol, and 244 for sulfamethoxazole. Photocatalysis with TiO_2 immobilized on ceramics can be used to treat water contaminated with mixtures of pharmaceuticals without observing a restrictive effect between the drugs.**

***Keywords:* photocatalysis; Titanium dioxide; emerging pharmaceutical contaminants.**

***Resumen* — Este estudio evaluó la eficacia de la remoción de estradiol, sulfametoxazol y paracetamol del agua utilizando dióxido de titanio (TiO_2). Para ello se fijó una capa de TiO_2 sobre baldosas cerámicas y se expuso a una solución de los fármacos bajo luz ultravioleta de 254 nm. El TiO2 presentó un área superficial de 2.78 m^2/g con Azul de Metileno lo que favorece a la adsorción y posterior degradación de los fármacos. La foto remoción después de 60 minutos de exposición del estradiol fue del 69.43%, del paracetamol 50.84% y del sulfametaxazol 24.80%. Además, se determinó que la cinética de degradación de cada fármaco variaba, ajustándose algunos a modelos de primer orden (estradiol y paracetamol) y otro a segundo orden (sulfametaxazol). El tiempo en minutos necesario para bajar al 10% la concentración original de los fármacos fue de 107 para el estradiol, 190 minutos para el paracetamol y 244 para el sulfametaxazol. La fotocatálisis con TiO_2 inmobilizado en cerámicas puede ser utilizada para el tratamiento de aguas contaminadas con mezclas de fármacos sin que se observe un efecto restrictivo entre los fármacos.**

***Palabras Clave:* fotocatálisis; Titanium dioxide; emerging pharmaceutical contaminants.**

I. INTRODUCTION

THE presence of pharmaceuticals in wastewater can cause toxicity in aquatic organisms due to their ability to bioaccumulate and the adverse effects they can produce. The bioaccumulation capacity of these compounds can be manifested in the food chain, resulting in dangerous concentrations of pharmaceuticals in food due to biomagnification, affecting not only aquatic organisms but also predators, including humans [1], [2], [3]. Among the different pharmaceuticals, those of greatest interest due to the associated risks are endocrine disruptors and antibiotics.

Continuous exposure to low concentrations of pharmaceuticals or other endocrine disruptors through the consumption of contaminated water or aquatic products can have long-term effects on human health, such as alterations to the endocrine system and other hormonal problems. One compound that acts as an endocrine disruptor and is present in various pharmaceuticals is estradiol. Estradiol is a steroid hormone that, as an endocrine disruptor, can interfere with the hormonal systems of aquatic organisms, causing effects such as feminization of males, reduced fertility, and alterations in reproductive behaviors. Additionally, estradiol is persistent in the environment and can bioaccumulate, leading to higher concentrations in the food chain and eventually affecting humans, increasing the risk of hormone-dependent cancers and other health problems. Evaluating the presence of estradiol in wastewater is necessary to make decisions to mitigate risks to ecosystems and public health, since exposure through contaminated drinking water can have negative effects, especially in vulnerable populations [4], [5], [6].

The presence of antibiotics can contribute to the development and spread of antibiotic-resistant bacteria, complicating the treatment of infections and representing a serious public

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health problem. One of the most commonly used antibiotics is sulfamethoxazole, often used in combination with trimethoprim, and has a wide range of applications for different types of infections [7], [8]. This compound is resistant to degradation in conventional wastewater treatment plants, allowing its release into the aquatic environment. The presence of sulfamethoxazole in water can cause toxicity in aquatic organisms, including sublethal effects on the growth and reproduction of fish, invertebrates, and algae. Additionally, the bioaccumulation of this antibiotic can lead to harmful concentrations in the food chain, affecting top predators, including humans. The continuous presence of this compound in the environment can contribute to the proliferation of resistant bacteria [9], [10].

Another emerging contaminant of concern is paracetamol, a widely used analgesic and antipyretic, being one of the most used worldwide. Conventional wastewater treatment systems do not always completely remove paracetamol, allowing its release into surface waters. Although paracetamol can degrade relatively quickly under aerobic conditions, it can persist in the environment and its degradation byproducts can be toxic to aquatic organisms [11], [12], [13].

Therefore, it is important to remove these compounds from water. For their treatment, the mechanisms must be considered, as some pharmaceuticals can generate toxic metabolites during their degradation, further complicating the remediation of contaminated waters [14]. Photocatalysis over titanium dioxide (TiO_2) is a technique that has been widely investigated in recent decades due to its effectiveness in the degradation of organic compounds, which can include pharmaceuticals. Studies have shown that photocatalysis can efficiently decompose pharmaceuticals such as ciprofloxacin, sulfamethoxazole, and tetracycline, which are resistant to degradation in conventional wastewater treatment plants. Additionally, photocatalysis has shown effectiveness in the removal of other common pharmaceutical compounds, such as paracetamol and ibuprofen, as well as in the degradation of endocrine disruptors such as bisphenol A and nonylphenol, which can cause adverse effects on health and the environment [15], [16], [17].

This technique is generally carried out in aqueous suspensions of TiO_2, with the most efficient form being the use of titanium dioxide in the form of nanoparticles [18], [19]. The photocatalysis mechanism can be seen in Fig. 1, and is based on the stimulation or excitation of the TiO_2 semiconductor by UV or visible light, whether of natural or artificial origin. The process begins when TiO_2 absorbs photons with energy equal to or greater than its bandgap (3.2 eV for anatase, 3.0 eV for rutile, and 2.8 eV for brookite); this absorption promotes an electron from the valence band to the conduction band, creating an electron-hole pair (e-/h+). These pairs in the catalyst participate in redox reactions, see Figure 1. The holes (h^+) can oxidize water molecules or hydroxyl ions to form hydroxyl radicals ($^{\bullet}OH$), which are highly reactive species and capable of oxidizing organic contaminants to their complete mineralization, i.e., converting them to CO_2 and H_2O, see Eq. 1 and Eq. 2.

$$h^+ + H_2O \rightarrow \cdot OH + H^+ \tag{1}$$

$$h^+ + OH^- \rightarrow \cdot OH \tag{2}$$

On the other hand, electrons (e-) can reduce dissolved oxygen in water to form superoxide radicals ($O_2^{\cdot -}$) which also participate in the degradation of contaminants [20]. This can be seen in equations Eq. 3, Eq. 4 and Eq. 5

$$e^- + O_2 \rightarrow O_2 \cdot^- \tag{3}$$

$$O_2 \cdot^- + H^+ + \rightarrow HO_2 \cdot \tag{4}$$

$$2HO_2 \cdot \rightarrow H_2O_2 + O_2 \tag{5}$$

In most TiO_2 catalyzed photocatalytic studies, the focus has been on determining the optimal operating conditions to achieve the best performance in terms of degradation and mineralization [8], [21]. The operational parameters frequently investigated include photocatalyst loading, initial concentration of the pharmaceutical under investigation, type of photocatalyst, pH of the solution, wavelength, and light intensity [22], [23], [24].

One of the main drawbacks is the difficulty in recovering the catalyst; however, there is little information on studies to immobilize the catalyst and determine its effectiveness. Titanium dioxide can be anchored to a glass or ceramic surface to increase its stability and durability, which promotes a uniform distribution of TiO_2 particles, increasing the stability of the catalyst and allowing it to be reused several times without significant loss of catalytic activity. The ability to fix TiO_2 allows for the optimization of its surface area, which favors greater photocatalytic activity and also reduces secondary contamination that may occur due to the presence of free TiO_2 nanoparticles, which in some cases can have undesirable effects on the environment and human health. Surfaces with anchored TiO_2 can be used to purify water and air, especially organic contaminants, emerging contaminants, volatile organic compounds (VOCs), bacteria, and other toxic substances that may be present in these media. Based on this evidence, so-called self-cleaning surfaces have been developed, which, when exposed to light, can degrade organic contaminants and microorganisms [25].

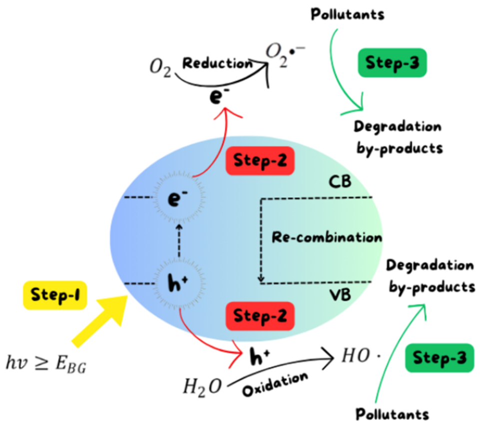

Fig. 1. Photocatalytic process the formation of photoinduced charge carriers (e^-/h^+) on the surfaces of semiconductor particles of TiO_2.

TiO_2-based photocatalytic processes have proven to be highly effective, making it necessary to leverage this knowledge to easily implement and low-cost methodologies to address the growing problem of pharmaceutical residues in small and medium-sized cities. The objective of this work was to evaluate the photodegradation of estradiol, sulfamethoxazole, and paracetamol on a TiO_2-coated glazed ceramic surface as a tertiary wastewater treatment that could be used in small cities or for the treatment of hospital wastewater.

II. MATERIALS AND METHODS

A. TiO_2 Photocatalytic Treatment System

White commercial tiles with a glazed surface were used. A uniform layer of TiO_2 (rutile) paste was spread over these tiles, followed by calcination (900 °C for 4 hours) to ensure proper fixation of the TiO_2 on the substrate. Food-grade TiO_2 (rutile) (supplier: Freire Mejía) was used because it is the most commonly available form and is cost-effective ($TiO_2 \geq 94\%$, rutile $\geq 98\%$, particle size ≥ 45 µm, max. 0.05%). A 23 W UV lamp emitting 254 nm light served as the radiation source. The distance between the radiation source and the reaction surface was set at 20 cm, as shown in Figure 2, which displays the experimental setup.

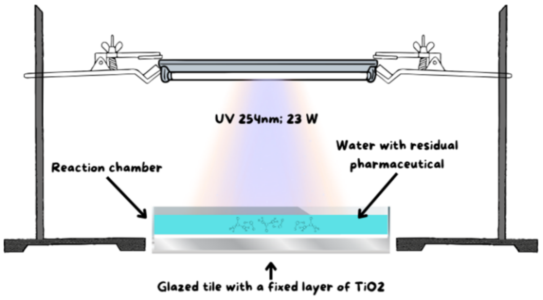

Fig. 2. Representation of the apparatus used for photodegradation

B. Removal tests

An aqueous solution was prepared, consisting of a mixture of three pharmaceuticals at different concentrations: 500 ppb estradiol (Sigma Aldrich), 10 ppm sulfamethoxazole (Sigma Aldrich), and 10 ppm acetaminophen (Sigma Aldrich). For the assay, 50 mL of the solution was placed and exposed to 254 nm UV radiation in the setup shown in Figure 2. Every 10 minutes, 0.5 mL samples were taken for a total of 80 minutes. The treated samples were analyzed by high-performance liquid chromatography (HPLC). The equipment used was an HPLC (Thermo Ultimate 3000) equipped with a 20 cm C18 column, with a detector set at 254 nm for estradiol and 272 nm for sulfamethoxazole and acetaminophen. The mobile phase was a mixture of water (53%), methanol (24%, Fisher chemical, ACS), and acetonitrile (23%, Supelco-LiChrosolv) of HPLC grade. Isocratic conditions were employed, and the column temperature was maintained at 25 °C with a flow rate of 0.9 mL/min. Removal was determined using Eq. 6, where C_{A0} is the initial concentration of the contaminant and C_A is the concentration at time t.

$$\%R = \frac{C_{A0} - C_A}{C_{A0}} \times 100 \tag{6}$$

B. Methodology for determining the surface area of Methylene Blue and BET

The adsorption area of titanium dioxide (TiO_2) was determined using a 5.155 ppm solution of Methylene Blue (MB) dissolved in deionized water. The adsorption process was carried out in a batch system with varying amounts of TiO_2. Subsequently, the concentration of unadsorbed methylene blue was quantified using UV-Vis spectrophotometry at a wavelength of 660 nm, employing a Thermo Scientific Evolution 60 UV-Vis spectrophotometer.

The surface area was determined by N_2 adsorption using the Brunauer, Emmett, and Teller (BET) theory at 77.6 K using a Micromeritics Autochem 2920 instrument. Each sample was degassed at 373 K for 720 minutes under a vacuum of 10^{-4} Pa.

C. TiO_2 Photocatalytic Treatment System

The kinetic calculations were performed using the Langmuir-Hinshelwood (L-H) kinetic model. This model assumes that the rate-determining step (RDS) of the photocatalytic reaction involves the presence of the reactant as a monolayer at the photocatalyst/solution interface. The Eq. 7 for this model is:

$$r_0 = \frac{(k_C K[C_0])}{(1 + K[C_0])} = k_{obs}[C_0]^n \tag{7}$$

Where:

r0 is the initial rate of photo-oxidation of the solute.
kc is the rate constant of the photo-oxidation ste==p.
K is the stability constant of the drug-TiO_2 equilibrium.
n is the order of the kinetics.

For n=0, the kinetics follow a zero-order rate law, and the Langmuir-Hinshelwood (L-H) model simplifies to a simpler expression (Eq. 8).

$$r_0 = k_{obs} \tag{8}$$

In this work, both constants (kc and K) were obtained by regression analysis of initial rates and corresponding initial drug concentrations. Calculations were performed using the Solver add-in in Microsoft Excel.

III. RESULTS AND DISCUSSION

A. Measurement of the specific surface area of TiO_2

The experimental data for the adsorption isotherm calculations are summarized in Table 1. Co represents the initial Methylene Blue concentration, Ce the equilibrium concentration, and qe the amount adsorbed per unit mass of TiO_2. Methylene blue (MB)

($C_{16}H_{18}ClN_3S$) is an organic compound widely used in the determination of the specific surface area of clays, activated carbon, and other types of adsorbents. In solution, this dye ionizes, forming a cation that is electrostatically attracted to negatively charged surfaces. By measuring the amount of MB adsorbed, it is possible to calculate the total exposed area of the adsorbent particles. The adsorption of MB onto titanium dioxide (TiO_2) fits the Langmuir isotherm model, characteristic of a monolayer coverage [26]. The Langmuir nature of the adsorption process is evident from the isotherm plot in Figure 3.

TABLE I
VALUES FOR THE METHYLENE BLUE
ADSORPTION ISOTHERM CALCULATIONS ON TIO_2

Masa TiO_2 (g)	Co mg/L	Ce mg/L	qe mg/mg
0.0095	5.155	3.052	0.0221
0.0439	5.155	2.191	0.0067
0.1408	5.155	1.518	0.0025
0.6124	5.155	0.160	0.0008

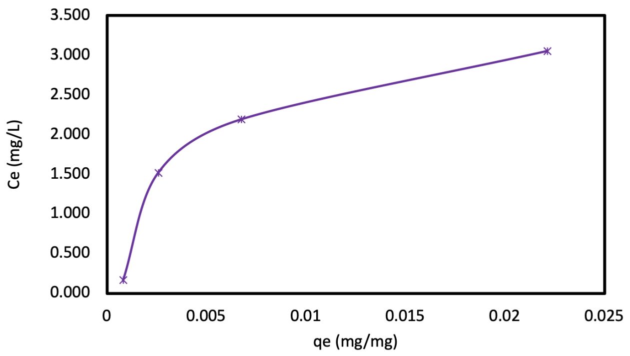

Fig. 3. Methylene Blue adsorption isotherm on TiO_2

From the Langmuir isotherm results, it was determined that the TiO_2 used had a surface area of 2.78 m²/mg, as measured by Methylene Blue.

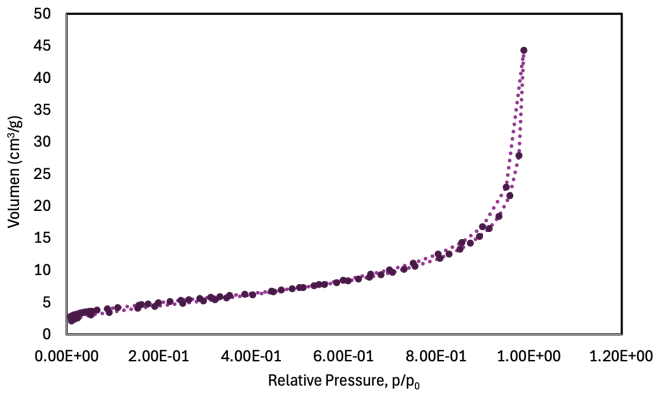

Fig. 4. BET isotherm with N_2 of TiO_2

Figure 4 shows a typical type IV isotherm characteristic of mesoporous solids. A pore volume of 0.033 cm³/g, a 12 m²/g surface area, and a measured pore width of 14.48 nm were obtained.

The N_2 BET isotherm (Figure 4) results of the TiO_2 correspond to a type IV curve characteristic of mesoporous materials, indicating the presence of intermediate-sized pores with a hysteresis H4. The pore volume of 0.033 cm³/g suggests moderate porosity, confirming the highly porous nature of TiO_2. The average pore width of 14.48 nm indicates that these pores are large enough to allow the diffusion of considerably sized molecules. These values characterize TiO_2 as a mesoporous solid with high adsorption capacity and excellent catalytic activity.

The surface area results obtained by the BET method, and the surface area measured by the MB method show a high correlation. It is important to consider that the MB method, based on the adsorption of large molecules such as methylene blue, may underestimate the total surface area, especially in microporous materials, due to the limitations of access of these molecules to the smallest pores. In this case, the correlation between both methods suggests that most of the accessible surface area is composed of mesopores, large enough to allow the adsorption of methylene blue molecules. This characteristic is of great relevance for applications involving the adsorption of considerably sized organic molecules, since the extensive mesoporous surface provides numerous accessible adsorption sites for these molecules [27].

B. Removal of pharmaceuticals

The concentration of the pharmaceuticals used in the mixture was measured using HPLC. The results of the chromatograms obtained from the measurements over time have been compiled and are presented in Figure 5.

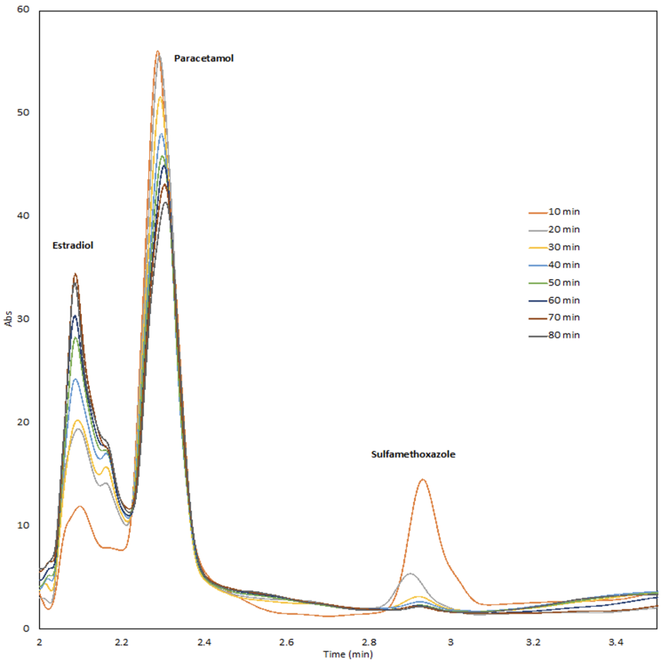

Fig. 5. Drug degradation measurement chromatograms

Figure 5 shows a change in the analyte concentration over time due to exposure to 254 nm UV radiation. The results of applying the integral method to the concentrations are shown in Figure 6. Estradiol (k = 0.0218 1/min) and paracetamol (k = 0.046 1/min) showed a better fit to the first-order model, while sulfamethoxazole (k = 0.048 L/mg min) fit a second-order kinetic model.

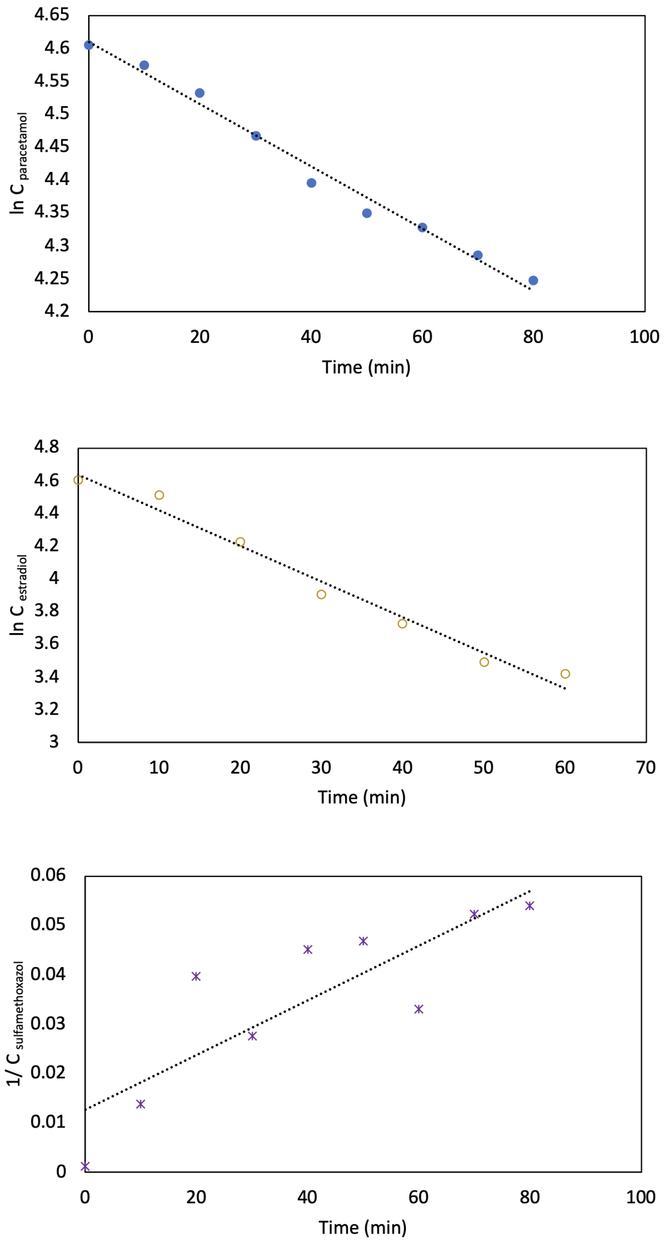

Fig. 6. The concentration changes of the pharmaceuticals due to the removal process and the relationship needed to determine their kinetic type: (a) Estradiol fitted to the first-order integral model; (b) Paracetamol fit to the first-order model; (c) Sulfamethoxazole fitted to a second-order integral kinetic model.

The rate constants, kc and K, for the photo-oxidation reactions were obtained by non-linear GRG optimization using Solver. The optimized values, subject to the constraints of positive kc and K > 1, are summarized in Table 2.

TABLE II
K AND KC RATE CONSTANTS OF THE PHOTO-OXIDATION EQUATION AND TIME TO REDUCE DRUG CONCENTRATION TO ONE-TENTH OF ITS INITIAL VALUE

Pharmaceuticals	**K (L/mg)**	**kc (mg/L min)**	**t (c/10 min)**
Estradiol	4.13	0.056	107
Paracetamol	1.15	0.97	190
Sulfametoxazol	1.2	0.010	244

The results demonstrate a clear correlation between the reaction rate constant (kc) and the time required to reduce drug concentration by 90% (t(C/10)). Higher kc values indicate faster degradation rates. Pharmaceuticals adsorbed on TiO_2 are expected to be more mobile, enhancing their interaction with hydroxyl radicals. This mobility can be influenced by drug chemistry, drug-surface interactions, and TiO_2 surface roughness [28].

The experimental data obtained corroborate the efficacy of the proposed method for the degradation of the studied pharmaceuticals. The observed reduction percentages after 60 minutes of irradiation are significant, especially in the case of estradiol (69.43%), although for the other pharmaceuticals the removal was also significant, with paracetamol (50.84%) and sulfamethoxazole (24.80%), indicating a high potential of this technology for the treatment of wastewater contaminated with pharmaceutical compounds [29]. However, it is important to note that the efficiency of degradation can vary depending on various factors, such as the initial concentration of the pharmaceuticals, the intensity of the radiation, and the presence of other compounds in the water. To optimize photocatalytic processes and expand their application to a variety of contaminants, more detailed studies are needed on the reaction mechanisms, the influence of operational variables, and the combination of photocatalysis with other treatments [28].

IV. CONCLUSIONS

The kinetic constant values presented in Table 2 offer a simplified view of the photo-oxidation of a drug mixture. The Langmuir-Hinshelwood model used, while useful for understanding fundamental processes, does not capture the complexity of real systems, where the presence of multiple compounds and the formation of degradation products can significantly alter the reaction kinetics. Although the obtained results provide an initial estimate of degradation rates, it is essential to recognize that to accurately describe these systems and optimize wastewater treatment processes, more sophisticated kinetic models are required that incorporate the dynamics of competitive adsorption and the formation of reaction products.

The degradation percentages obtained in this study are consistent with the results reported in previous research on heterogeneous photocatalysis using titanium dioxide under various experimental conditions [11], [17], [29]. This consistency suggests that the proposed method, based on the application of a titanium dioxide layer and exposure to UV radiation, demonstrates considerable robustness. Unlike other approaches that require complex operating conditions, the system allows for significant degradation levels to be achieved with a simplified configuration. This simplification reduces implementation complexity and cost, facilitating its application in diverse environments. The viability of a system that relies on accessible factors such as UV light and a basic catalytic surface opens new possibilities for decentralized wastewater treatment. The ability to obtain results comparable to other methods, without relying on additional reagents, underscores the potential of this technology as a practical and economical solution to address water contamination at various scales

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