Radiation collector systems comparison in Contaminants of Emerging Concern degradation by solar heterogeneous photocatalysis

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Abstract

Wastewater with Contaminants of Emerging Concern (CEC) can be generated from different sources as industry, agriculture and urban and hospital wastes. Heterogeneous Photocatalysis (HP) with TiO$_2$ is one of the Advanced Oxidation Processes (AOPs) most suitable for water treatment with CEC. In this research, three CEC: Safranin T (SF), 2,4-dichlorophenoxyacetic acid (2,4-D) and Sulfacetamide (SAM) degradation was evaluated by solar-HP in a quartz wall reactor. First, 365 nm wavelength radiation was used and the best operating conditions was determined under the high flow and aeration configuration, obtaining a removal rate of 48.05% for SF, 11.64% for 2,4-D and 6.98 for SAM. Then, under these conditions, SF, SAM and 2,4-D degradation with solar lighting was made on 4 radiation collector systems configurations, Flat Plate Collector (FPC), V Collector (VC), Parabolic Collector (PC) and Compound Parabolic Cylinder Collector (CPC) until reaching the same value of accumulated energy (122.77 kJ m$^{-2}$) finding that the PC had the best performance in the treatment for the three pollutants. Finally, the Collector Impact Ratio Factor (CIRF) for the pollutants was calculated, achieving until 12 times degradation for SAM.

Keywords

Contaminants of Emerging Concern, Advanced oxidation processes, Radiation collectors, TiO$_2$, Solar radiation, Safranin, 2,4-dichlorophenoxyacetic acid, Sulfacetamide.
Declarations

Ethics approval and consent to participate: Not applicable.

Consent for publications: Not applicable.

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Authors' contributions:

Fidel Granda-Ramírez: Designed and conducted the experiments, analyzed, interpreted data and was a major contributor in writing the manuscript.

Melissa Barrera: Designed and conducted the experiments, analyzed, interpreted data and helped writing the manuscript.

Sara Castrillón: Designed and conducted the experiments, analyzed, interpreted data and helped writing the manuscript.

Lady Rueda: Designed and conducted the experiments, analyzed, interpreted data and helped writing the manuscript.

Juan Pino-Arango: Conducted the experiments.

Gina Hincapié-Mejía: Check the manuscript.

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1. Introduction

Currently, there is a growing interest in Contaminants of Emerging Concern (CEC) whose presence in the environment has largely gone unnoticed in terms of distribution and/or concentration (Tanga 2020). These involve a wide range of chemical compounds that are used in daily life, including medicines, personal hygiene products, soaps, surfactants, industrial additives, plasticizers, pesticides and a wide variety of chemical compounds that are present in the environment (Rodil 2019). Many of these pollutants are not eliminated through conventional systems; and, for the most part, correspond to substances not regulated by the environmental authorities. Among the dyes belonging to the CEC group, is Safranin T (SF), which is a biological dye that is frequently used to dye tissues, the detection of structures in eukaryotic and prokaryotic cells and also, it’s most common use it is in Gram staining (Aguirre 2012). 2,4-Dichlorophenoxyacetic Acid is one of the most widely used compounds in commercial herbicides, being responsible for causing devastating effects on the environment, especially in aquatic ecosystems and human health. Due to its persistent nature, it is not degraded efficiently by wastewater treatment plants, and it is necessary to use other more effective degradation methods such as Advanced Oxidation Processes (Samir 2015). On the other hand, Sodic Sulfacetamide (SAM) is an ophthalmic drops or ointment in the treatment of eye infections, it is provided orally for the treatment of acne and seborrheic dermatitis, and is rapidly excreted in the urine and it can be found in soap, shampoo, cream and washing solutions (Bendjama 2018).

Advanced Oxidation Processes (AOP) are defined as effective methods for the removal of a large number of persistent contaminants. These make changes in the chemical structure of pollutants until they reach mineralization, transforming organic matter into carbon dioxide and water (Samir 2015). One of the most AOP used is the Heterogeneous Photocatalysis (HP) in which a semiconductor is irradiated to generate reactive species and the greatest used in photocatalytic applications is TiO$_2$, particularly the P-25 (Evonik®), because it presented a higher photocatalytic activity, is not toxic, is stable in aqueous solutions and it is not expensive (Bendjama 2018). In photocatalytic process with TiO$_2$ in aerobic environments, when ultraviolet light radiation striking a surface of a semiconductor, this one is capable of generating electron-hole pairs and Reactive Oxygen Species (ROS) such as hydroxyl radicals (•OH), superoxide anions (•O$_2^-$), hydroxide radicals (•OH$_2$) or Hydrogen Peroxide (H$_2$O$_2$) (Kanakaraju 2018).
photocatalytic processes with TiO$_2$, in addition to properly locating the catalyst (either dispersed or through the implementation of supports) it is necessary to achieve an efficient exposure to the useful light, to ensure an adequate process, said exposure can be carried out using systems of radiation collectors. Globally, various collector designs have been proposed which seek to increase efficiency and reduce the costs of photocatalytic systems for decontamination and water treatment (Zhu 2019). HP with TiO$_2$ as a catalyst uses only the UV fraction of the solar spectrum (direct or diffuse). An important aspect of the collectors is the refractive surface that covers them, since this has the purpose of directing and reflecting the useful light towards the reactor to achieve maximum use of it and avoid unnecessary losses. Among the best reflective materials are aluminum-based mirrors (Blanco 2004).

An important aspect of the collectors is the refractive surface that covers them, since this has the purpose of directing and reflecting the useful light towards the reactor to achieve maximum use of it and avoid unnecessary losses. Among the best reflective materials are aluminum-based mirrors (Bandala 2004). The most widely used collector designs are Parabolic Collectors (PC) (Figure 1), V (VC) (Figure 2) and Compound Parabolic Cylinder (CPC) (Figure 3) among others (Bandala 2004).

![Figure 1. Parabolic Collector (PC).](image1)

![Figure 2. V Collector (VC).](image2)

![Figure 3. Compound Parabolic Cylinder Collector (CPC).](image3)

Due to the problem that has triggered the generation of CEC, in recent years, proposals have been proposed for the treatment of these compounds through the AOP. Collectors without radiation concentration have been widely used in the HP, the CPC system being one of the most efficient and with the best technology available (Olleros 2013). The HP implementation with TiO$_2$ using sunlight as a radiation medium and using a CPC as a collector has proven to be an adequate system for the degradation of a mixture of 15 CEC, obtaining degradation of 90% of the pollutants (Maldonado).

Through this research, a comparison of 4 radiation collectors configurations Flat Plate Collector (FPC), V Collector (VC), Parabolic Collector (PC) and Compound Parabolic Cylinder Collector (CPC)
covered with a reflective material was performed, in which the most efficient collection system for SF,
2,4-D and SAM degradation using solar radiation was determined.

2. Materials and methods

In this research were used TiO$_2$ P-25 (Evonik®), Safranin T (Carlo Erba, 100%), as a source of
2,4-dichlorophenoxyacetic acid, a commercial herbicide PROFIAMINA® 720 SL was used with a
content of 720 g L$^{-1}$ thereof and Sodium sulfacetamide was provided by Corpaol (Medellín, Colombia). In
the first stage of this research, a 1 liter reservoir was used to contain the solution to be treated; this
solution is recirculated by an immersion pump (JAD Reference FP-750) to the cylindrical reactor with
quartz walls (internal diameter 2.0 cm, external diameter 2.2 cm and length of 13 cm). This reactor has an
effective irradiation volume of 35.5 cm$^3$. The system was equipped with an UV lamp (MoodLites,
maximum emission wavelength at 365 nm and 13 W of power) to determine the best reaction conditions
and thus proceed to degrade the CEC with solar irradiation. For all tests, 500 mL of a solution at
20 mg L$^{-1}$ of the pollutants was prepared, corresponding to the amounts found in previous studies
(Granda-Ramírez 2017). Initially, for determination of the best degradation conditions using UV lamp, SF
was used. Subsequently, the high and low flow rates were determined by averaging nine measurements,
obtaining the values of 94.84 mL s$^{-1}$ and 30.73 mL s$^{-1}$. Likewise, the effect of the presence of oxygen in
the system through an air supply was evaluated with an aerator pump (Jeneca Reference AP-9800 with a
flow at 1.6 L min$^{-1}$). Variations in flow rate and aeration (Table 1) were made with the objective of
finding a reactor operating condition that allows the best possible degradation of the contaminant. In the
follow of pollutants degradation a spectrophotometer Jenway – 7200 series was used. The SF was
measured at 520 nm, 2,4-D at 280 nm and SAM at 260 nm (Figure 4).

| Exp. | Air | Q (mL s$^{-1}$) | Rad | TiO$_2$ |
|------|-----|----------------|-----|---------|
| C1   | NO  | High           | X   |         |
| C2   | NO  | Low            | X   |         |
| C3   | NO  | High           | X   |         |
| E1   | NO  | Low            | X   | X       |
| E2   | NO  | High           | X   | X       |
| E3   | YES | Low            | X   | X       |
| E4   | YES | High           | X   | X       |

Table 1. Experimental design to determine the best operation conditions.
Figure 4. First stage´s reaction system.

TiO$_2$ was dispersed in the system with a concentration of 1 g L$^{-1}$ (Granda-Ramírez 2017). A dark absorption test was initially applied to establish the affinity between safranin and the catalyst, additionally (C1, Table 1), evaluation of the pollutant photolysis operating at high and low flow rates was made (C2 and C3, Table 1). The HP was evaluated by varying the flow rate and the air presence as described in Table 1 (E1 – E4). Once the best reaction conditions had been established in the first stage, the treatments of the contaminated waters with SF, SAM and 2,4-D (20 mg L$^{-1}$) were carried out under solar irradiation using 4 radiation Collectors configurations FPC, VC, PC, CPC coated by an aluminum reflective material (second stage). The comparison of the pollutants degradation was made until to accumulate 122.77 kJ m$^{-2}$ of energy in the system. Collectors designed were made through 3D modeling with SketchUp® software and the elaboration was done with a Fused Form 600 ® brand 3D filament printer, using a PLA filament.

3. Results and discussion

3.1 First stage: determination of best degradations conditions.

For the first stage, Figure 5 shows that SF Dark adsorption showed a 9.8% of contaminant adsorbed in the catalyst, establishing that there is an affinity between SF and TiO$_2$ that favors its degradation. Through the photolysis experiments, a higher degradation of SF at a high flow rate was determined obtaining a removal of 8.68%, while a 2.98% at a low flow rate was obtained. According to the results obtained it was determined that the oxygen presence favors the degradation of the contaminant. On the other hand, it was determined that the high flow rate (94.84 mL s$^{-1}$) gives a greater degradation of the contaminant. This flow has a speed 3 times higher compared to the low flow (30.73 mL s$^{-1}$), this indicates that the solution to be treated experiences a longer contact time with the radiation which allows a greater degradation and efficiency of the system. Analyzing the flow and aeration parameters, the best operating condition for degradation was provided by the E4 (Table 1) experiment (48.05%) set by a high flow rate and air supply.
Figure 5. First stage results to determine the best conditions for SF degradation.

In this way, it is determined that the air supply present in experiments E3 and E4 favors the degradation of the pollutant. Since the air present in the system allows oxygen to react with the excited electron, forming the radical superoxide anion (\(\bullet O_2^-\)) and other ROS contributes to the oxidation of the pollutant (Doménech 2004). On the other hand, it was possible to observe that the high flow rate (94.84 mL s\(^{-1}\)) grants greater pollutant degradation as evidenced in experiments E2, E4 and in high flow photolysis. This flow has a speed 3 times higher compared to the low flow (30.73 mL s\(^{-1}\)), this indicates that the solution to be treated experiences a longer contact time with radiation, which allows better degradation and operation of the system.

Analyzing the flow and aeration parameters, the best operating condition for degradation was provided by experiment E4 (48.05%) configured by a high flow rate and air supply (Figure 5). With the previous results, the other pollutants selected for this study (2,4-D and SAM) were degraded. Figure 6 displays the 3 pollutants degradation rates using the optimal conditions found for SF, showing the resistance that some molecules show to being degraded.

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

3.2 Radiation collectors design.

Collectors design was made based on the reactor internal (2.1 cm) and external (2.2 cm) diameter and its length (13 cm). For each of the collectors, except for the plane, the corresponding design equations used for each geometry will be presented below.

For V Collector (VC), its design was based on the absolute value function (Equation 1) (Bandala 2004), with values for x between -3 and 3, which provides an angle of 90 degrees, it was possible to design the sensor through 3D modeling (SketchUp®) as shown in Figure 7.

\[
f(x) = |x|
\]  

(1)

Figure 7. V Collector. a) 3D SketchUp® model. b) 3D printed VC.
On the other hand, for the parabolic collector (PC) with the reactor measurements and based on
the parabola function (Equation 2) (Blanco 2004), Equation 3 is obtained, with which the parabolic
collector 3D modeling (Figure 8a) and subsequently 3D printed (Figure 8b).

\[(h; k)^2 = 4p(y - k)\]  \hspace{1cm} (2)

Where:

\((h; k)\) is the vertex with coordinates \((0; 0)\)

\(p\) is the focal length (2.2 cm)

\((h; k + p)\) is the focus with coordinates \((0; 2.2)\)

\(f(x) = \frac{x^2}{8.8}\)  \hspace{1cm} (3)

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

Knowing that a Compound Parabolic Cylinder Collector (CPC) is going to be designed with a
collection factor equal to 1 and that the photoreactor’s external radius is 11 mm, when using the
radiation concentration equation (Equation 4), it was determined that the CPC opening \((a)\) is 69.11 mm
(Blanco 2004).

\[CR = \frac{a}{2\pi r}\]  \hspace{1cm} (4)

Where:

\(CR\): concentration factor.

\(a\): collector opening (mm).

\(r\): reactor external radius (mm).

Then, based on the involute equation (Equation 5) and with the reactor measurements, equation 6
is obtained, which corresponds to the reactor involute that would be one of the CPC sheets (Blanco 2004).

\[f(\theta) = DIR + RI(\theta)\]  \hspace{1cm} (5)
Where:

DIR: reactor internal diameter (mm)

RI: reactor internal radius (mm)

\[ f(\theta) = 21 + 10.5(\theta) \]  \hspace{1cm} (6)

Finally, in order to print the CPC, it is necessary to transform equation 6 to rectangular coordinates (Equation 7).

\[ (\frac{x^2}{2} + \frac{y^2}{2})^2 - 10.5\tan^{-1}\left(\frac{y}{x}\right) = 21 \]  \hspace{1cm} (7)

From the x, y coordinates obtained from Equation 7, the CPC was designed in 3D (Figure 9a) and finally its 3D printing (Figure 9b).

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

3.3. Solar photodegradation of pollutants.

In the second stage, using the best conditions in SF degradation (air presence and high flow), degradation tests of the 3 pollutants chosen for this research (SF, 2.4 - D and SAM) were carried out using 1 g L\(^{-1}\) of TiO\(_2\) under solar radiation using the designed collectors until achieving 122.77 kJ m\(^{-2}\) of accumulated energy in the system. Figure 10 shows the pollutants degradations obtained with the use of the 4 types of radiation collectors.

**Figure 10.** Degradations achieved using the 4 radiation collectors for the 3 pollutants.

First, it can be observed that the use of solar radiation collectors improves in significant percentages the chosen molecules photodegradation for this study, since the percentages of degradation increase between 1.5 and 12 times the remotes obtained without them.
For SF, although a degradation of 90.82% was achieved with the CP (Figure 10), its enhancing effect was only 1.89 times since its initial removal with a lamp was 48% under better treatment conditions. On the other hand, when analyzing the behavior of 2,4-D, is why it can be observed that although the effect of the use of collectors improved its degradation between 1.27 and 2.89 times (greater than for SF, Figure 10) only achieved a contaminant degradation of 33.66%, this is due to the high stability and low degradability of the molecule (Álvarez 2007). Finally, in the case of SAM (molecule which the greatest effect of the use of collector was obtained in its photodegradation), removals were achieved between 10.91 - 12 times those achieved with lamps without radiation collector (Figure 10), which may be due to because this pollutant absorbs radiation in the visible spectrum, which favors its denaturation (Granda-Ramírez 2017).

From the point of view designed collectors comparison, it is observed those although the best geometry was the parabolic (Figure 10), in general there are not significant differences between their performances, since for each of the molecules studied a similar photodegradation percentages was achieved, demonstrating the importance of correctly designing the radiation collector in photocatalytic applications.

To determine the impact of the use of solar collectors, the Collector Impact Ratio Factor (CIRF) was determined by the relationship between the degradation obtained with each collector by the degradation obtained with the system without a collector. A ratio higher than 1 indicates an improvement in the degradation system, less than 1 a decline in degradation, and a ratio equal to 1 indicates that the collector does not affect degradation either positively or negatively (Figure 11).

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

Figure 11 shows that in general, the use of collectors improved the photocatalytic system used performance (Blanco 2004 and Doménech 2004), observing that the parabolic collector (PC) is the one that most increases the degradation of the 3 pollutants, therefore This geometry type is one of the most
widely used generally in solar applications (Maldonado 2015 and Olleros 2013). Analyzing the behavior of each pollutant in the designed collectors, it’s can see that there was the greatest impact for SAM, in which the degradation increase was around 11 to 12 times, this can be explained by the fact that SAM is sensitive to visible light and solar radiation have about 45% of this kind of radiation (Hincapié-Mejía 2020). On the other hand, it can be observed that the molecule that has a lower value of the CIRF was the SF, since the degradation improved between 50 and 89%, going from a degradation of 48% without collector to 90.82% with the CP (Figure 10). Finally, although the CIRF value for 2,4-D is higher than that of SF, its removal levels are not so high, since around of 33.66% were achieved in the best case (Figures 10 and 11), thus showing the great stability that this molecule has. Finally, it can be said that although the performance of the CP was the best of the 4 collectors designed, actually the difference in their performance is similar in each of the pollutants studied (Figures 10 and 11).

4. Conclusions

First, Dark adsorption test determined that there is a contaminant affinity with the photocatalyst, thus facilitating its degradation at the time of irradiating the system. Second, aeration supplied to the degradation system allows greater degradation of pollutants due to the formation of reactive oxygen species (ROS) which potentiates degradation; also, It was possible to determine that the high flow favors the pollutants degradation since this, having a speed 3 times higher than the low flow, lets a radiation longer exposure time of the solution to be treated, thus achieving a better contaminants remotion. Respect to the using solar radiation intensified with collectors improves the pollutants degradation due to its adequate design, especially with the most resistant molecules under the conditions evaluated, since improvements were obtained in the degradation of pollutants up to 12 times that achieved without radiation collectors. Finally, although the collector with the best performance was the parabolic (PC), no significant differences were found in the magnification of the use of solar radiation by the collectors designed for the photochemical processes studied in this research.

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Figure 1. Parabolic Collector (PC).

Figure 2. V Collector (VC).

Figure 3. Compound Parabolic Cylinder Collector (CPC).
**Figure 4.** First stage’s reaction system.
Figure 5. First stage results to determine the best conditions for SF degradation.
Figure 6. CEC degradation in best conditions using a UV lamp without collector.
Figure 7. V Collector. a) 3D SketchUp® model. b) 3D printed VC.
Figure 8. Parabolic Collector. a) 3D SketchUp® model. b) 3D printed PC.
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