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Assessing the validity of solar membrane distillation for disinfection of contaminated water

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ABSTRACT

Together with desalination, reuse of wastewater is considered as a solution to mitigate the water deficit in many areas. Membrane distillation (MD) is a technology increasingly proposed for desalination and water treatment. The process is supposed to produce pure distillate, and this is what this paper evaluates. Biologically contaminated water was used in a commercial prototype of spiral-wound liquid-gap MD coupled with solar thermal energy to assess the suitability of the process for removing pathogens. Tests were done during several hours for different operating conditions. The produced distillate, the rejected concentrated solution and the feed water were monitored through time and samples were taken for microbiological analysis. Results proved the efficiency of solar MD to produce a contaminant-free distillate when *Escherichia coli*, *Fusarium solani* and *Clostridium* sp spores were present in the feed water. Furthermore, in the first two cases the population of biological contaminants in the concentrated decreased below the detection limit during the experiments. However, in the case of *Clostridium* sp spores, these were not totally inactivated in the concentrated solution. Therefore, it was necessary to apply a post treatment before reclaiming the concentrated solution. The technology used was a photo-Fenton process carried out in a compound parabolic collector reactor. The combination of MD and photo-Fenton achieved complete abatement of *Clostridium* sp. spores, which was not accomplished in previous experiments when photocatalysis alone was applied to wastewater.

Keywords: Solar water treatment; Membrane distillation; Solar disinfection

1. Introduction

The scarcity of freshwater is a global problem that requires finding new sources of supply. The increase in the global scarcity of water is caused by climate change and the increased demand for freshwater due to the growth in industrial, agricultural and recreational activities. The agricultural sector is the largest consumer of freshwater, using 70–95% for irrigation [1]. Over 97.5% of the total volume of water on Earth

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is salt water. So, desalination is a solution to provide freshwater in dry areas with access to brackish resources and/or seawater. Many researchers have focused on developing a desalination process that uses regenerative energy sources. Solar desalination is an ever-growing alternative to produce freshwater in areas with water deficit and high solar irradiation. Another source of freshwater can be the reuse of wastewater. Wastewater can contain a great diversity of chemical contaminants and pathogens which must be removed or transformed into harmless compounds. Amongst the more abundant pathogens in urban wastewater are faecal bacteria like *Escherichia coli*, with a concentration that can be of 10^3–10^5 total coliforms per 100 ml [1]. More harmful pathogens can be responsible for waterborne infectious diseases, like *Cryptosporidium* sp. Other pathogens present in wastewater can be problematic if reusing wastewater for irrigation, like *Fusarium* sp. These pathogens need to be eliminated before wastewater reuse. Advanced oxidation processes (AOPs) have been demonstrated to be reliable for wastewater treatment due to their high capacity to oxidise almost all organic contaminants. However, the low concentration of pathogens in the water to be treated can make these processes slow and low efficient.

Membrane distillation (MD) is a thermally driven separation process in which only water vapour or other volatile molecules are transported through a hydrophobic porous membrane. When a temperature difference is created between both sides of the membrane, a vapour pressure difference appears, constituting the driving force of the process. Evaporation takes place at the hot side, vapour is transported through the membrane and its condensation as distilled water occurs on the cold side. Due to the hydrophobicity of the membrane, the liquid phase does not penetrate the pores, as long as its hydrostatic pressure does not exceed the minimum entry pressure of the porous membrane (the so-called Liquid Entry Pressure). Therefore, membranes have a high rejection rate for the non-volatile solutes, such as ions, macromolecules, colloids, and microorganisms, obtaining almost pure water [2]. The main applications of MD are: production of water of high quality from saltwater; concentration/removing of non- volatile compounds of aqueous solutions such as juices, whey or alcoholic solutions; and removing volatile compounds or microorganisms from wastewater. However, most of these applications have been tested only at laboratory scale [3]. Nowadays, there are some MD pilot plants developed mainly to desalinate salt water [4], although some research centres are starting to treat wastewater with MD [5–9]. Other filtration technologies are commonly used to concentrate wastewater for easier post-treatment [10] before reusing it or discharging to the environment. There are several problems when these technologies are applied to wastewater. One is fouling, which imposes high maintenance costs, with chemical cleaning of the membrane and even its replacement. Another is the fact that the production of permeate is not completely free of some contaminants (such as Boron, Arsenic, etc.). MD has the advantage that the hydrophobicity of the membrane protects it from fouling. Moreover, the distillate is theoretically pure water.

Plataforma Solar de Almería (PSA) is working on solar MD at pilot plant scale. So far, the research has been focused on analysing the performance of different commercial prototypes in desalination. The present study is focused on further application of MD, namely, treating contaminated water to obtain an effluent free of microorganisms and a concentrate for easier elimination. Theoretically, the MD process should produce pure distillate without pathogens. However, this has not been proved at pilot scale. Commercial prototypes use real membranes with a nominal pore size but they do not guarantee a uniform distribution of them. Pore wetting and even leakage have been observed in some modules [11,12], which could contaminate the distillate.

The aim of this study is to assess whether real-size modules of MD can be used to treat urban wastewater with biological contaminants. The goals are to check whether pathogens can be kept out of the distillate, and whether the MD process can have a positive influence on wastewater treatment. The experiments have been done with a commercial module from Solar Spring GmbH. Three different pathogens have been chosen to analyse this: *E. coli*, *Fusarium solani* and *Clostridium* sp. *E. coli* usually accounts for the majority of the faecal coliform group. The concentration established by WHO for reusing treated water with these coliforms is less than or equal to 1000CFU of faecal coliforms/100 ml [1]. *Fusarium* is a model pathogen due to its high resistance to standard disinfection and its harmful effect over plants and even humans. *Fusarium* is a fungus found in soil that affects a wide variety of crops, and it can be spread through the water. The agriculture in Almeria, a province of the southeast of Spain, accounts for an important percentage of the economy. Water from the Andarax River is used. *Fusarium* is a problem for the crops in this area [13] and causes river contamination which ultimately affects the Mediterranean Sea, which is widely used for desalination (as a matter of fact, the desalination plant of the city of Almería is located very near the river estuary). *Clostridium* sp. is a genus of anaerobic

and Gram-positive bacterium which is capable of reducing sulphite and lives in different habitats such as soil, water and intestinal tract of humans and animals. This pathogen produces spores highly resistant to heat, chemical and desiccation [14]. Spores of sulphite-reducing clostridia are used as indicators of oocysts of *Cryptosporidium* sp. [15]. Under specific environmental conditions, this latter genus can cause waterborne infectious diseases.

2. Material and methods

2.1. MD module

The module studied is a spiral-wound module based on liquid-gap MD. It was designed by the German research institute Fraunhofer ISE and is marketed by Solar Spring as part of the Oryx 150 desalination unit [16]. The module contains three channels: the evaporator channel, the condenser channel and the distillate channel (see Fig. 1). The operation of the module is as follows: firstly, feed water from the feed water tank (475 l) is pumped into the condenser channel circulating previously through a cartridge filter (300 μm). In this channel, the temperature of the water is increased by the internal heat recovery and then it flows through the heat exchanger in counter-current flow with hot water from the heat source. Then, this water flows through the evaporator channel where evaporation takes place and the concentrated solution is returned to the feed tank. Vapour passes through the membrane, condenses in the condenser foil and is collected outside the module. This pilot unit has an automated system for refilling the feed tank with cooler water as its level decreases during the operation. In order to maintain the concentration of contaminants in the feed tank, this system was deactivated. As a result of this, the temperature of the feed tank was increasing throughout the operation due to the residual heat carried by the rejected water from the module.

The membrane has a total surface of 10 m^2 with a nominal pore size of 0.2 μm, a porosity of 80% and a thickness of 70 μm. The different compounds of the module are made of thermoplastic materials, more specifically, the membrane is of Teflon (PTFE), the condenser foil of ethylene tetrafluoroethylene, the spacers of low-density polyethylene and the shell consists of glass-reinforced plastic [17]. Due to the thermal resistance of the materials, the temperature in the evaporator channel should not be higher than 85°C.

2.2. Test-bed facilities

The necessary thermal energy to achieve the temperature difference inside the module was provided by a field of stationary solar thermal collectors. The installation is described elsewhere [4]. All the temperatures, flow rates and pressures were monitored and controlled by a Supervisory Control And Data Acquisition connected through a Programmable Logic Controller. The monitoring system of the Oryx 150 unit was enhanced with further measurements of

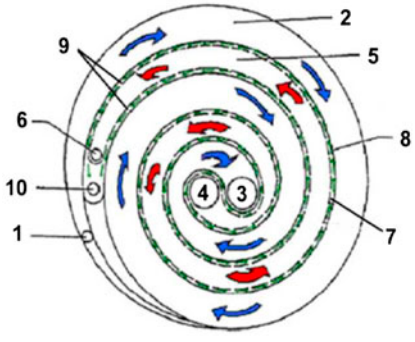

Fig. 1. Spiral-wound module Oryx 150: (1) condenser inlet, (2) condenser channel, (3) condenser outlet, (4) evaporator inlet (5) evaporator channel, (6) evaporator outlet, (7) membrane, (8) condenser foil, (9) distillate channel, and (10) distillate outlet (adapted from [17]).

Fig. 2. Oryx 150 unit at the facilities of PSA.

temperature. Fig. 2 shows an image of the Oryx 150 unit at PSA.

2.3. Indicator detection and quantification of microorganisms

In this study, three different pathogens were analysed. For these experiments, *E. coli* strain K12 (CECT 4624) and *F. solani* strain (CECT 20831) were acquired from the Spanish Culture Collection. To prepare liquid culture of *E. coli,* 200 ml of Luria-Bertani nutrient medium (LB Broth, Panreac) was divided into 14 bottles and one colony of *E. coli* from stocks were added to each one. Next, the bottles were incubated at 37˚C with rotatory shaking during 24 h. Bacterial suspensions were centrifuged at 3,000 rpm for 10 min. Then, the supernatant was retired and the pellet was re-suspended in phosphate-buffered saline solution (PBS). Finally, the bacterial suspension was dissolved into the feed tank of the MD unit, achieving an initial concentration of 10^6 CFU/ml approximately. The enumeration of the colonies of *E. coli* was done using the standard plate counting method, through serial threefold dilutions in PBS. Sixty microlitres of each dilution of feed and concentrate were plated on Luria-Bertani agar (LB Agar, Panreac), and distillate on Chromo-Cult® Coliform Agar (Merck KGaA, Darmstadt, Germany). The colonies were quantified after incubation at 37˚C for 24 h. The detection limit (DL) of this method was 2 CFU/ml.

F. solani colonies were transferred to the sporulation medium with KCl in Petri dishes exposed to UV-C radiation for 15 d at 25˚C. Spores concentration generated was 10^5 CFU/plate approximately. The spores were separated from the agar by washing the plate with 10 ml of demineralized sterile water obtaining the stock solution. Then, the solution was diluted in the MD feed tank to obtain an initial concentration of 10^3 CFU/ml. Sample volumes of 250 and 500 µl, respectively, were plated on acidified malt agar (Panreac) and the colonies were counted after 2 d of incubation at 28˚C. DL of this method was also 2 CFU/ml.

The experiments with *Clostridium* sp. were performed using real municipal wastewater treatment plant effluents. These had a natural presence of these spores with a concentration of 10^6 CFU/100 ml. For counting the colonies of spores of *Clostridium* sp., the medium sulphite polymisin sulphadiazine (cultimed, Panreac) agar was fused and the samples were heated in a water bath to 80˚C. When both were approximately 60˚C, 20 ml of the medium and 20 ml of the sample were mixed and homogenised. Finally, the mixture was incubated at 44˚C under anaerobic conditions for 24 h. The DL of this method was 2 CFU/100 ml.

2.4. Water types

For the experiments with *E. coli* and *F. solani*, demineralized water obtained from tap water treated with RO and electrodeionization was used. This water had a conductivity of 44.6 ± 0.2 µS/cm, a pH of 6.6 ± 0.1 and a turbidity of 0.11 ± 0.01 NTU. The Total Carbon (TC) was 0.81 ± 0.02 mg/l and Inorganic Carbon (IC) was 0.56 ± 0.01 mg/l. The concentration of ions was lower than the DL of the equipment (0.1 mg/l). The wastewater used for the experiments with *Clostridium* sp. was collected from a municipal wastewater treatment plant, El Bobar (Almeria, Spain). This is a wastewater treatment plant consisting of a pre-treatment, a first treatment and a secondary treatment. In the pre-treatment, the water is subjected to a grinding process, sanding and degreasing. Then, in the primary treatment, the water is stored in a tank, where solids are decanted. Finally, the secondary treatment consisted of biological treatment by activated sludge followed by a decanting. At PSA, water from the secondary treatment was diluted with demineralized water in a proportion of 1:3. Table 1 shows the main ions and some physicochemical characteristics. Ionic concentration was measured by ion chromatography using a DX-600 model chromatograph for anions and a DX-120 model for cations. IC and TC were determined

Table 1
Ionic composition and physicochemical characteristics of the real municipal wastewater treatment plant effluents used in the study

Cations (mg/l)	
Na^+	193
NH^{4+}	94
K^+	27
Mg^{2+}	33
Ca^{2+}	62
SO_4^{2-}	123
Anions (mg/l)	
Cl^-	436
NO^{3-}	2
PO_4^{3-}	24
pH	7.53
Conductivity (µs/cm)	1,193
Turbidity (NTU)	11.1
TC (mg/l)	127
IC (mg/l)	102

by a Shimadzu-5050A TOC analyser and turbidity was measured with a turbidimeter Model 2100N.

3. Results

3.1. Tests with E. coli

The concentration of *E. coli* was studied with continuous recirculation of the concentrate at the feed tank of MD in darkness conditions, with and without heat supply. In order to carry out the test, 200 ml of a solution with 10^9 CFU/ml of *E. coli* was spiked in 200 l of demineralized water, resulting in feed water with 10^5 CFU/ml concentration of this microorganism. The feed flow rate for the MD process was 600 l/h and the temperature inside the evaporator channel varied between 55 and 60 °C (Table 2). The feed was recirculated during 120 min and samples of distillate, concentrate and feed were taken every 15 min. The results (Fig. 3) show that the distillate was free of *E. coli* (concentration below the DL) at all time. This suggests that the MD effectively filters out the bacteria. The concentrate rejected by the module was also free of *E. coli* during the whole experiment. *E. coli* inactivation rates can be dependent on stress [18]. An experiment was performed without heat supply in order to evaluate the possible effects of temperature and mechanical stress during the circulation of the feed inside the module (see Section 2.1). As shown in Fig. 4, the concentration of *E. coli* did not decrease more than two orders of magnitude when the feed water was kept at ambient temperature during the circulation through the module. Thermal stress, on the other hand, seems a more plausible explanation for the inactivation. *E. coli* does not resist high temperatures, being its decimal reduction time 4–6 min at 55 °C and 2 min at 60 °C [19]. The temperatures measured in the module are shown in Table 2. Considering that the residence time inside the module for feed flow rate of 600 l/h is roughly around 2 min, the total temperature integral received by the feed water does not seem as large as

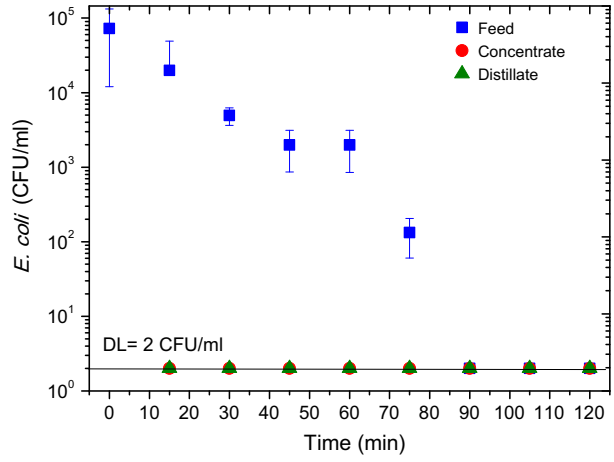

Fig. 3. Concentration of *E. coli* after MD treatment.

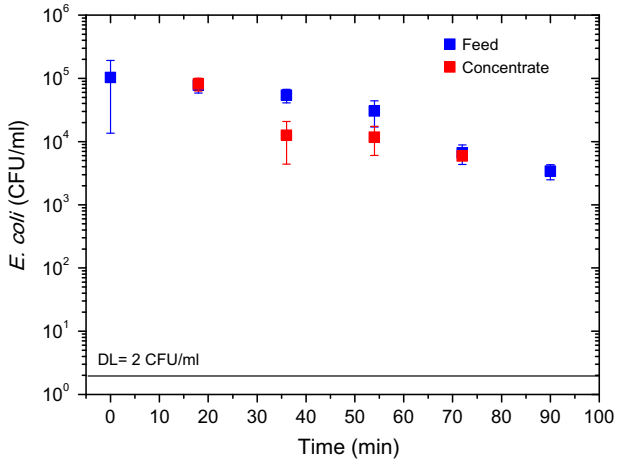

Fig. 4. Concentration of *E. coli* after MD treatment without temperature.

Table 2
Temperatures measured in the MD module during the *E. coli* experiments shown in Fig. 3

Time from start (min)	$T_{cond\ in}$ (°C)	$T_{cond\ out}$ (°C)	$T_{evap\ in}$ (°C)	$T_{evap\ out}$ (°C)
15	32.1	47.8	55.8	33.4
30	34.1	54.0	58.7	37.5
45	36.9	53.8	58.4	39.9
60	39.2	54.9	59.1	42.1
75	41.5	55.8	59.7	44.2
90	43.4	56.7	60.2	45.9
105	44.9	57.5	60.7	47.5
120	46.0	57.7	60.8	48.4

Note: cond: condenser channel; evap: evaporator channel.

the one indicated. However, the fact that full inactivation was reached from the beginning of the experiments suggests that the mechanical stress suffered inside the evaporation channel could make this pathogen more vulnerable to the thermal stress.

The presence of *E. coli* in the feed tank was decreasing with time, reaching complete inactivation after 90 min. This time interval is longer than what it takes to fully recirculate the tank through the module (feed flow rate was 600 l/h for total tank volume of 200 l). The explanation for this is that the Oryx 150 feed tank has a dead volume of about 24 l which is not affected by the feed pump. Therefore, during the recirculation this stagnant volume of contaminated feed water was slowly mixed with the disinfected concentrate returning from the module.

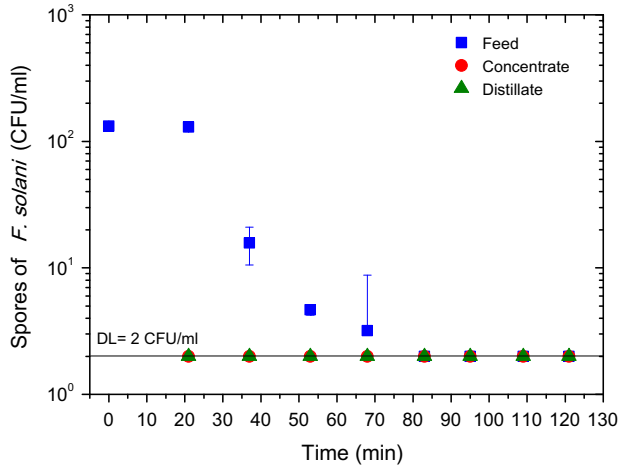

Fig. 5. Concentration of spores of *F. solani* after MD treatment.

3.2. *Tests with* F. solani

The tests with water contaminated by *F. solani* were carried out with recirculation in the same regime as before. The volume of the feed was 200 l with a concentration of the spores of 10^2 CFU/ml. Feed flow rate was 600 l/h and the temperature inside the evaporator channel was kept at 55°C (Table 3). Samples of the feed, concentrate and distillate were taken every 15 min approximately during 120 min. Results are shown in Fig. 5. The distillate was completely free of this microorganism during the whole experiment. The concentrate and feed coming out of the module did not have any culturable spore of *F. solani* either. For the total inactivation of spores of *F. solani* in the feed tank, 80 min of recirculation were necessary. Again, the slow mixing of the disinfected concentrate with the stagnant contaminated water of the dead volume of the feed tank is responsible for the long time duration. However, as in the previous case, an explanation must be found for the inactivation of *F. solani* after circulating through the module. *F. solani* spores are inactivated when kept above temperatures of 50°C during 30 min [20]. For the feed flow rate used, the residence time inside the module was 2 min approximately. As before, it seems that this shorter exposition to higher temperatures (see Table 3) inside the module was enough to inactivate the *F. solani* spores.

3.3. *Tests with* Clostridium *sp.*

In order to perform a more definite study, a new model of pathogen more resistant to temperature was chosen. As explained above, *Clostridium* sp. are very tolerant to high temperatures (a period ranging between 5 and 35 min at least at 90°C is required to inactivate them [19]). In this case, the tests were performed with real municipal wastewater treatment plant effluents with a concentration of 3.4×10^2 CFU/100 ml of *Clostridium* sp. This feed water was passed through the module and returned to the feed tank in a closed

Table 3
Temperatures measured in the MD module during the *F. solani* experiments shown in Fig. 5

Time from start (min)	$T_{cond\ in}$ (°C)	$T_{cond\ out}$ (°C)	$T_{evap\ in}$ (°C)	$T_{evap\ out}$ (°C)
21	24.8	45.2	52.7	28.0
37	28.0	50.3	56.3	32.3
53	31.3	51.3	56.9	35.3
68	34.0	52.6	57.8	37.8
83	36.3	53.8	58.4	39.9
95	37.0	54.3	58.6	40.6
109	38.8	53.8	57.2	41.9
121	40.1	53.7	57.0	43.0

Note: cond: condenser channel; evap: evaporator channel.

circulation which lasted for 240 min. The distillate produced was collected in the distillate tank. The feed flow rate used was 600 l/h to guarantee the maximum distillate production and the maximum temperatures in the module were increasing from 47 to 76 ℃ during the test (Table 4). Samples of distillate, concentrate and feed were taken every 30 min. Fig. 6 shows the results obtained in this test. The produced distillate was completely free of these harmful and resistant pathogens all along the operation. Unlike in the previous tests, *Clostridium* sp. was not inactivated inside the feed channel, so its population in the feed water tank only decreased marginally during the experiment, being its final concentration 90 CFU/100 ml. This suggests that even though this pathogen is resistant to higher temperatures than the other two, mechanical stress (and to a lesser degree in this case, thermal stress) could have made a selection and decreased its population after circulating through the module. Before discharging to the environment or reusing this water, a further post-treatment is required. Previous tests carried out with AOPs, more specifically, with photo-Fenton at different pH, 3 and 8, did not achieve a complete removal of this pathogen [15]. In this case, a photo-Fenton treatment was applied to the contaminated water remaining in the feed tank after the MD process. Sixty litres of that water were treated in a compound parabolic collector (CPC)-reactor using solar radiation with 10 mg/l of iron and doses of 20 mg/l of H_2O_2 added when this was consumed (total amount of 140 mg/l). The pH was adjusted to 4 to favour the presence of dissolved iron and the removal of carbonates and bicarbonates that reduce the efficiency of the process. The concentration of *Clostridium* sp. decreased from 90 CFU/100 ml to below the detection level of 2 CFU/100 ml in 240 min (Fig. 7).

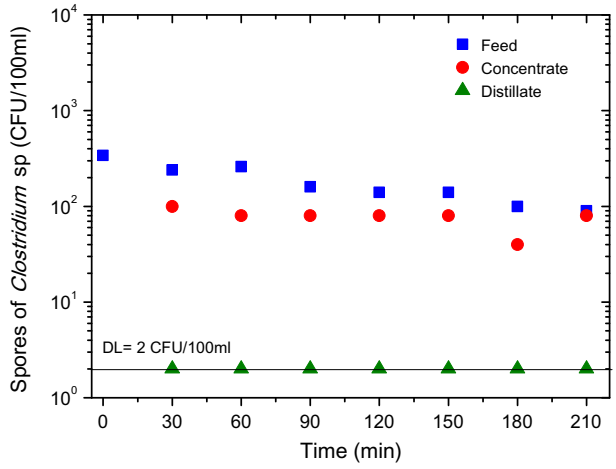

Fig. 6. Concentration of spores of *Clostridium* sp. after MD treatment.

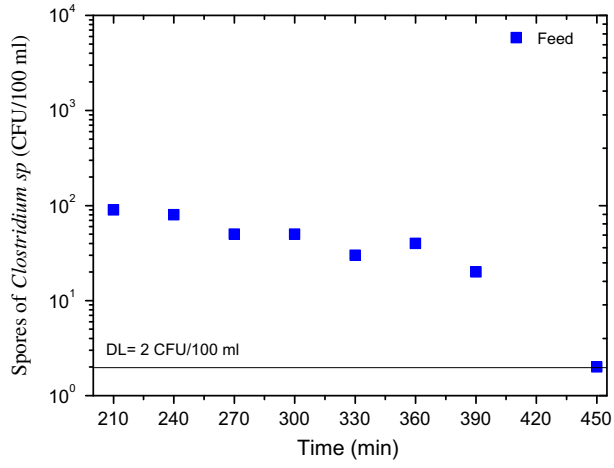

Fig. 7. Concentration of spores of *Clostridium* sp. during a photo-Fenton process in solar CPC reactor.

Table 4
Temperatures measured in the MD module during the *Clostridium* sp. experiments shown in Fig. 6

Time from start (min)	$T_{cond\ in}$ (℃)	$T_{cond\ out}$ (℃)	$T_{evap\ in}$ (℃)	$T_{evap\ out}$ (℃)
30	14.7	47.9	58.3	23.1
60	19.9	54.2	64.3	29.1
90	25.2	60.6	70.6	34.6
120	30.0	66.5	75.8	39.6
150	34.4	66.5	74.3	43.1
180	38.0	68.4	75.8	46.3
210	40.5	68.4	74.7	48.5

Note: cond: condenser channel; evap: evaporator channel.

4. Conclusions

This work demonstrates for the first time the efficiency of a MD system to remove *E. coli*, *F. solani* and spores of sulphite-reducing *Clostridium* from contaminated water. A pure distillate was obtained in all cases. *E. coli* and *F. solani* pathogens were also inactivated in the feed water after circulating through the module due to the temperatures of the process, even though the operation was at minimum temperature level (hardly exceeding 60˚C). On the other hand, MD was demonstrated to be a very efficient pre-treatment to wastewater contaminated with spores of *Clostridium* sp. The combination of mechanical and thermal stress suffered inside the MD module and the attack of hydroxils radical in the photo-Fenton process achieved the complete elimination of spores of sulphite-reducing *clostridium* in municipal wastewater treatment plant effluent. This is an important result, since it achieves for the first time the total removal of this pathogen with the photo-Fenton process. More importantly, this result adds to the production, from real municipal wastewater treatment plant effluent, of a distillate completely free of pathogens which could be directly reused.

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