

Solar homogeneous catalysis to the removal of organic matter from slaughterhouse effluents undergone to a prior biological process

J. A. Hurtado, L. F. Valdez and C. J. Escudero  *

Departament of Biotechnology and Environmental, Universidad Autónoma de Guadalajara, Av. Patria 1201, 45129 Zapopan, Mexico

*Corresponding author. E-mail: carlos.escudero@edu.uag.mx

 CJE, 0000-0002-4085-943X

ABSTRACT

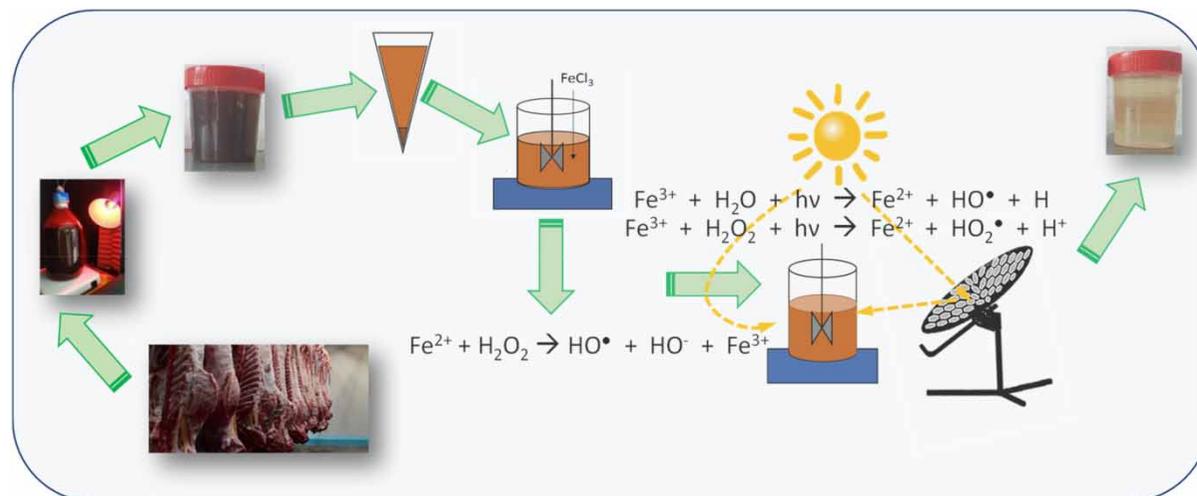
This study shows the effectiveness of the wastewater treatment from a municipal slaughterhouse that has undergone a previous biological treatment applying a sequence of stages, reaching a 75% of elimination of the chemical oxygen demand (COD) using sedimentation in combination with coagulation–flocculation, using 0.5 g/L FeCl_3 which is one of the best known inorganic coagulants. Then, the elimination of COD was around 98% adding the Fenton process in which 1,000 mg/L H_2O_2 and FeSO_4 were used. In addition to the COD, other water quality parameters were measured to evaluate the level of purification of the test samples, such as solids of different types, pH, DOC and so on. With the above process, it can be noted that the Fenton process produced a slight improvement in the effluent quality by using a solar concentrator in the now-called photo-Fenton process, reaching around 99% of COD removal (0.36 g/L), 91% of total suspended solids (0.32 g/L) and 89% of dissolved organic carbon (0.20 g/L). These results were the best achieved within a proposed treatment train for this type of complex wastewater. Moreover, this last part of the process adds an improvement by the usage of renewable energy sources such as sunlight.

Key words: coagulation–flocculation, Fenton, organic matter, slaughterhouse wastewater, solar photo-Fenton

HIGHLIGHT

- This article deals with work on real wastewater treatment under real working conditions, such as the use of sunlight. In addition, a parabolic solar concentrator built from reused material was used. So the project has elements of sustainability.

GRAPHICAL ABSTRACT



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INTRODUCTION

The effluents from municipal slaughterhouse contain a high concentration of thick solids, coat, grease, blood and organic matter, which makes their treatment difficult (Bustillo-Lecompte & Mehrvar 2015; Al-Najar & Nassar 2019). In Mexico there are more than 1,000 meat processing facilities with the average water consumption for processing being 1,000 L/cow and 450 L/pig processed (Paramo-Vargas *et al.* 2015). The production of cattle meat in Mexico during the last ten years increased to near 20 million tons from 85 million sacrificed animals, which implied the generation of 1 million cubic metres of wastewater (Hernandez *et al.* 2018). The poor management of the waste generated from the activities related to the agri-food sector, such as slaughterhouse wastewater, cause serious water pollution of many rivers in Mexico such as the Lerma–Santiago basin. This hydrological region is located in the central part of the country and is the most polluted watercourse in Mexico and one of the most committed basins in the world (Sedeño-Díaz & López-López 2007; Rizo-Decelis & Andreo 2016).

Biological treatments, such as anaerobic processes, can be effective in removing organic matter and nutrients, although the main disadvantage is the long stabilization time and specific operating conditions such as temperature (Aziz *et al.* 2019). However, biological processes can be treatment technologies prior to the application of physicochemical treatment or even advanced oxidation processes (AOP) to high load effluents such as those from slaughterhouse wastewater (Deng & Zhao 2015; Brink *et al.* 2018).

Precisely, coagulation–flocculation is one of the important processes that is involved in conventional wastewater treatment. Coagulation may be defined as adding those substances to remove colloidal impurities from water. The purpose of coagulation is to turn the small particles that generate turbidity into large flocs either as precipitates or suspended particles, which correspond to the flocculation stage (Riaz *et al.* 2012).

There have been several reports from researchers on slaughterhouse wastewater treatment, containing less than 4.0 g/L of chemical oxygen demand (COD), via coagulation–flocculation using different reagents, and reaching high efficiencies of organic matter removal (70–80% COD removal). Nevertheless, the concentrations of other parameters are still high, for example turbidity, color, or suspended solids, which implies requiring other treatment steps (Ha & Huong 2017; Gökçek & Özdemir 2020).

AOPs are technologies that generate and use highly oxidizing agents such as hydroxyl radicals (HO[•]) and facilitate the degradation of pollutants present in wastewater because these processes are not selective (Escudero *et al.* 2019). The application of AOPs such as Fenton and photo-Fenton processes turns out to be interesting for the treatment of wastewater that is difficult to treat via conventional methods. The Fenton process is characterized by the use of iron salts in combination with hydrogen peroxide (H₂O₂) in an acidified medium to generate highly oxidizing species such as HO[•] to degrade the organic pollutant. The reactions in the Fenton process are shown in Equations (1)–(4) (Ameta *et al.* 2018):



The combination of H₂O₂ and UV radiation with a Fe²⁺ or Fe³⁺ ion produces more HO[•] radicals and in turn, it increases the rate of degradation of organic pollutants. This process is known as the photo-Fenton process, for which the photochemical regeneration of ferrous ions by photo-reduction of ferric ions occurs in the following Equations (6) and (7) (Ameta *et al.* 2018):



The reduction of Fe³⁺ also creates another hydroxyl radical resulting in the hydrogen peroxide being fully converted into two radicals. Both UV and solar light can be used but a wavelength of below 310 nm will help with the production of radicals

seen in Equation (8) (ÓDowd & Pillai 2020):



In the present work the treatment of wastewater from a slaughterhouse that had undergone a previous anaerobic treatment was tested applying a treatment sequence composed of sedimentation followed by coagulation–flocculation and ending with an improved solar Fenton process (photo-Fenton) with direct lighting and using a parabolic concentrator.

The interest of working with an effluent from a biological reactor is to help close a treatment cycle between research groups, in which the group that treats the slaughterhouse wastewater via biological means obtains a by-product of interest such as the biomass proteins. The effluent derived from an anaerobic biological reactor is treated using processes that represent an environmentally friendly solution, such as the Fenton and solar photo-Fenton based on sunlight provides a cleaner energy than sources based on fossil fuels (Foteinis *et al.* 2018).

METHODOLOGY

The wastewater studied was collected from a research laboratory at the University of Guanajuato (Mexico) where samples from a municipal slaughterhouse located in the central part of Mexico received anaerobic biological treatment using purple phototrophic bacteria. For practical purposes in this study, the slaughterhouse wastewater sample from the biological reactor will be named raw wastewater (RWW).

These pre-treated samples via biological means were characterized in accordance with standardized procedures, shown in Table 1. The organic matter was measured in terms of the COD, the total suspended solids (TSS), settleable solids, pH, conductivity and dissolved organic carbon (DOC). It should be noted that these parameters are the basic ones used for evaluating water quality, some are included in Mexican regulations.

The potentiometer used to measure pH was from Hanna and the conductivity was determined in a Horiba device. The equipment used for the determination of the COD and the DOC were a digester (Hach) (Figure 1(a)) and a TOC-L instrument (Shimadzu Scientific Instruments) (Figure 1(b)), respectively.

To increase the effectiveness of the processes, a prior sedimentation (Sedim) of the RWW was applied for around 8 h (Figure 2(a)) using the standard Imhoff cone method. This stage is justified because the high concentration of solids present in the samples could hinder subsequent treatments, and sedimentation is one of the separation mechanisms commonly applied to slaughterhouse effluents (Bustillo-Lecompte & Mehrvar 2015).

It is important to emphasize that the performance of the Fenton process and coagulation–flocculation is dependent on many factors, such as the pH, chemical dose rates, reaction times and others. However, the conditions under which this part of the research was performed were subject to the results previously found by our research group with samples of high organic load (Escudero *et al.* 2020).

After sedimentation of the study sample, part of the clarified material obtained was treated for 30 min by Fenton and photo-Fenton processes under direct sunlight using 0.5 g/L of ferrous sulfate (FeSO₄, Golden Bell) and 1.0 g/L of H₂O₂ in 30% solution (J.T. Baker). In these last two AOP, the pH of the samples was adjusted to 4 with a few drops of sulfuric acid solution (H₂SO₄, J.T. Baker) before these treatments as has usually been done in tests prior to this research (Escudero *et al.* 2020).

Table 1 | Applied methods for the characterization of the wastewater samples studied

Physicochemical parameter	Unit	Applied methods
COD	g/L	NMX-AA-030-SCFI-2001
TSS	g/L	NMX-AA-034-SCFI-2015
Settleable solids	mL/L	NMX-AA-004-SCFI-2013
pH	Units of hydrogen potential	NMX-AA-008-SCFI-2016
Conductivity	μS/cm	NMX-AA-093-SCFI-2018
DOC	g/L	N-POC method from Shimadzu

*NMX = Mexican standards.

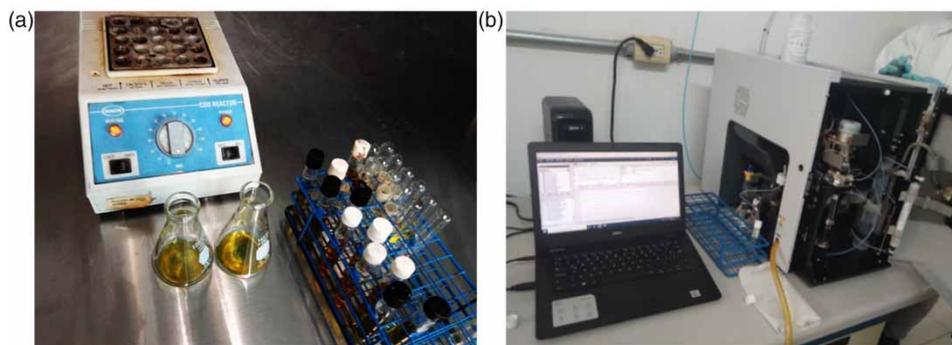


Figure 1 | (a) Reactor–digester for chemical oxygen demand determination and (b) total organic carbon analyzer of manual injection. Please refer to the online version of this paper to see this figure in colour: <http://dx.doi.org/10.2166/wst.2021.438>.

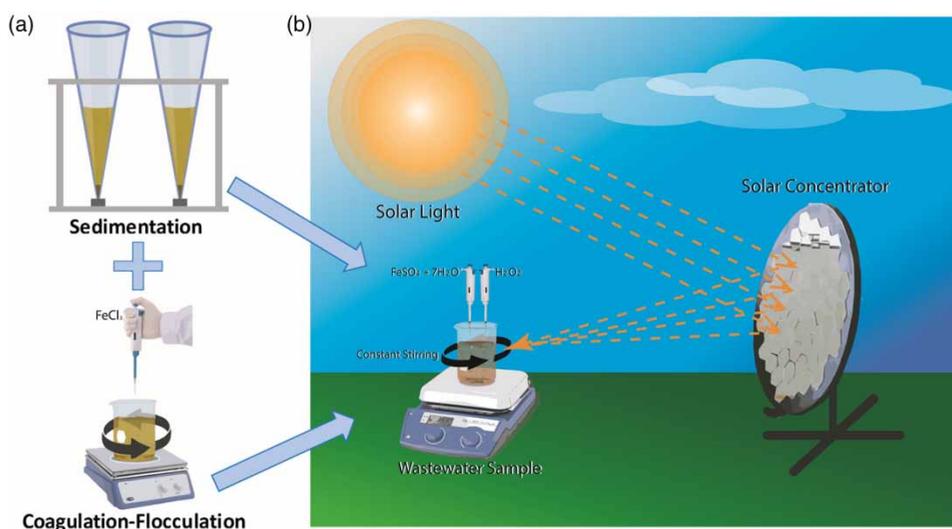


Figure 2 | Schematic diagram of (a) primary treatments applied to samples and (b) solar photo-Fenton system with parabolic concentrator. Please refer to the online version of this paper to see this figure in colour: <http://dx.doi.org/10.2166/wst.2021.438>.

Another part of the clarified material was treated by coagulation–flocculation (Coag-Floc) at the natural pH of the sample (around 8) for 30 min using 0.5 g/L of ferric chloride (FeCl₃, PanReac) and after this last primary treatment (Figure 2(a)) the Fenton and photo-Fenton processes were applied using the doses of the reagents described above and setting the pH to a value of 4.

Solar photochemical treatment was performed at an irradiance between 80 and 85 mW/cm², which is an average of the environmental solar irradiance reported by the Mexico National Weather Service. This process was also assessment using a solar parabolic concentrator (Figure 2(b)), made with reused materials using 57 hexagonal mirrors (5 cm × 5 cm) with a total reflective surface of 4,275 cm². An image of this solar concentrator device is shown in Figure 3, in which the arrangement of the mirrors on the reflector plate can be observed.

In order to corroborate the power of solar radiation per unit area that is emitted towards the samples with direct sunlight and using the solar parabolic concentrator, the irradiance was measured with a photo-radiometer (DeltaOHM, Mod.HD-2122.1) provided with a sensor in the spectral region 400 to 1,050 nm (LP-472 RAD).

After each test, the characterization of the treated effluent was carried out. It is important to note that all the analyzes of the studied samples were carried out in duplicate to ensure the behaviour of the experimental data.

ANALYSIS AND RESULTS

As wastewater comes from a biological reactor, it implies that the amount of biomass increases and that increases the amount of organic matter, solids, and other water quality parameters. This was confirmed by the data in Table 2, where the RWW

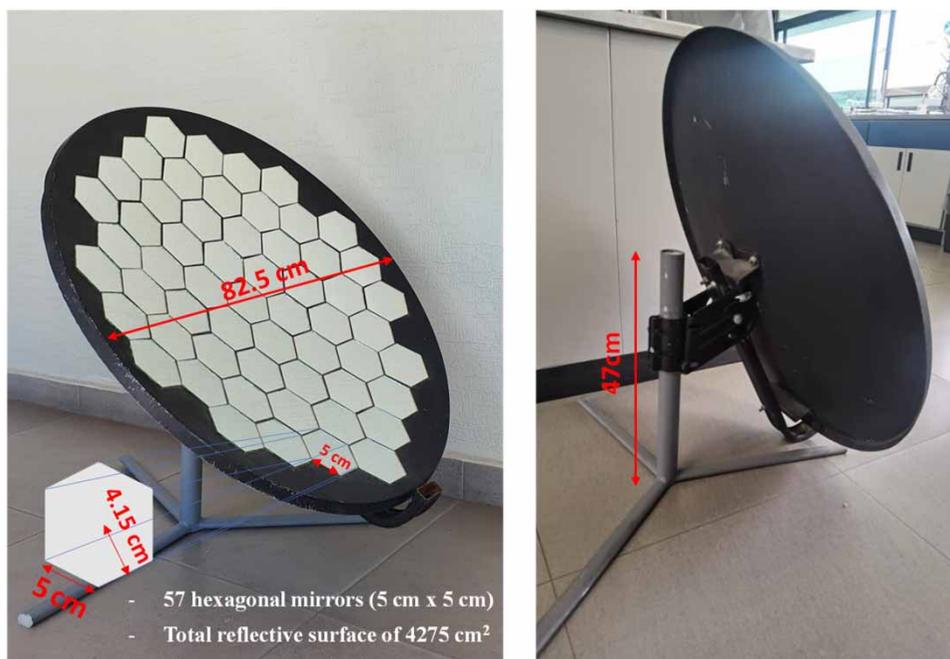


Figure 3 | Solar parabolic concentrator with the dimensions of (left) the mirrors arranged on the reflector plate and (right) the concentrator support. Please refer to the online version of this paper to see this figure in colour: <http://dx.doi.org/10.2166/wst.2021.438>.

Table 2 | Physicochemical characteristics of the raw sample; samples with primary treatment (sedimentation and coagulation–flocculation); and effluents from the primary treatment treated via photo-Fenton with direct sunlight and with solar concentrator

Parameter	Raw sample	Sedim + Coag-Floc	Sedim + Coag-Floc + Solar Photo-Fenton	Sedim + Coag-Floc + Solar Conc. Photo-Fenton
COD (g/L)	24.00 ± 5.66	6.00 ± 2.83	0.40 ± 0	0.36 ± 0
TSS (g/L)	4.72	1.68	0.32	0.43
Settleable solids (mL/L)	7.78	65.71	120.00	120.00
pH	8.26 ± 0.01	8.19 ± 0.09	3.18 ± 0.04	3.19 ± 0.02
Conductivity (µS/cm)	930.5 ± 62.23	6.50 ± 0.99	3.54 ± 0.10	4.05 ± 0.35
DOC (g/L)	1.75	0.29	0.25	0.20

contains concentrations of COD above 20 g/L, TSS above 4.0 g/L and more than 1.5 g/L of DOC, when generally, slaughterhouse wastewater has an approximate average of 4.20 g/L COD, 1.20 g/L TSS and 0.55 g/L DOC (Bustillo-Lecompte & Mehrvar 2015). Therefore, in this project a wastewater more complex than common characteristics of a wastewater that comes directly from a slaughterhouse was treated.

The results of the different treatment stages applied to the slaughterhouse wastewater in terms of the COD and TSS removal are depicted in Figure 4. Figure 4(a) shows that sedimentation reached 16% of COD removal and 54% of TSS reduction, obtaining a less complex sample than would have undergone treatment via AOPs. These results show the importance of applying a pretreatment to the slaughterhouse wastewater to settle out the suspended solids, which is a low-cost mechanical separation as no chemical reagents are required and that can represent an advantage when integrating a wastewater treatment flow with sustainable processes such as the use of energy from the wind or solar (Giorgi *et al.* 2018).

After sedimentation the samples treated with Fenton and photo-Fenton processes reached more than 96% of COD removal and more than 82% of TSS removal, with either of both processes. Thereby, it is noted that AOPs allow the oxidation of pollutants present in samples. In terms of percentage of organic matter removal, the process with the solar concentrator does not lead to a significant improvement over the process without light, however 1% difference in COD removal corresponds to an additional reduction of about 300 mg/L of COD.

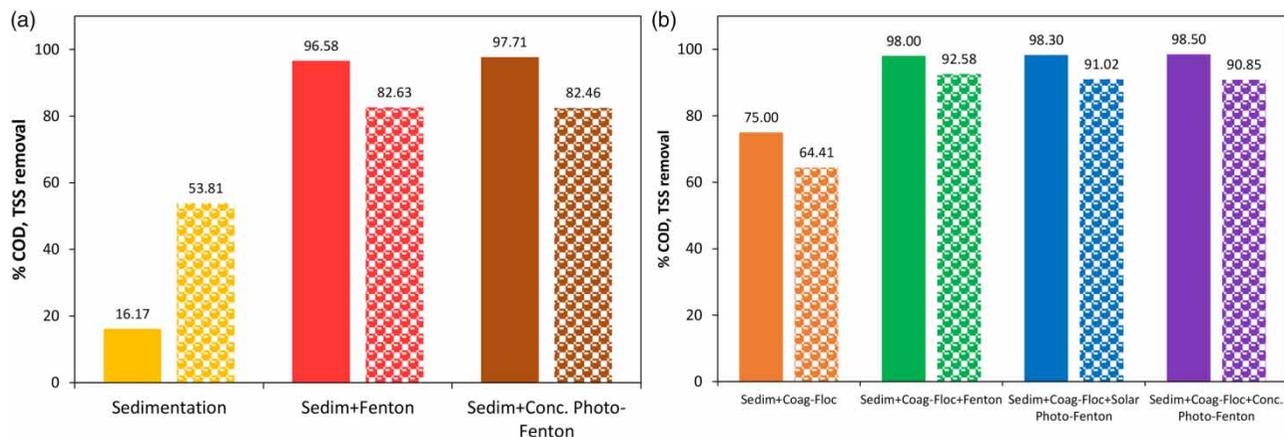


Figure 4 | Comparison of the COD (solid block) and TSS (dotted block) removal in (a) the settled samples treated by Fenton and photo-Fenton and (b) the effluents from sedimentation and coagulation–flocculation treated via Fenton and photo-Fenton processes. Please refer to the online version of this paper to see this figure in colour: <http://dx.doi.org/10.2166/wst.2021.438>.

Figure 4(b) shows the behaviour of the samples to which sedimentation and coagulation–flocculation were applied as primary treatment, where it can be observed that this allowed an effluent to be obtained with 75 and 64% of the COD and TSS removal, respectively. This indicates the effectiveness of the process in improving effluent quality by obtaining around 60% additional removal of COD compared to sedimentation. These results agree well with reports by other authors who indicated that the coagulation using ferrous ion reaches the maximum efficiency at pH between 8 and 11, because in these pH ranges ferrous ions mainly exist in the form of $\text{Fe}(\text{OH})_2$ and show effectiveness in treatment (Ha & Huong 2017; Gökçek & Özdemir 2020).

The Fenton treatment to which this effluent was subjected achieved a 98% COD reduction and 93% TSS removal. These results indicated that coagulation–flocculation has a greater influence on the elimination of suspended solids than on the COD elimination, since an additional 10% removal of this parameter was achieved when comparing the values obtained with the sample only previously settled.

Regarding solar photo-Fenton test of the pre-treated samples, applying direct sunlight a 98% COD removal was achieved, while using the solar concentrator the COD was reduced up to 99%, not obtaining improvement in the additional elimination of TSS (up to 91%).

Figure 5(a) shows the behaviour of the elimination of dissolved solids contained in the treated samples. The data in this figure revealed that the coagulation–flocculation prior to the application of AOP leads to the highest production of TDS (more than 6 mg/L). However, the solar concentrator allowed the concentration of dissolved solids, which were less than this range, although this value is greater than at the initial concentration presented in the raw sample. This can be seen due to remnants of ionized substances resulting from the reagents used in coagulation–flocculation and can be corroborated with the conductivity values that are explained later.

The experiment carried out with the solar concentrator reached an elimination of 120 mg/L more of COD compared to the process without light (Fenton), this improvement can be attributed to the photochemical cleavage of H_2O_2 by solar light absorption resulting in an increase rate of production HO^\bullet (Atallah *et al.* 2016).

Conversely, in Figure 5(b) the generation of settleable solids is presented, where it can be observed that the more complete combined processes led to the highest generation of residual sludge. These settleable solids were measured in the last process of the applied treatment flow, which may be due to the amount (remnants) as well as the type of reagent used, in addition to the greater transformation of organic matter that occurred in these processes (Gutierrez-Mata *et al.* 2017).

It is important to remark that solar catalytic treatments were carried out under natural conditions, so monitoring of the irradiance of the environment and the temperature of the sample were measured. In Figure 6 the behaviour of these variables are depicted for the different experiments performed. In this figure, it can be seen that the irradiance is not constant during the tests. However, the short experimental period (30 min) allows working in ranges that are not so discordance.

It should be pointed out that the temperature increases around 5 °C in the photo-Fenton treatment with direct sunlight. Conversely, the photochemical treatment under sunlight using the concentrator leads to an increase of around 15 °C at

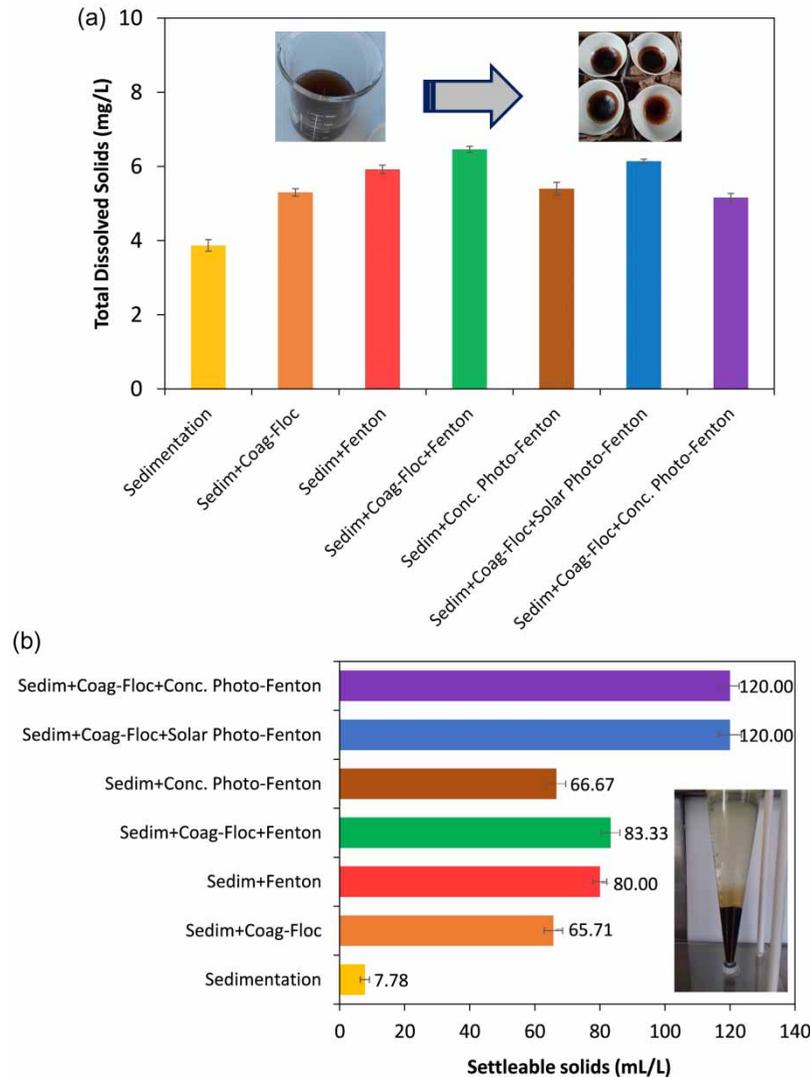


Figure 5 | Comparison of (a) total dissolved solids concentration and (b) settleable solids production of the samples of slaughterhouse wastewater treated under different applied processes. Please refer to the online version of this paper to see this figure in colour: <http://dx.doi.org/10.2166/wst.2021.438>.

the end of the process. Although during the tests there were decreases in irradiance, the temperature of the samples continued to increase, which translates into the adsorption of heat from the treated effluent.

The comparison of the physicochemical properties of the slaughterhouse effluent from the biological reactor and of the treated samples using two and three processes are presented in Table 2.

Table 2 shows that applying the sedimentation and coagulation to the slaughterhouse samples decreases four-fold the COD and around three-fold the TSS in comparison with the raw sample. In spite of more settleable solids being generated applying a solar photo-Fenton compared to pre-treated samples, the photochemical catalysis allows a decrease of COD and TSS to smaller values of 0.40 g/L and 0.43 g/L of the COD and the TSS, respectively. The photo-Fenton treatment with a solar concentrator provided the best results in terms of COD removal (99%) and mineralization (89%) of the samples.

The final pH of the samples that received some chemical treatment showed a slight decrease, this change has also been reported by Imran *et al.* (2012) who explain that this change in this parameter may have been due to the removal of some suspended and dissolved compounds from wastewater, which agrees with the TSS results shown in Table 2.

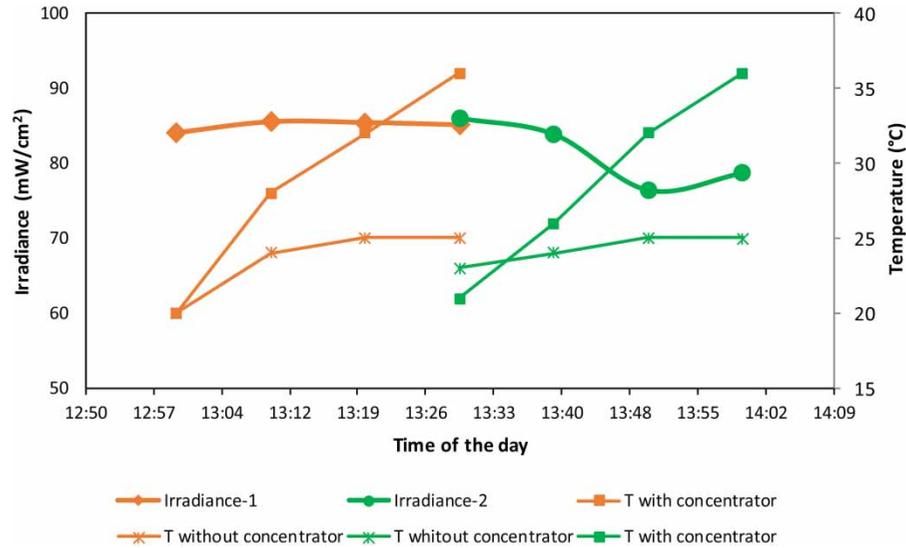


Figure 6 | Behaviour of environmental solar irradiance and temperature of samples treated by photochemical processes. Please refer to the online version of this paper to see this figure in colour: <http://dx.doi.org/10.2166/wst.2021.438>.

Figure 7 shows the aspects that presented the RWW sample and the treated sample via sedimentation + coagulation–flocculation + solar photo-Fenton with the concentrator (TWW-SPC). The improvement in the quality of the treated water with respect to the initial appearance is visually evident. This is confirmed with the characterization carried out on the sample.

Finally, in Figure 8 is shown an image of the experimentation of the photo-Fenton tests under direct sunlight and using the parabolic concentrator. In this figure it can be seen that using the concentrator promotes greater illumination of the sample exposed to sunlight. An additional experiment from our research group corroborated this behaviour by measuring irradiance on a clear day using a photo-radiometer, in which the results indicate that the solar concentrator promotes a 47% increase in the irradiance that reaches the sample compared to that illuminated by direct sunlight (185.18 mW/cm² vs 98.14 mW/cm²).

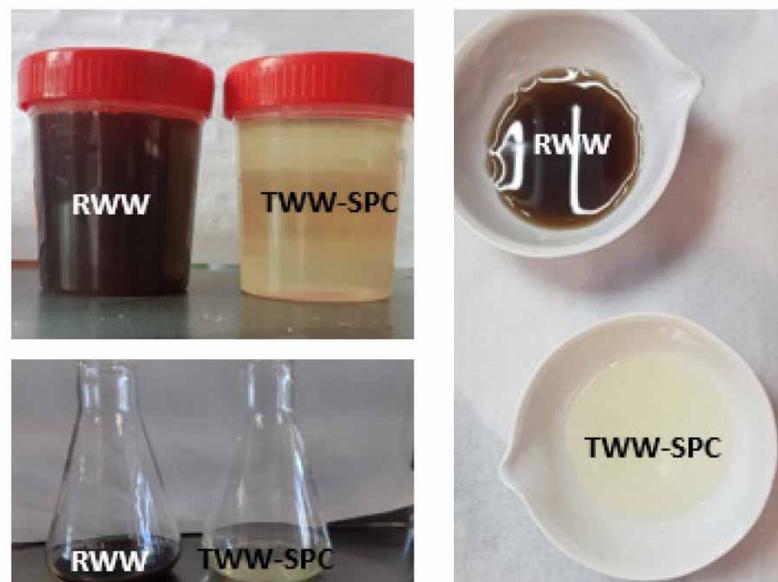


Figure 7 | Appearance of the raw sample from slaughterhouse and the effluent with the best applied treatment. Please refer to the online version of this paper to see this figure in colour: <http://dx.doi.org/10.2166/wst.2021.438>.



Figure 8 | Image of the experimentation of the photo-Fenton tests under direct sunlight and using the solar parabolic concentrator. Please refer to the online version of this paper to see this figure in colour: <http://dx.doi.org/10.2166/wst.2021.438>.

CONCLUSIONS

In this work, it was possible to corroborate the effectiveness results of the treatment of effluents that had undergone biological processes from a municipal slaughterhouse by establishing serial stages. The primary treatments applied to the test sample allowed a 75% COD removal and 64% of the TSS reduction. With the above, the use of these previous stages to remove high solids content from samples is confirmed.

Applying the solar photo-Fenton process to the pre-treated samples it was possible to achieve up to 99% of the COD elimination, 91% of TSS removal and 89% of mineralization, which reflects the effect of the parabolic concentrator in improving the process, representing a sustainable treatment alternative. This is explained because with the photochemical process the generation of additional HO^\cdot radicals led to more oxidation of pollutants present in the wastewater samples.

In addition, the improvement in terms of the total dissolved solids concentration and the settleable solids production was evidenced, with respect to the samples treated photochemically without the solar concentrator. The high percentage of mineralization obtained (close to 90%) motivates a more detailed characterization of the treated effluent to establish an alternative to reuse the water from the best treatment.

It is important to control the temperature, especially if it is necessary to extend the catalytic processes with sunlight for a longer time. However, the results achieved so far facilitate the treatment of samples with the characteristics of those studied in short periods.

According to the Mexican regulations on the allowable maximum limits of pollutants in the wastewater discharges to water bodies, the treatment that was closest to regulatory compliance was the treatment flow that integrates the processes of sedimentation + coagulation–flocculation + solar photo-Fenton with the concentrator.

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DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

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