Solar water disinfection studies with supported TiO_2 and polymer-supported Ru(II) sensitizer in a compound parabolic collector

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Abstract

Solar water disinfection was performed by using TiO_2 and Ru(II) complex as fixed catalysts implemented in a compound parabolic collector (CPC) photoreactor. Studies were performed in the laboratory as well as at a greenfield site. Under laboratory conditions, natural water contaminated with cultured bacteria was photocatalytically treated and the influence of the photolysis as well of both catalysts was studied. Experiments were performed with contaminated water flowing at 12 L/min, under these conditions, photocatalytic experiments performed with Ahlstrom catalyst showed it to be effective in degrading bacteria in water; the Ru-complex catalyst, however did not show clear evidence for disinfecting water and its efficiency was comparable to the photolysis. Under on-site experiments, bacteria contaminated water from the Yaurisque river at Cusco was treated. As a general trend, after photocatalytic treatment a reduction in the *E-coli* population present in water, was observed. Whenever disinfection was achieved in the experiments, no regrowth of bacteria was observed after 24 h. However, a reduction of the prototype efficiency was observed both in laboratory and on-site experiments. This was ascribed to aging of the photocatalyst as well as due to the deposition of particles onto its surface. In cases in which incomplete disinfection resulted, a low rate of *E-coli* growth was observed 24 h after terminating the experiment. However, pseudomones seems to be resistant to the treatment.

1. Introduction

Water disinfection in isolated rural areas in Latin America is a difficult task since operational expenses must be necessarily low. For this reason, several alternative low-cost effective methods to remove bacteria to the widespread chlorination, have been considered for domestic drinking water in rural areas. The well-known advanced photochemical oxidation (APO) process, which is based on the production of highly oxidizing species, primarily the hydroxyl radical ($^{\bullet}OH$) and in some cases singlet oxygen, by photochemical reactions has been shown to be effective in treating contaminated water and air [1]. Sunlight can be used to induce the photochemical reactions for the APO process. Hence, solar photocatalysis is becoming increasingly important due to its essential role in the potential solution of many of today's environmental and energy-source problems. The sensitized APO process can be broadly categorized as dye-sensitized and heterogeneous photocatalysis, and does not require the addition of consumable chemicals and does not produce hazardous waste products.

The heterogeneous photocatalysis is based on the production of hydroxyl radicals when a catalytic semiconductor powder, e.g. TiO_2, is photoexcited with near UV radiation ($\lambda < 390$ nm) in presence of water. Illumination of TiO_2 with photons of energies greater than the band gap energy promotes electrons from the valence band to the conduction band, producing electron/hole pairs [2]. The energy level of the lowest occupied state in the valence band is enough to promote the generation of hydroxyl radicals. The hydroxyl radical is a powerful oxidizing agent and attacks organic pollutants present at or near the surface of the photocatalyst reaching, under optimum conditions, the complete mineralization of the contaminant. An important limitation of heterogeneous photocatalysis is, however, the need of UV-A light to initiate the catalysis. In a dye-sensitized APO process, visible light is absorbed by a sensitizing dye, which is excited to a higher energy state. The excited dye then transfers some of its excess energy

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to other molecules present in the water stream, producing a chemical reaction. When dissolved oxygen is present, it accepts energy from a sensitizer (e.g. ruthenium(II) complexes with polyazaheterocyclic ligands), the dissolved O_2, is converted to singlet molecular oxygen, $^1O_2$, which, like ($^{\bullet}OH$) is also an effective oxidant [3]. If a photoreactor uses both processes, the dye-sensitized and the heterogeneous photocatalysis, their complementary effect offers substantial advantages for solar water disinfection. Therefore, solar photocatalytic water disinfection could be an alternative technology for water supply in rural areas due to its low cost, easy implementation and the possibility of using the visible and UV-A (310-400 nm) component of the solar radiation.

The SOLWATER reactor prototype was developed to detoxify and disinfect contaminated drinking water in rural areas. Titanium dioxide catalysis and Ru(II) polypyridyl complex will be used to produce hydroxyl radical and singlet oxygen, respectively. This prototype was developed within the framework of a research project supported by the European Commission [4]. Experiments under direct solar radiation were carried out using two SOLWATER reactors, one of these was placed at the Universidad Nacional de Ingenieria (UNI), Lima, and the other at Yaurisque, a rural area in Cuzco, Peru. This paper describes the results obtained on the catalyst efficiencies determined for bacteria disinfection in water. The direction of planned long term studies is also briefly discussed.

2. Experimental

2.1 The SOLWATER Photoreactor

The SOLWATER reactor is based on the idea of synergism of the photocatalysis and photo sensitization processes, and operates in the UVA and visible range of sunlight, respectively [5]. Recently, Ahlstrom © non-woven paper was coated with TiO_2 [6] and successfully used in a solar photo reactor [7,8]. On the other hand, Ru(II) tris-chelate complex has been prepared as singlet oxygen photosensitizer and immobilized on porous silicone; it was successfully used to disinfect water [9,10]. The supported photosensitizer was designed and provided by G. Orellana, Universidad Complutense, Madrid, Spain [11]. The reactor consists of a borosilicate glass tube of 1.8 mm thick and 50 mm outer diameter containing a supported TiO_2 catalyst supported on a cellulose fiber (NW1047 from Ahlstrom ©) or supported photosensitizer, $[Ru(4, 7\text{-diphenyl-}1, 10\text{-phenantroline})_3]Cl_2$ bound to strips of a porous polymeric substrate (see Fig. 1). The photocatalyst or the photosensitizer were placed onto an inner concentric tube (see inset in Fig. 1), leaving an annulus for water circulation. The tubes are placed on a compound parabolic collector (CPC) (AoSol, Portugal, under the license of Ecosystem Environmental Services, Spain). The SOLWATER photoreactor uses CPC technology because it efficiently collects the UV radiation present in the diffuse part of the solar radiation using non-imaging optics [12,13]. For optimal optical efficiency of the solar collector, this concentric configuration must constructed to fulfill that the condition that the quotient between the inner diameter of the glass tube (D_i) and the external diameter of the polypropylene cylindrical bar (d_e) is equal to the refractive index of the fluid (n): $D_i/d_e = n$ [12]. In our case, the water (n = 1.33), the support was chosen to have a 32.8 mm diameter and the supported catalyst thickness of 2 mm, since the glass tube is 46.4 mm in inner diameter. The water was circulated using a centrifugal pump.

The photoreactor used at UNI (Fig. 1), has five borosilicate glass tubes of 1.5 m length in a configuration consisting of three independent systems each pumped with a 12 W electrical pump. Two of these systems are composed of two tubes, and the last is composed of one tube, having a treated volume of water 12 and 10 L, respectively. The systems composed of two tubes were used to test each of the the catalysts (supported TiO_2 catalyst provided by the Ahlstrom, or Ru complex deposited onto a silicone strip), and the other one with only one tube was used to analyze the photolysis. The recirculation rate was 12 L/min.

The SOLWATER photoreactor for on site experiments has four borosilicate glass tubes of 1 m length connected in series. The reactor was located at 13 deg latitude S, 72 deg longitude W. The tilt angle of the receptor surface was fixed at 13 deg facing north. In this case, considering its application in rural areas, the water was recirculated using a centrifugal pump Swiftech MPC 650 12 VDC, powered by a battery connected to a photovoltaic solar panel (ATERSA model A38, 38W). The CPC was covered with a

plane glass in order to avoid dust deposition on the reflectors and glass tubes. The collector surface was 1 m^2, and the first two tubes containing a supported Ru complex and the last two containing the Ahlstrom NW1047 TiO_2 catalyst. The vessel was filled with 14 L of water and the recirculation rate was 12 L/min. At the top of the vessel is placed a cotton filter to ensure the aeration and avoid contamination by dust particles.

The global solar radiation and UV-A component (310-400 nm) was measured with a home made calibrated radiometer and a UDT system coupled to a 268UVA detector, respectively.

Figure 1: a) Scheme of the laboratory reactor. The inset shows a cross section of the irradiation systems.

2.2 Description of water sources

Two different experiments were carried out. Natural water obtained from a well at Carabayllo, Lima was used for UNI experiments. In order to test the efficiency of the solar desinfection, the natural water was artificially contaminated with *Escherichia coli* bacteria ATCC 25922 to 10^5 colony forming units per milliliter (CFU/ml).

For the field test the reactor was located in the Training Center for Development (CECADE) in Yaurisque, Cusco. CECADE is a non government organization currently working in the dissemination of environmentally friendly technologies to the surrounding rural communities. Yaurisque is situated 3300 m above sea level. A study of the mean annual solar radiation at Cusco concludes that each year it receives approximately 5.2 kWh/m^2-day – a level which should ensure the applicability of our solar based disinfection technology. The water was taken from Yaurisque River; its physicochemical characteristics are shown in Table 1.

Table 1: Physical chemical characteristics of the Yaurisque River water.

Physical chemical characteristics	Value
pH	7.8
Total hardness, $CaCO_3$ mg/L	540
Total alkalinity mg/L	150
Total acidity CO_2 mg/L	2.2
Chloride Cl^- mg/L	30
Sulfate SO_4^- mg/L	210
Turbidity NTU	3.0
Conductivity S/cm	620

2.3 Bacteria counting technique

The evaluation of bacteria in water during the experiment was performed using the Oxfam-DelAgua Portable Water Testing Kit. The kit is designed to provide information about the bacteriological quality of water supplies in rural areas where laboratory facilities do not exist. The analysis of water samples for total coliform bacteria is carried out by passing a measured quantity of water through a sterile filter. Any bacteria present in the water are caught in the filter. The filter is then placed onto a paper pad soaked in a liquid growth medium (Luria Broth) which feeds coliform bacteria, but inhibits the growth of any other bacteria caught in the filter. The system after incubation at 37 ^oC for 18 h will give the possibility of counting *E-coli* and *pseudomones*. DelAgua equipment also has provisions to measure the turbidity, temperature, hardness and alkalinity of water.

3. Results

3.1 UNI experiments

The long term stability of the catalyst (TiO_2 paper and Ru stripes) using bacteria contaminated water was investigated in repeated batch experiments. A batch experiment consists of a 4 h solar irradiation at around noon time. The photolysis present in the experiments was also evaluated. The initial *E-coli* concentration was around 10^5 CFU/ml, and a 30 min recirculation in the dark was implemented at the beginning of every experiment. The accumulated *E-coli* population as a function of the accumulated energy for the two catalysts was tested and the photolysis was plotted in Fig. 2. The accumulated energy, Q_{UV}, incident on the reactor is used because it allows easier comparison with other photocatalytic experiments [14]. The bactericidal efficiency

of the supported TiO_2 catalyst (Ahlstrom NW1047) is higher. The bactericidal efficiencies of the supported Ru (II) complex and of the photolysis are similar. This fact apparently contradicts the reported one in [10] in which a positive influence of the Ru (II) complex was observed for the water disinfection: A possible explanation is that the higher flow rate used, 12 L/min compared to 2 L/min in [10], influence the degradation rate. Manjon *et al* [15] demonstrated that one of the main factors that determine the performance of the photosensitizing single oxygen production is the fluid rheology that is related to the water flow rate.

The results of repetitive batch experiments performed with the supported TiO_2 catalyst (Ahlstrom NW1047) are shown in Fig. 3. The bactericidal efficiency of the catalyst decreased as a function of the batch number.

Further, water disinfection is achieved up to sixth batch with the supported TiO_2 catalyst, whereas for the supported Ru (II) complex (Fig. 2) only for the first batch. To understand this behavior, elemental analysis and sample morphology was studied by neutron activation analysis and optical microscopy, for the same sample after different batches. Table 2 shows the weight percentage of TiO_2 obtained before use, and after the 7^{th} and the 18^{th} batch obtained by neutron activation analysis. Clearly, the Ti percentage decreases proportionally to the batch number. This result seems to indicate that the catalyst NW1047 is loosing TiO_2

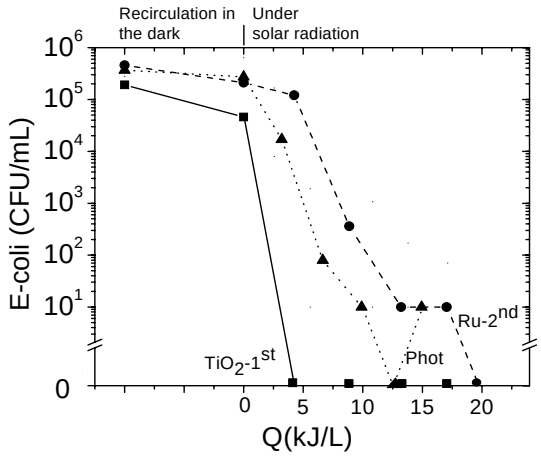

Figure 2: *E-coli* concentration as a function of UV-A integrated energy received by the photocatalytic reactor CPC type (fig. 1). Results for TiO_2 first and ninth use, Ru complex first and seventh use and photolysis are presented.

preferentially from its surface instead of loosing TiO_2-Binder-cellulose in equal proportion: Up to now the reason is unclear however, solar radiation could age the non-woven paper (NW1047) or the water flow could erode the paper coating.

Table 2: Weight percentage of TiO_2 obtained by neutron activation analysis for the Ahlstrom NW1047 catalyst before use and after different batches.

Stage of catalyst use	Weight % of TiO_2
Before use	13.4
7^{th} batch	12.2
18^{th} batch	8.1

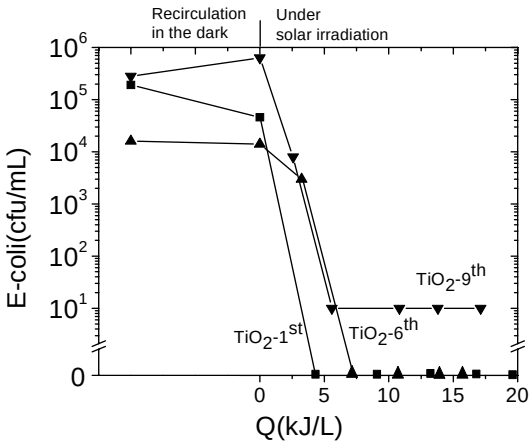

Figure 3: *E-coli* concentration as a function of integrated UV-A received energy by the photocatalytic reactor CPC type (Fig. 1).

The optical micrographs of the supported TiO_2 catalyst before use, and after the 7^{th} and 18^{th} batches are shown in Figure 4. The non-woven paper coated with TiO_2 is produced by interlocking networks of fibers resulting in a porous sheet structure. The paper has two regions, the main network and the interstitial space between the fibers. The micrographs on right side are from the fibers in the network; the micrographs on the left are of the interstitial part between the networks. Poor mechanical stability of the supported TiO_2 catalyst after the 20^{th} batch was noted.

Fig. 5 shows the micrographs of the Ru stripe before use, and after the 4^{th} batch. The Ru stripes, also show some degradation.

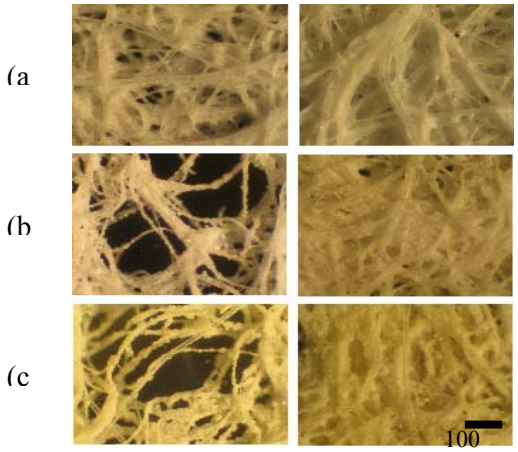

Figure 4: Micrographs of the Ahlstrom NW1047 catalyst: (a) before use, (b) after the 7th batch, and (c) after the 18th batch. Pictures on the right side show a close-ups of the fibers composing the catalyst. Pictures on the left are close-ups of the interstitial part between the fibers that form the catalysts.

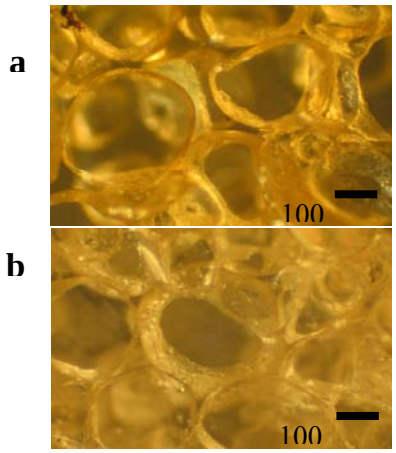

Figure 5: Micrographs of the photosensitizer strip, Ru complex: (a) before use and (b) after the 4th batch.

3.2 On site experiments

Runs of one day duration, in the following termed "Experiment" were routinely performed with the SOLWATER prototype during 14 sunny and partially cloudy days. Measured solar global radiation on these days, integrated to 2.5 and 5 h. are presented in Fig 6. Values could be normalized with the measured ones under laboratory conditions at sea level (see 3.1) by taking into account that the percentage of the UV-A of the solar global radiation is about 5%. This results in an estimate of 25 kJ/L of UV-A average radiation impinging on the CPC reactor per liter unit. In order to standardize these results with the ones obtained in Lima (see 3.1), it is necessary to note that the UV-A radiation is expected to increase at a rate of 10% per thousand meter increase in altitude. Thus, calculating from Lima, situated at sea level to Yaurisque, Cusco (3400 meters above see level) an increment of about 30% is to be expected in the UV-A radiation) [16].

From comparison between the accumulated energies, we point out that the first seven days were partially cloudy whereas the following days were clear.

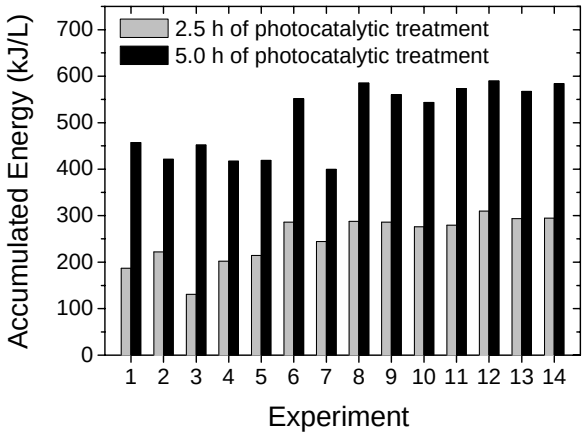

Figure 6: Integrated energy calculated from the measured solar global radiation in Yaurisque. The term "Experiment" in the plot refers to a run of one day duration.

Every day, water for the tests was taken manually from the Yaurisque river, and the photo reactor was not cleaned between runs. Then beginning at around noon solar radiation of ca. 5 h duration, was commenced recirculating the water in the dark for 30 min. The course of the bacterial count with solar irradiation was observed for several experiments (see Figs. 7, and 8).The inactivation of Excherichia coli resulting from solar irradiation in the SOLWATER prototype for several "Experiments" is shown in Fig. 7.

Complete water disinfection was obtained by the fifth run. As the efficiency was decreasing, some *E-coli* colonies remained in the water after the treatment from the sixth run. A similar result was noted in laboratory experiments. (Fig. 2). Bacteria counting was also performed 24 h after the experiment in a sample that was kept in dark and room temperature. The results indicate that the growth of bacteria is affected by the photocatalytic treatment showing a residual post-irradiation effect. This is in agreement

with [17]. In fact, when complete disinfection is achieved using photocatalysis, no bacteria count is observed 24 h after finishing the experiment. As a control, the sample was kept in the dark and measured at the end of the experiment.

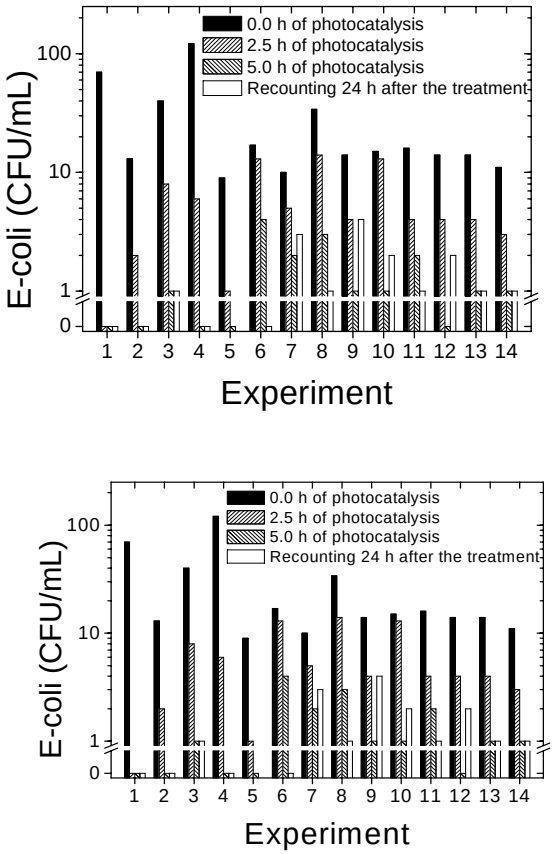

Figure 7: *E-coli* concentration degradation with the photocatalytic reactor. One-day run is expressed as Experiment in the plot. Experiments were performed using water from the Yaurisque river contaminated with bacteria.

The *E-coli* bacteria in water had been degraded by using the SOLWATER prototype and solar radiation. However, during the water treatment, two different kind of *pseudomone* growths were noted (see Figs. 8 and 9). This kind of bacteria seems to be resistant to the photocatalytic treatment and their behavior was erratic. For the case of the *Aeruginose pseudomone* in some tests effective disinfection was achieved (Fig. 8), however in a few tests the *Spp pseudomone* is detected only after the treatment. This suggests that the effect is related to reactor contamination, however further tests are needed to study the behavior of the *pseudomones*. *Pseudomones* are not however considered to be dangerous to humans. Additionally, elemental analysis of the water carried out by neutron activation analysis: before, during and after the water treatment, are shown in Table 3.

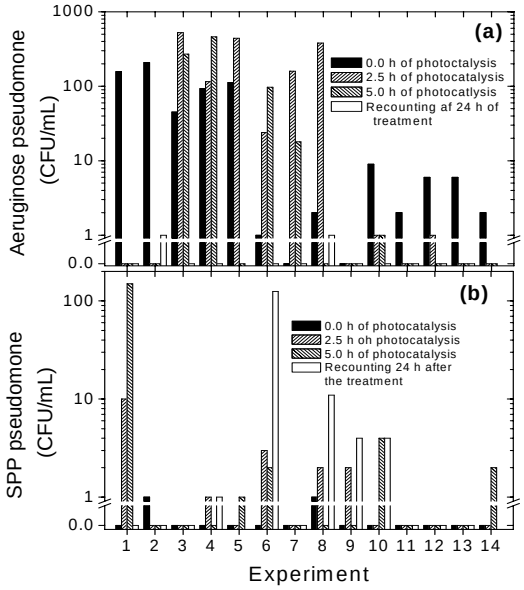

Figure 8: Aeruginose (a) and SPP (b) pseudomone concentration in a long term catalyst use for bacteria degradation. One-day run is expressed as Experiment in the plot. Experiments were performed using water from the Yaurisque river contaminated with bacteria.

As a general trend, the elemental concentration of Ba, Ca, Co, Cd, present in water, decreases as the photocatalytic experiment progresses, whereas Na, Cl and Sr fluctuate. The cause of this effect may be that the TiO_2, which has a conduction band at -0.3V at pH= 0 and 25^{o}C [18-20], could photocatalytically reduce Co with a reduction potential of –0.28 V at pH= 0 and 25^{o}C. Another possibility is Cd, even with its –0.4 V redox potential, (only slightly higher than the energetic position of the conduction band of the TiO_2) has also been reported to be photocatalytically reduced [21]. However in the case of Ba, and Mg, well known strong reduction agents, surface absorption seems to be responsible for the decrement in the water concentration, because an analysis performed by neutron activation analysis of the catalyst after several uses (Table 4), shows an increasing concentration of Al, Ba, Mg, Mn, Sr and V per unit area. This may be the reason for the slow change of the catalyst color to brownish. The fact that the Ti concentration decreases after the first 10 batches and then remains constant while the concentration per unit area of these metals

increases, suggests to us that there is trace adsorption onto the catalyst surface, shadowing and eventually decreasing its efficiency.

Table 3: Trace elements present in Yaurisque River water during a photocatalytic experiment performed on 25 July 2004, using a new Alshtrom paper catalyst. Analysis was carried out by neutron activation analysis and atomic absorption spectroscopy (*).

1. Sample/ 2. Analys used	Ti	Na	Al	Ba	Mg	Mn	Sr	V
new	8260 0± 1800	1890 ± 150	-	-	-	-	-	-
10 d.u	7920 0± 1200	653 ± 82	-	325. 0 ± 7.1	-	112. 0± 7.1	-	66.0 ± 2.8
20 d.u	6989 0± 510	780 ± 220	3100 ± 1100	323 ± 20	-	144.5 ± 2.1	302. ± 35	96.5 ± 3.5
34 d.u	3580 0± 4400	566. 5 ± 3.5	2840 ± 820	327 ± 30	1514 ± 28	149. 0± 4.2	475. 5± 2.1	115. 5± 6.4
45 d.u	547 ± 25	860 ± 140	6690 ± 390	323 ± 13	2920 ± 530	170. 5± 6.4	547 ± 25	151. 5± 6.4

Table 4: Elemental content of the NW1047 after different batches. Experiment performed in Yaurisque using the final SOLWATER prototype. Units are in mg/kg.

Element	Unit	Ower Detec. Limit.	Water sample at the 9:00 h	Water Sample at 12:30 h	Water Sample at 16:00 h
Al	µg /L	< 50	ND	ND	ND
Ag	µg /L	< 5	ND	ND	ND
As	µg/L	< 2	ND	ND	ND
Ba	µg/L		93 ± 36	44.4 ± 7.2	38.4 ± 0.44
Ca	mg/L		156.4 ± 6.30	144.7 ± 6.3	140.6 ± 2.3
*Cd	µg/L		0.035 ± 0.003	0.015 ± 0.003	0.014 ± 0.002
Cl	mg/L		64.1 ± 1.6	60.51 ± 0.82	62.5 ± 3.3
Co	µg/L		0.88 ± 0.26	0.72 ± 0.10	0.65 ± 0.17
Mg	mg/L		6.9 ± 1.5	7.55 ± 0.79	6.2 ± 2.1
Mn	µg/L	< 10	ND	ND	ND
Mo	µg /L	< 10	ND	ND	ND
Na	mg/L		51.7 ± 0.86	49.55 ± 0.28	51.9 ± 3.9
*Pb	µg/L	< 0.05	ND	ND	ND
Sb	µg /L	< 5	ND	ND	ND
Se	µg/L	< 10	ND	ND	ND
Sr	mg/L		1.66	1.37 ± 0.63	1.50 ± 0.22
V	µg/L	< 5	ND	ND	ND

4. Conclusions

A CPC solar photoreactor has been demonstrated to be efficient for bactericidal disinfection by solar photocatalysis with supported TiO_2 and Ru (II) complex, working in series, during treatment periods of 4-5 hours. Bactericidal deactivation by sunlight in a CPC solar collector occurs whether or not the catalyst is present. The total photocatalytic deactivation of pure *E. coli* suspensions is a consequence of the combined effect of sunlight and the oxidant species generated in the supported TiO_2. However, while sunlight alone deactivates *E. coli* suspensions, it does not completely deactivate them, since regrowth of bacteria was detected. The situation is improved when the catalyst was used. The irradiated area in the CPC collector plays a key role in the bacteria inactivation by solar irradiation.

Under our experimental conditions, the disinfection rate is dependent on the catalyst used, being least efficient with increased usage than with a fresh catalyst. Results showed supported TiO_2 to be more efficient than Ru (II) complex. On site tests of the SOLWATER reactor confirmed its capacity to remove bacterial contamination from water. The efficiency was very good for six batches, and limitation of the bacteria growth was observed after the photocatalytic treatment. However, *pseudomones* resisted the treatment.

5. Acknowledgments

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