ing-NOVA
INVESTIGACIÓN E INNOVACIÓN EN INGENIERÍA

RESEARCH MANUSCRIPT

Comparison of radiation collector systems in the degradation of Contaminants of Emerging Concern by heterogeneous photocatalysis with solar radiation

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ABSTRACT

Wastewater contaminated with Emerging Concern Contaminants (CEC) can originate from various sources, including industry, agriculture, urban areas, and hospitals. Among the Advanced Oxidation Processes (AOPs) used for water treatment with CEC, Heterogeneous Photocatalysis (HP) with TiO_2 has proven to be highly effective. In this research study, the degradation of Safranin T (SF), 2,4-dichlorophenoxyacetic acid (2,4-D), and Sulfacetamide (SAM) was investigated using solar-HP in a quartz wall reactor.

Initially, the best operating conditions were determined using a lamp emitting at a wavelength of 365 nm. Subsequently, under these favorable conditions (high flow and aeration), solar light was employed to degrade SF, SAM, and 2,4-D, with four different radiation collector systems: Flat Plate Collector (FPC), V Collector (VC), Parabolic Collector (PC), and Compound Parabolic Cylinder Collector (CPC). The aim was to achieve an equal value of accumulated energy (122.77 kJ m^{-2}) in the system for all configurations. The results showed that the Parabolic Collector (PC) exhibited the best performance in treating all three pollutants.

Finally, the researchers calculated the Collector Impact Ratio Factor (CIRF) for the pollutants, revealing a significant degradation rate of up to 12 times for SAM. These findings highlight the effectiveness of solar-HP, specifically with the Parabolic Collector (PC), in efficiently treating wastewater contaminated with Safranin T, 2,4-dichlorophenoxyacetic acid, and Sulfacetamide.

Keywords: 2,4-Dichlorophenoxyacetic acid; Advanced oxidation processes; Contaminants of emerging concern; Radiation collectors; Safranin; Sulfacetamide.

Para citaciones: F. Granda Ramírez, G. Hincapié Mejía, M. Barrera, S. Castrillón, L. Rueda & J. Pino Arango, "Comparison of radiation collector systems in the degradation of Contaminants of Emerging Concern by heterogeneous photocatalysis with solar radiation", *Ing-Nova*, vol. *2*, no. 2, pp. 146-158, Jul. 2023.
https://doi.org/10.32997/rin-2023-4346

Recibido: 14 de junio de 2023
Revisado: 11 de julio de 2023
Aprobado: 14 de julio de 2023

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Editor: Miguel Ángel Mueses. Universidad de Cartagena-Colombia.

Universidad Editorial Universitaria de Cartagena

Comparación de sistemas colectores de radiación en la degradación de Contaminantes de Preocupación Emergente por fotocatálisis heterogénea con radiación solar

RESUMEN

Las aguas residuales contaminadas con contaminantes de preocupación emergente (CPE) pueden provenir de varias fuentes, incluidas la industria, la agricultura, las áreas urbanas y los hospitales. Dentro de los Procesos de Oxidación Avanzada (PAOs) utilizados para el tratamiento de agua con CEC, la Fotocatálisis Heterogénea (FH) con TiO_2 ha demostrado ser altamente efectiva. En este estudio de investigación, se investigó la degradación de safranina T (SF), ácido 2,4-diclorofenoxiacético (2,4-D) y sulfacetamida (SAM) utilizando FC solar en un reactor de pared de cuarzo.

Inicialmente, las mejores condiciones de operación se determinaron usando una lámpara que emitía a una longitud de onda de 365 nm. Posteriormente, bajo estas condiciones favorables (alto flujo y aireación), se empleó luz solar para degradar SF, SAM y 2,4-D, con cuatro sistemas diferentes de colectores de radiación: Colector de placa plana (FPC), Colector en V (VC), Colector Parabólico (PC) y Colector Cilindro Parabólico Compuesto (CPC). El objetivo era conseguir un valor igual de energía acumulada (122,77 kJ m^{-2}) en el sistema para todas las configuraciones. Los resultados mostraron que el Colector Parabólico (PC) exhibió el mejor desempeño en el tratamiento de los tres contaminantes.

Finalmente, los investigadores calcularon el factor de relación de impacto del colector (CIRF) para los contaminantes, lo que reveló una tasa de degradación significativa de hasta 12 veces para SAM. Estos hallazgos destacan la efectividad de la energía solar-HP, específicamente con el Colector Parabólico (PC), en el tratamiento eficiente de aguas residuales contaminadas con Safranina T, ácido 2,4-diclorofenoxiacético y Sulfacetamida.

Palabras clave: Ácido 2,4-diclorofenoxiacético; Procesos de oxidación avanzados; Contaminantes de preocupación emergente; Colectores de radiación; Safranina; Sulfacetamida.

1. Introduction

Currently, there is a growing interest in Contaminants of Emerging Concern (CEC) due to their unnoticed presence and varying concentrations in the environment [1]. These CECs encompass a wide range of chemical compounds commonly used in everyday life, including medications, personal care products, soaps, surfactants, industrial additives, plasticizers, pesticides, and various other chemicals found in the environment [2]. Many of these pollutants are not effectively removed by conventional treatment systems, and in some cases, they are not regulated by environmental authorities. One such CEC is Safranin T (SF), a biological dye frequently used for tissue staining, detecting structures in eukaryotic and prokaryotic cells, and the popular Gram staining technique [3]. Another compound of concern is 2,4-Dichlorophenoxyacetic Acid, widely used in commercial herbicides, known for its detrimental effects on the environment, particularly in aquatic ecosystems and human health. Due to its persistence, it is not efficiently degraded by conventional wastewater treatment plants, necessitating the use of more effective degradation methods such as Advanced Oxidation Processes (AOPs) [4]. Sodic Sulfacetamide (SAM) is an ophthalmic medication used for treating eye

infections and is also administered orally for acne and seborrheic dermatitis. It is rapidly excreted in urine and can be found in soaps, shampoos, creams, and washing solutions [5].

AOPs are considered highly effective for the removal of numerous persistent contaminants, transforming organic matter into carbon dioxide and water through changes in chemical structure [5]. Heterogeneous Photocatalysis (HP) is one of the most commonly used AOPs, utilizing a semiconductor material, particularly TiO_2 (such as P-25 from Evonik®), which exhibits high photocatalytic activity, stability in aqueous solutions, non-toxicity, and cost-effectiveness [5]. In the photocatalytic process with TiO_2 under aerobic conditions, when exposed to ultraviolet light radiation, the semiconductor generates electron-hole pairs and Reactive Oxygen Species (ROS) such as hydroxyl radicals ($^{\bullet}OH$), superoxide anions ($^{\bullet}O^{2-}$), hyperoxide radicals ($^{\bullet}OH_2$), or Hydrogen Peroxide (H_2O_2) [6]. Proper positioning of the catalyst (either dispersed or supported) and efficient exposure to useful light are crucial in achieving an effective photocatalytic process. Radiation collector systems have been developed to enhance efficiency and reduce costs in photocatalytic decontamination and water treatment [7]. These systems employ reflective surfaces, with aluminum-based mirrors being among the best materials for directing and reflecting useful light towards the reactor, maximizing its utilization and minimizing losses [8].

The Heterogeneous Photocatalysis (HP) process with TiO_2 has also been implemented using collectors without radiation concentration, with the Compound Parabolic Cylinder (CPC) system being highly efficient and technologically advanced [9]. Utilizing sunlight as the radiation source, HP with TiO_2 and a CPC collector has proven to be an effective system for degrading a mixture of 15 CECs, achieving a degradation rate of 90% for the pollutants [10]. Commonly used collector designs include Parabolic Collectors (PC) (Figure 1), V Collectors (VC) (Figure 2), and Compound Parabolic Cylinders (CPC) (Figure 3), among others [11].

Figure 1. Parabolic Collector (PC).

Figure 2. V Collector (VC).

Figure 3. Compound Parabolic Cylinder Collector (CPC).

In recent years, the use of Advanced Oxidation Processes (AOPs) has been proposed to address the issue of Contaminants of Emerging Concern (CEC). In this research, a comparison was conducted among four configurations of radiation collectors: Flat Plate Collector (FPC), V Collector (VC), Parabolic Collector (PC), and Compound Parabolic Cylinder Collector (CPC), all covered with reflective materials. The objective was to determine the most efficient collection system for degrading Safranin T (SF), 2,4-Dichlorophenoxyacetic acid (2,4-D), and Sulfacetamide (SAM) using solar radiation.

The main focus of this research is to investigate the enhancement of photodegradative systems for the treatment of CEC using solar radiation. The solar collectors for photocatalytic applications in CEC removal were designed based on the measurements of the reactor to be powered. The study also examined the impact of using radiation collectors specifically designed for photocatalytic processes in the treatment of CEC. The ultimate goal of this study is to expand knowledge in the design and utilization of solar collectors for the photocatalytic oxidation of CEC using solar light.

2. Materials and Methods

In this research, several chemicals were used, including TiO_2 P-25 (Evonik®), Safranin T (Carlo Erba, 100%), a commercial herbicide PROFIAMINA® 720 SL containing 720 g L^{-1} of 2,4-dichlorophenoxyacetic acid, and Sodium sulfacetamide provided by Corpaul (Medellín, Colombia).

The first stage of the research aimed to determine the best reaction conditions. A one-liter reservoir was employed to hold the contaminated solution, which was recirculated using two immersion pumps (JAD References FP-750 and FP-250). The solution was directed to a cylindrical reactor with quartz walls, having an internal diameter of 2.0 cm, external diameter of 2.2 cm, and a length of 13 cm. The effective irradiation volume of the reactor was 35.5 cm^3. The setup included a UV lamp (MoodLites) with a maximum emission wavelength at 365 nm and power of 13 W. For all tests, a 500 mL contaminated solution with a pollutant concentration of 20 mg L^{-1} was prepared, based on previous studies [12].

To determine the best degradation conditions, an experimental design was established using Safranin T as the pollutant. The operating conditions included the presence of air (through an aerator pump Jeneca Reference AP-9800), high and low flow rates using two different pumps. The experimental design involved a n^k factorial design with 2 factors: air flow (0 and 0.6 L min-1) and recirculation flow of the solution (30.73 mL s^{-1} and 94.84 mL s^{-1}). Additionally, three control experiments were conducted: dark adsorption, photolysis with a flow rate of 30.73 mL s-1, and photolysis with a flow rate of 94.84 mL s^{-1} (referred to as C1, C2, and C3, respectively). The experimental setup and variations in flow rate and aeration are presented in Table 1. A total of 7 experiments were performed, including 4 with varying conditions and 3 control experiments, each replicated twice.

The degradation of Safranin T and 2,4-D was monitored using a spectrophotometer Jenway - 7200 series. Safranin T was measured at 520 nm, while 2,4-D was measured at 280 nm. For the degradation analysis of Sodium sulfacetamide, a UHPLC THERMO 3000 system was used with a C18 reverse-phase column (2.7 mm x 3 mm x 150 mm, Restek®), a flow rate of 0.5 mL min^{-1}, and an acetonitrile/Buffer mobile phase (Formic acid/formiate: 65/35). A UV detector at 257 nm was employed. Figure 4 illustrates the experimental setup used in this stage.

Table 1. Experimental design to determinate the best operation conditions.

Experiment	Air Flow (L min^{-1})	Q (mL s^{-1})
1	0	30.73
2	0	94.84
3	1.6	30.73
4	1.6	94.84

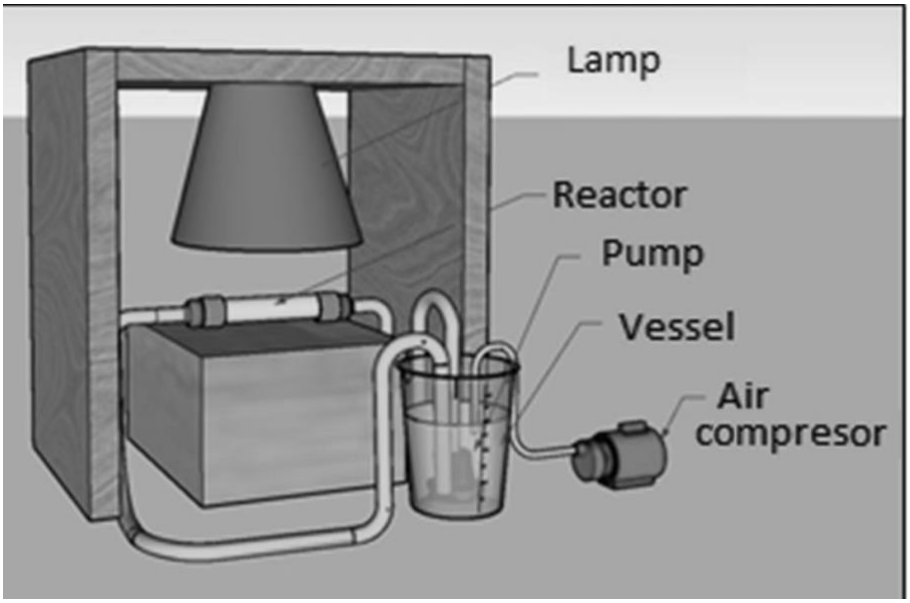

Figure 4. Reaction system in the first stage.

TiO_2 was dispersed in the system at a concentration of 1 g L^{-1} [12]. In the initial stage, a dark absorption test (C1) was conducted to assess the interaction between safranin and the catalyst. Photolysis of the pollutant was then evaluated at high and low flow rates (C2 and C3). The Heterogeneous Photocatalysis (HP) process was examined by varying the flow rate and air presence according to the conditions specified in Table 1 (Experiments 1-4). Once the best reaction conditions were determined in the first stage, the treatment of contaminated water containing Safranin T (SF), Sulfacetamide (SAM), and 2,4-Dichlorophenoxyacetic acid (2,4-D) at a concentration of 20 mg L^{-1} was performed under solar irradiation using four different radiation collector configurations: Flat Plate Collector (FPC), V Collector (VC), Parabolic Collector (PC), and Compound Parabolic Cylinder Collector (CPC), all coated with an aluminum reflective material (second stage). The comparison of pollutant degradation was conducted until an accumulated energy of 122.77 kJ m^{-2} was reached in the system. The collectors were designed using 3D modeling software, SketchUp®, the designed collectors were fabricated using a Fused Form 600® 3D filament printer, utilizing a PLA (polylactic acid) filament.

3. Results and Discussion

First stage: determination of the best degradation conditions

In the first stage of control experiments, dark adsorption of Safranin T (SF) resulted in a degradation of 9.8% (C1), indicating an affinity between SF and TiO_2, which promotes its degradation. Photolysis experiments (C2 and C3) revealed that SF degradation was higher at a high flow rate (94.84 mL s^{-1}) with a degradation percentage of 8.68%, while a lower degradation of 2.98% was observed at a low flow rate (30.73 mL s^{-1}), as depicted in Figure 5.

Regarding the effect of radiation (C1) at the studied wavelength and the dark adsorption of the contaminants Sodium sulfacetamide (SAM) and 2,4-Dichlorophenoxyacetic acid (2,4-D) on the catalyst, no significant decreases in the concentration of these pollutants were observed.

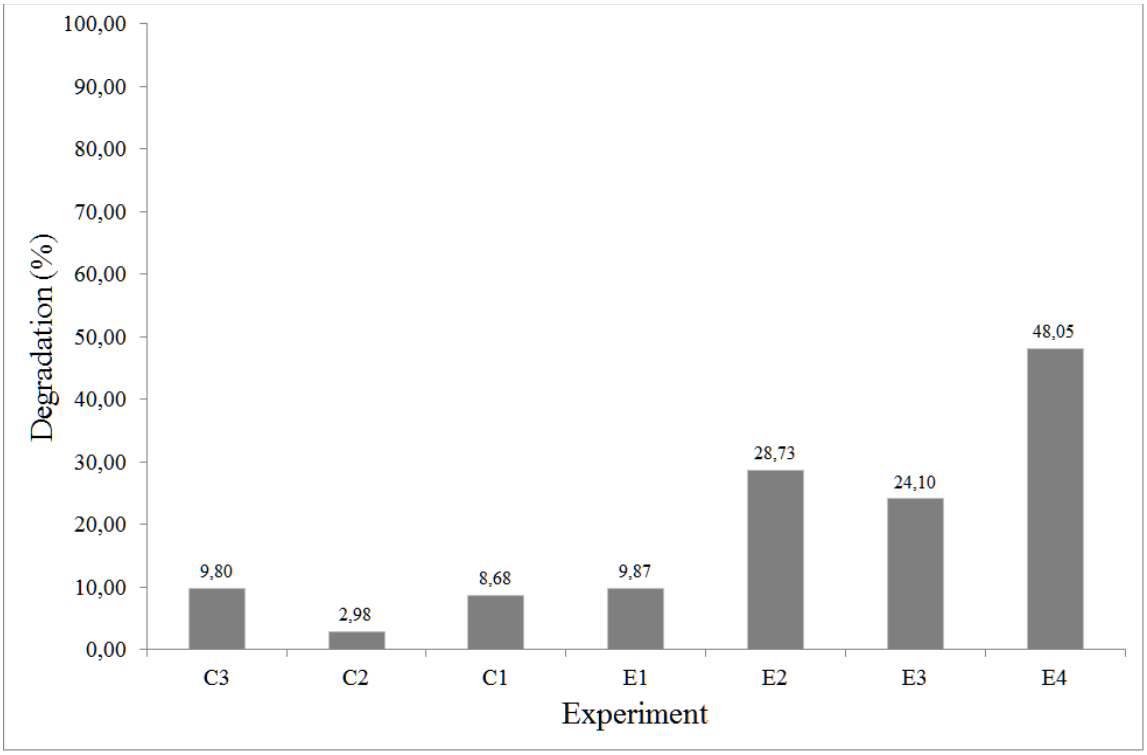

Figure 5. First stage results to determine the best conditions for SF degradation.

Based on the results shown in Figure 5, it was observed that the presence of oxygen and high flow rate significantly favored the degradation of the contaminants. This indicates that a longer contact time between the solution and radiation occurs, leading to enhanced degradation and system efficiency. Among the flow and aeration parameters, Experiment 4 (Table 1) was found to be the optimal operating condition for pollutant degradation, achieving a degradation rate of 48.05% for Safranin T (SF).

When analyzing the influence of the solution flow rate, it was observed that higher flow rates (Experiments 2 and 4, Table 1) resulted in greater degradation. Several factors contribute to this observation. Firstly, in a recirculation system, the higher flow rate provided three times more photons to the solution, generating a greater number of electron-hole pairs and reactive species through the interaction between light and catalyst. Additionally, the increased flow rate promotes turbulence and improved mass transfer, facilitating contact between the reacting species and leading to higher levels of contaminant removal. Importantly, in the heterogeneous system with suspended TiO_2, no precipitation or adhesion of the catalyst was observed on the walls or in the reservoir throughout the flow regimes studied. Therefore, no shielding effect or changes in the amount of titanium in the system were observed.

Furthermore, the presence of air in the system allows oxygen to react with the excited electron, generating the superoxide anion radical ($^{\bullet}O_2^-$) and other reactive oxygen species (ROS), which contribute to the oxidation of the pollutants [13]. Based on these findings, the other selected contaminants, 2,4-D and SAM, were degraded under the same best conditions. Figure 6 presents the degradation rates of the three pollutants using the best conditions determined for SF, illustrating the resistance exhibited by certain molecules towards degradation.

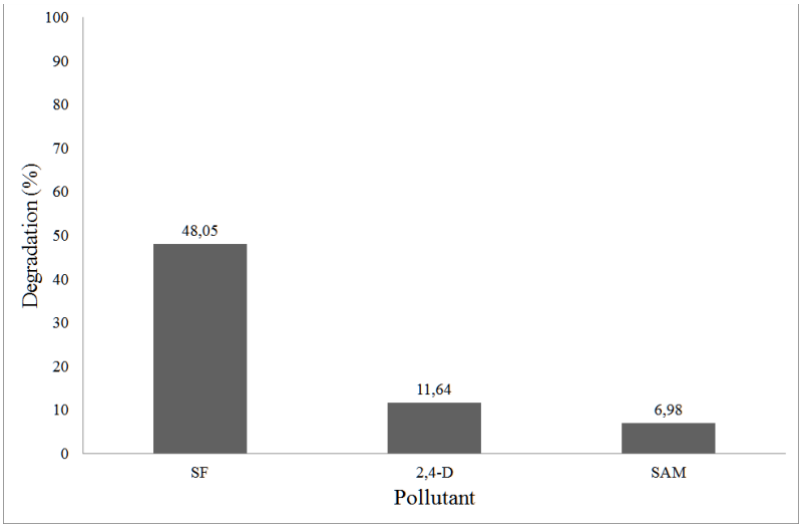

Figure 6. CEC degradation in best conditions using a UV lamp without collector.

Radiation collector's design

The design of the collectors was determined using the dimensions of the reactor, including its length (13 cm), internal diameter (2.1 cm), and external diameter (2.2 cm). Each collector (except for the Flat Plate Collector, FPC) was designed using specific equations corresponding to their geometries.

The V Collector (VC) design utilized the absolute value function (Equation 1) [11], with values for x ranging from -3 to 3, resulting in a 90-degree angle. This allowed for the creation of a 3D model of the collector using SketchUp® software. Figure 7 illustrates the design of the V Collector, which was subsequently used for the printing process.

$$f(x) = |x| \quad (1)$$

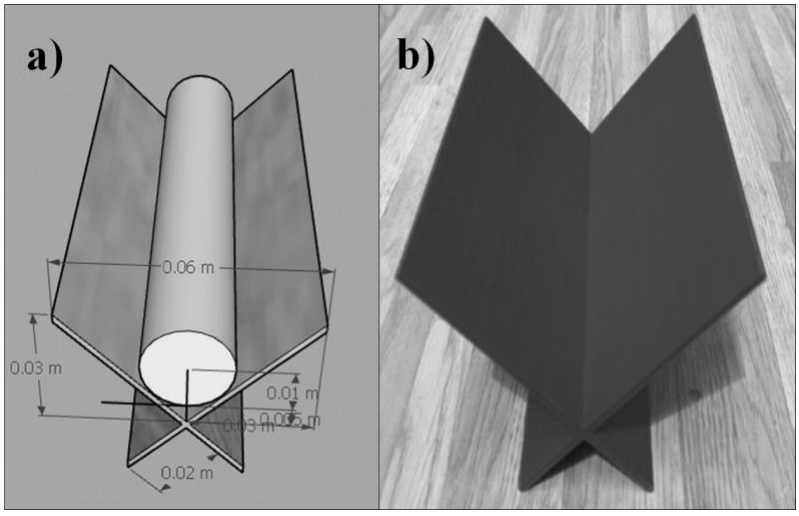

Figure 7. V Collector. a) 3D SketchUp® model. b) VC 3D printed.

On the other hand, for the Parabolic Collector (PC) with the reactor dimensions, the design was based on the parabolic function (Equation 2) [8]. By using Equation 3 derived from the parabolic function, the 3D model of

the Parabolic Collector was created (Figure 8a). The design was then translated into a physical collector through 3D printing (Figure 8b).

$$(h; k)^2 = 4p(y - k) \quad (2)$$

Where (h; k) is the vertex with coordinates (0; 0), p is the focal length (2.2 cm), and (h; k + p) is the focus with coordinates (0; 2.2)

$$f(x) = \frac{x^2}{8.8} \quad (3)$$

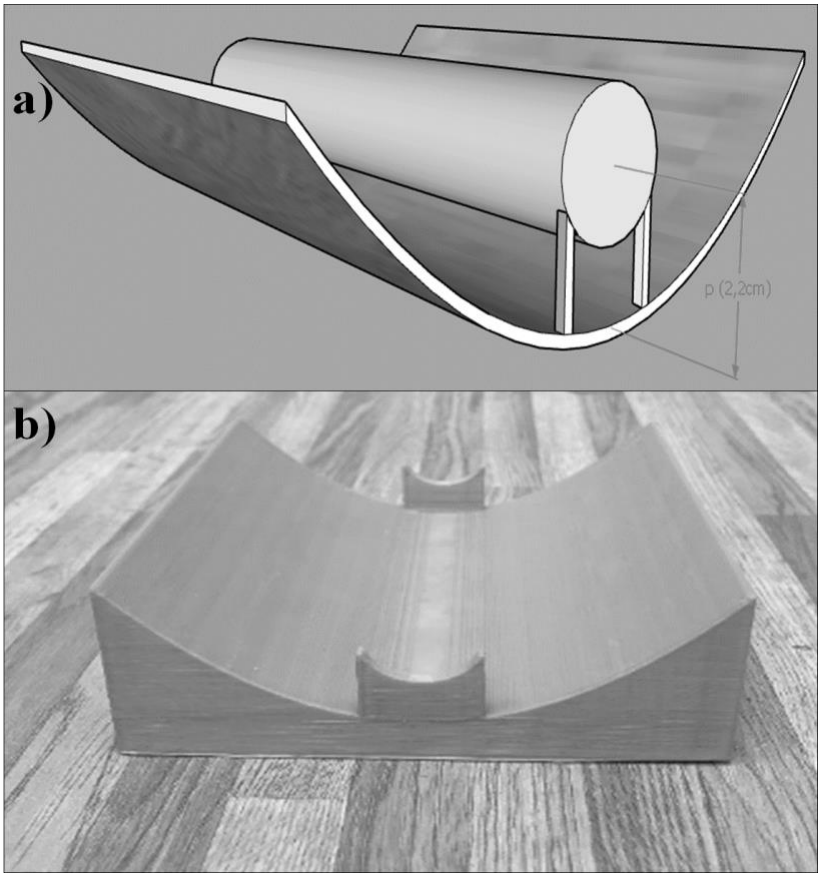

Figure 8. Parabolic Collector. a) 3D SketchUp® model. b) PC 3D printed.

It is important to highlight that 3D printing has become a widely accessible technology and has gained relevance in Advanced Oxidation Processes, as it enables the evaluation of small-scale degradation processes beforehand. PLA, the material used in 3D printing, is known for its environmentally friendly nature. Unlike materials derived from finite resources like oil, PLA is derived from renewable sources. Additionally, PLA-printed parts exhibit resistance, flexibility, and low flammability, making them suitable for the intended application.

Considering the design of a Compound Parabolic Cylinder Collector (CPC) with a concentration factor of 1, and given the external radius of the photoreactor as 11 mm, the CPC opening (a) was determined to be 69.11 mm using the radiation concentration equation (Equation 4) [8]. Subsequently, utilizing the involute equation

(Equation 5) and the measurements of the reactor, Equation 6 was derived, representing one of the sheets of the CPC involute that needed to be sketched.

To facilitate the printing of the CPC, Equation 6 was transformed into rectangular coordinates (Equation 7). Based on these transformed coordinates, the design of the CPC was finalized and prepared for 3D printing (Figures 9a and 9b).

$$CR = \frac{a}{2 * \pi * r} \tag{4}$$

Where *CR* is concentration factor, *a* is collector opening (mm), and *r* is reactor external radius (mm).

$$f(\theta) = DIR + RI\ (\theta) \tag{5}$$

Where *DIR* is the reactor internal diameter (mm) and *RI* is the reactor internal radius (mm)

$$f(\theta) = 21 + 10.5\ (\theta) \tag{6}$$

$$(x^2 + y^2)^{\frac{1}{2}} - 10.5 \tan^{-1}\left(\frac{y}{x}\right) = 21 \tag{7}$$

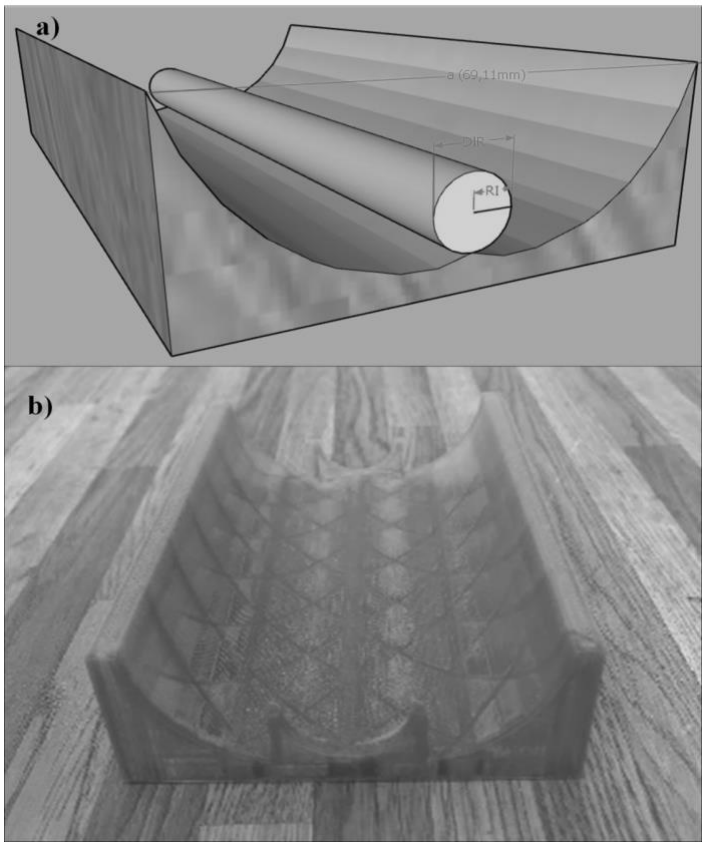

Figure 9. Compound Parabolic Cylinder Collector. a) 3D SketchUp® model. b) CPC 3D printed.

Solar photodegradation of pollutants

In the second stage of the research, the degradation tests of the three selected pollutants (Safranin T, 2.4-Dichlorophenoxyacetic acid, and Sodium sulfacetamide) were conducted using the best conditions determined earlier (presence of air and high flow rate) in combination with a TiO_2 concentration of 1 g L^{-1} and solar radiation. These tests were performed on the designed collectors until reaching an accumulated energy of 122.77 kJ m^{-2} in the system. Figure 10 presents the degradation results obtained for the pollutants using the four different radiation collectors.

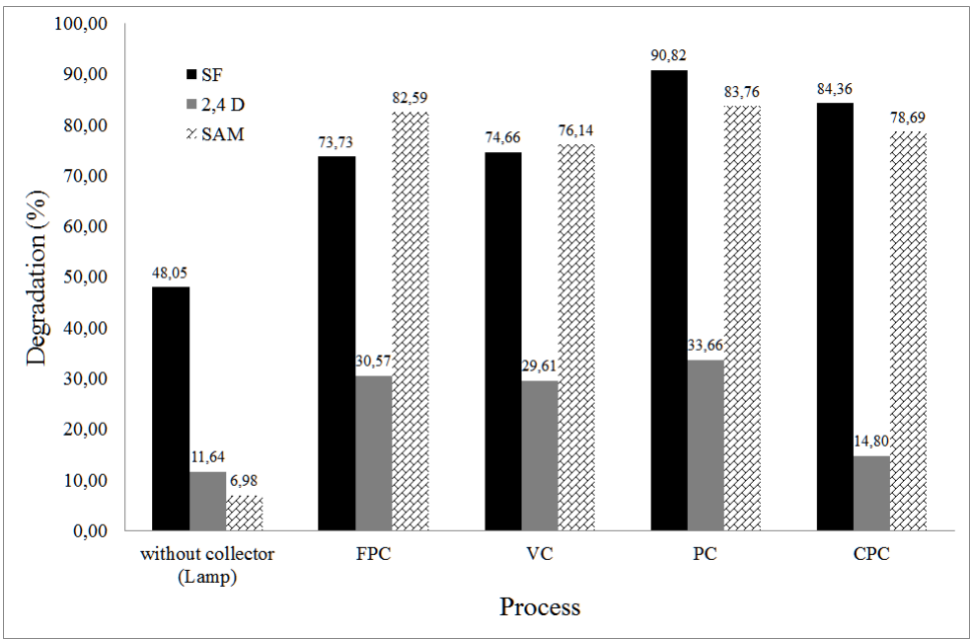

Figure 10. Degradations achieved using the 4 radiation collectors (FPC, VC, PC, CPC) for the 3 pollutants (SF, 2,4-D and SAM).

Firstly, it is evident that the use of solar radiation collectors significantly improves the photodegradation percentages of the selected molecules in this study, with enhancements ranging from 1.5 to 12 times compared to tests conducted without collectors. For Safranin T (SF), although a degradation of 90.82% was achieved with the Parabolic Collector (CP) (Figure 10), the enhancement effect was only 1.89 times since its initial removal with a lamp was 48% under the best treatment conditions. Conversely, when examining the behavior of 2,4-Dichlorophenoxyacetic acid (2,4-D), the use of collectors improved its degradation between 1.27 and 2.89 times (greater than for SF, Figure 10). However, the overall degradation achieved for 2,4-D was only 33.66% due to its high stability and low degradability [14].

In the case of Sodium sulfacetamide (SAM), which exhibited the most significant enhancement with the use of collectors, degradation rates of 10.91 - 12 times higher were obtained (Figure 10). This can be attributed to SAM's absorption of radiation in the visible spectrum, which favors its denaturation [12].

Regarding the comparison of the designed collectors, it was observed that the parabolic geometry performed the best (Figure 10). However, there were no significant differences in the overall performance between the collector designs, as similar photodegradation percentages were achieved for each molecule studied. This emphasizes the importance of correctly designing radiation collectors in photocatalytic applications.

To assess the impact of the designed systems when used with solar radiation, the Collector Impact Ratio Factor (CIRF) was calculated. This factor was estimated by comparing the degradation achieved with each collector system to the degradation obtained without a collector. A ratio greater than 1 indicates an improvement in the degradation system, a ratio less than 1 signifies a decline in degradation, and a ratio equal to 1 indicates that the collector has no effect on degradation, either positive or negative (Figure 11).

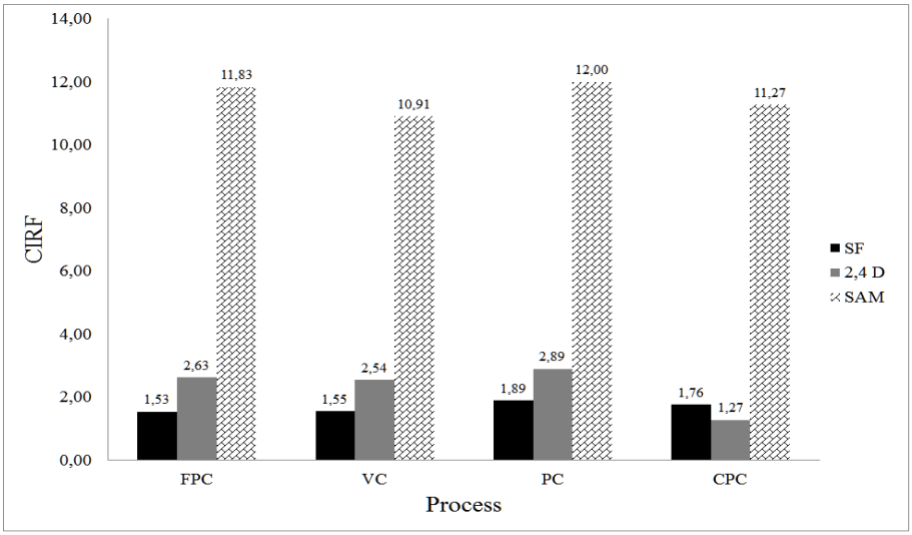

Figure 11. Collector Impact Ratio Factor (CIRF) to SF, 2,4-D and SAM photodegradation using solar radiation.

Overall, the use of collectors significantly improved the performance of the photocatalytic systems employed [8, 13]. Figure 11 demonstrates that the parabolic collector (PC) exhibited the highest increase in degradation for the three pollutants, highlighting its effectiveness in solar applications [10, 9]. Analyzing the behavior of each pollutant in the designed collectors, the greatest impact was observed for Sodium sulfacetamide (SAM), with degradation enhancement ranging from 11 to 12 times. This can be attributed to SAM's sensitivity to visible light, which constitutes approximately 45% of solar radiation [15].

On the other hand, Safranin T (SF) exhibited the lowest Collector Impact Ratio Factor (CIRF), as its degradation increased from 48% without a collector to 90.82% with the use of the CP (Figure 10). Comparatively, the CIRF value for 2,4-Dichlorophenoxyacetic acid (2,4-D) was higher than that of SF. However, the degradation levels achieved for 2,4-D were not as high, reaching approximately 33.66% under the best conditions (Figures 10 and 11). This underscores the considerable stability of the 2,4-D molecule.

While the CP demonstrated the best performance among the four designed collectors, it is important to note that the differences in performance between the collectors were similar for each of the pollutants studied (Figures 10 and 11).

4. Conclusions

The dark adsorption test confirmed the affinity between the contaminants and the photocatalyst, which facilitated their degradation upon irradiation. Secondly, the introduction of aeration into the degradation system promoted a higher level of pollutant degradation by facilitating the formation of reactive oxygen

species (ROS), thereby enhancing the degradation process. Additionally, it was observed that a higher flow rate contributed to the degradation of pollutants. This can be attributed to the longer exposure time of the solution to be treated under high flow conditions, leading to improved removal of contaminants.

Moreover, the use of solar radiation, particularly with the designed collectors, intensified the degradation of pollutants. The collectors' appropriate design played a crucial role, especially in the case of more resistant molecules under the evaluated conditions. The inclusion of radiation collectors resulted in remarkable improvements, with pollutant degradation levels reaching up to 12 times higher than those achieved without collectors.

Although the parabolic collector (PC) demonstrated the best performance among the designed collectors, no significant differences were observed regarding the enhancement of solar radiation utilization by the collectors used in the photochemical processes investigated in this study.

Acknowledgements: Authors thanks to Institución Universitaria Colegio Mayor de Antioquia through by the financial support.

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