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Water disinfection by solar photocatalysis using compound parabolic collectors

P. Fernández *, J. Blanco, C. Sichel, S. Malato

Centro de Investigaciones Energéticas, Medioambientales y Tecnológicas (CIEMAT), Plataforma Solar de Almería (PSA), P.O. Box 22, 04200 Tabernas, Almería, Spain

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Abstract

TiO_2 solar photocatalysis has been proven to be a degradation process for aqueous organic contaminant leading to total mineralisation of a large number of compounds. Furthermore, the interest in using this technique for water disinfection has grown in the last decade. Recent publications have reported photokilling of bacteria and viruses by TiO_2 photocatalysis. Therefore, solar photocatalysis disinfection seems to be a very promising process, which could help to improve public health in rural areas of developing countries.

The objective of this work was to assess the feasibility of using TiO_2 solar photocatalysis to disinfect water supplies for future applications in developing countries. This article reviews the viability of solar photocatalysis for disinfection in low cost compound parabolic collectors, using sunlight and titanium dioxide semiconductor, both applied as slurry and supported. We report on the bactericidal action of TiO_2 on a pure culture of *Escherichia coli* with a low cost photoreactor based on compound parabolic collectors. The influence of different experimental set-ups and parameters are also analysed.

The results and potential application of the solar photocatalysis technology to water disinfection are studied within the frame of two research EU projects whose objective consist on the development of a fully autonomous solar reactor system to purify drinking water in remote locations of developing countries.

Keywords: Compound parabolic collector (CPC); Solar photocatalysis; Disinfection; Titanium dioxide

1. Introduction

Nearly 1 billion people have serious problems in accessing the drinking water vital to their life and development [1], primarily in developing countries that lack freshwater resources and adequately developed treatment infrastructure. Nevertheless, most of these regions are the world's sunbelt. Therefore, water disinfection by solar radiation would seem to be a suitable method for helping to solve their water supply problems.

TiO_2 solar photocatalysis has been proven to be a degradation process for aqueous organic contaminants leading to total mineralisation of a large number of compounds. Furthermore, the interest in using this method for water disinfection has grown in the last decade. Indeed, recent publications have reported photokilling of bacteria and viruses by TiO_2 photocatalysis [2,3]. Therefore, solar photocatalytic disinfection could be of help in improving public health in these countries.

This is a well-known advanced oxidation process (AOP), which is based on the production of hydroxyl radicals ($^{\bullet}OH$) when a catalytic semiconductor powder, e.g. TiO_2, is photoexcited with UV radiation of wavelength equal or lower than 390 nm in presence of water. The absorption of one UV photon generates electron/hole pairs (e_{CB}^{-}/h_{VB}^{+}) separated in the conduction band (CB) and the valence band (VB) of the semiconductor, respectively. All the possible catalytic reactions of the TiO_2 photocatalysis are complicated and involve the water, the dissolved oxygen and the surface groups of the catalyst [4]. Nevertheless, they are summarised by Eqs. (1) and (2).

$$H_2O + h_{VB}^{+} \rightarrow {}^{\bullet}OH + H^{+} \quad (1)$$

$$O_2 + e_{CB}^{-} \rightarrow O_2^{\bullet -} \quad (2)$$

* Corresponding author. Tel.: +34 950387900; fax: +34 950365015.
E-mail address: pilar.fernandez@psa.es (P. Fernández).

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Sometimes, TiO_2 has been used for the treatment of bacteria by the photocatalytic process in water [3,5,6]. The recent studies performed by Rincón and Pulgarín give new information about the influence of several main parameters on the capability of the TiO_2 photocatalyst for water disinfection [2,7,8]. However, the combined use of this photocatalyst with the solar radiation has not been so usual [9], except the treatment of wastewater containing persistent pollutants, which shows a wide range of applications from small to pre-industrial scale [10,11]. Some relevant works have been carried out to analyse the viability of pilot plant installations to common bacteria inactivation [9]. Nevertheless, the study of water disinfection at pilot plant by solar photocatalysis needs more additional studies to develop practical applications.

This work is addressed to determine the capability and effectiveness of using solar photocatalysis with TiO_2 to disinfect water supplies in order to design future implementations to solve some problems related to the lack of drinking water in developing countries. The present paper illustrates the most relevant results found in the study of the solar photocatalysis with TiO_2 slurry for bacteria killing in a photoreactor based on low cost CPC photoreactors [10,12]. The main operational parameters related to this process are studied in this paper. The efficiency of the TiO_2 bacteria inactivation properties under solar radiation is proven. The effect of the catalyst concentration, the flow rate, the irradiated surface and the light intermittence are also analysed from the experimental point of view. Besides, this contribution reports some results about the bactericidal effect of supported TiO_2 on a fibreglass matrix.

The set of results and its potential application for water disinfection are studied within the frame of two EU research projects. The basic goal of those is to develop a complete autonomous solar system for the disinfection and degradation of trace organic pollutants in drinking water to be placed in remote locations, without using any chemicals. The final system shall treat water using photocatalytic processes activated by sunlight. This will be able to treat few hundreds litres per day. For this objective, one solar reactor based on hydroxyl radicals generation by supported TiO_2 over an inert matrix will be built up [13].

2. Experimental

2.1. Photoreactor

All the experiments were carried out under sunlight at the Plataforma Solar de Almería (PSA, latitude 37°N, longitude 2.4°W) using compound parabolic concentrators (CPC). CPCs are static collectors with a reflective surface designed to be ideal in the sense of Non-Imaging Optics and can be designed for any given reactor shape [12].

The photoreactors installed at the PSA consist of a CPC pilot plant with modules mounted on a fixed platform tilted 37° (local latitude) and connected in series so that the water flows directly from one to another and finally to a tank (Fig. 1). A centrifugal pump then returns the water to the collectors. These plants have been previously described in detail elsewhere [11,14,15]. The pilot plant consists of three CPC collectors, and each collector consists of two Pyrex tubes, which has a 0.25 m^2 collector surface. The photoreactor volume is 5.4 L and the total plant volume 11 L. This photoreactor was used for the experiments with suspended TiO_2.

The photoreactor was modified for the experiments with supported TiO_2. A solid support was employed to fix the catalyst. This support, made of tubular and watertight polypropylene (PP), was introduced into two of the glass tubes of the photoreactor (Fig. 2). For optimal optic efficiency of the solar concentrator, this concentric configuration might fulfil that the quotient between the inner diameter of the glass tube (D_i) and the external diameter of the concentric support (d_e) is equal to the refraction index of the fluid (n): $D_i/d_e = n$. For our case of study, the water ($n = 1.33$), the support was chosen to have a 22 mm diameter, since the glass tube has 32 mm of external diameter and 1.4 mm thickness. This ensured the maximal concentration ratio of the solar CPC collector [12].

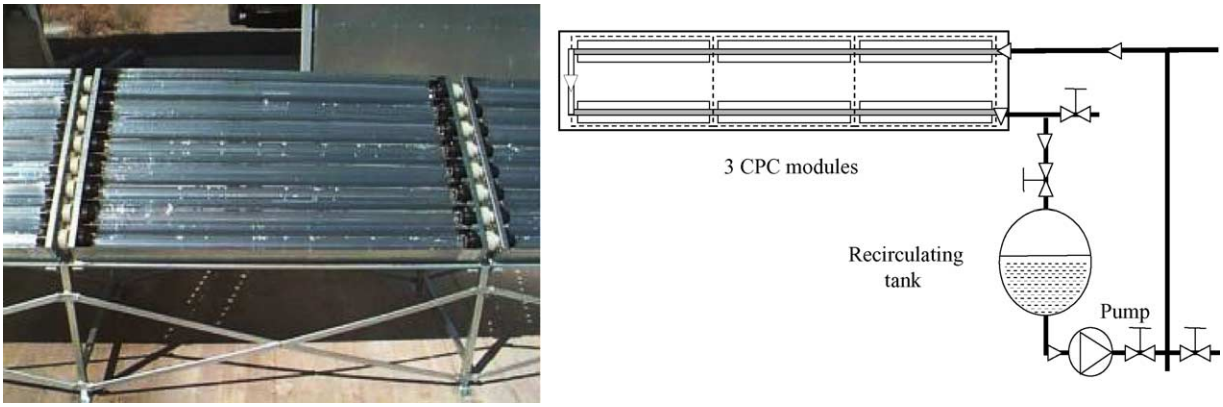

Fig. 1. View of one CPC collector module (left: photo made at PSA, Spain) and scheme of the installation (right).

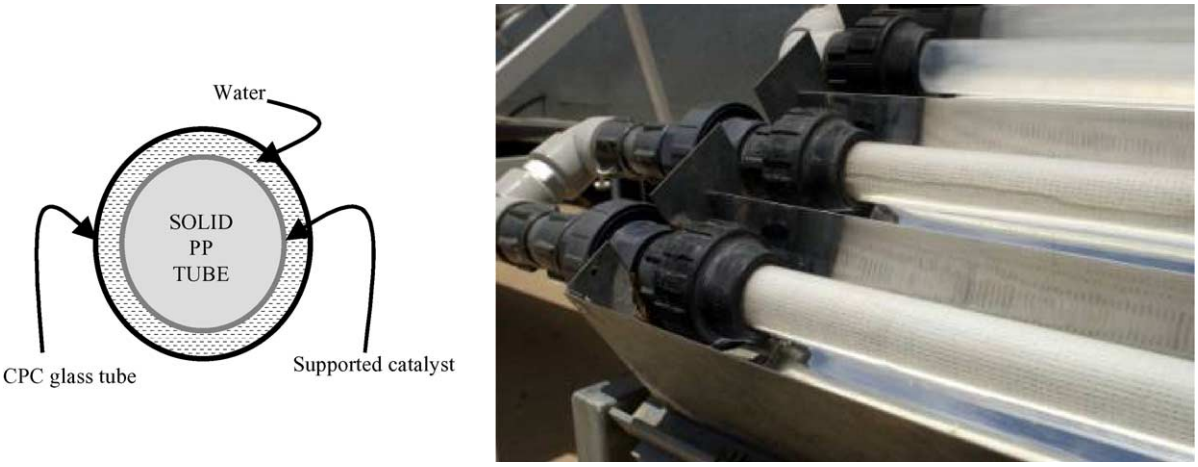

Fig. 2. Scheme of the supported TiO_2 configuration (left). View of the supported photocatalyst into the glass tubes of the CPC photoreactor (right).

At the beginning of the experiments, with collectors covered, the bacteria suspensions and/or powder catalyst are added one after the other to the tank and mixed until constant concentration is achieved throughout the system. If supported catalyst's tests were performed, the supports were prepared and introduced before the photoreactor filling, and then the bacteria were added. After that, in both cases, the cover is removed and samples are collected at predetermined times (t). Solar ultraviolet radiation (UV) was measured by a global UV radiometer (Mod. CUV3, KIPP&ZONEN), mounted on a platform tilted 37° (the same angle as the CPCs), which provides data in terms of incident $W_{UV}\ m^{-2}$. This gives an idea of the energy reaching any surface in the same position with regard to the sun. With Eq. (3), combination of the data from several days' experiments and their comparison with other photocatalytic experiments is possible [16].

$$Q_{UV,n} = Q_{UV,n-1} + \Delta t_n \overline{UV}_{G,n} \frac{A_r}{V_t}; \quad \Delta t_n = t_n - t_{n-1} \qquad (3)$$

where t_n is the experimental time of each sample, V_t the volume of each plant, A_r the illuminated surface of collectors, $\overline{UV}_G$ the average UV_G during Δt_n and $Q_{UV,n}$ is the accumulated energy (per unit of volume, kJ/L) incident on the reactor for each sample taken during the experiment. Sometimes, it is useful to explain the results in terms of illumination time instead of Q_{UV} because time is an easier parameter to handle.

2.2. Photocatalysts

The heterogeneous photocatalytic degradation tests were carried out using a slurry suspension of Degussa (Frankfurt, Germany) P-25 titanium dioxide (anatase:rutile = 3:1; surface area = 50 m^2/g, non-porous particles). The transmission electron microscopy (TEM) images of this TiO_2 show a highly disparity of particles 20–40 nm sized. This was suspended as received in a 0.5 L vessel and autoclaved before adding and dispersing into the photoreactor water.

The supported photocatalyst tests have been carried out with TiO_2 immobilised on an inert matrix that was prepared in this way. The powdered photocatalyst was Degussa P25 titania. Titania P25 was coated on glassfiber paper (synthetic fibres, 2 mm thick) using an inorganic binder. The binder was an aqueous dispersion of colloidal SiO_2. After washing, TiO_2 weighed 19.3 g/m^2 [17]. A 0.14 m^2 sheet of the glassfiber paper on which the titania photocatalyst had been deposited was fixed on each concentric PP support placed in the CPC photoreactor. The supported photocatalyst is named KN47 [18].

2.3. Bacterial strain and quantification

The bacterial strain used *Escherichia coli* K-12. *E. coli* was inoculated into nutrient broth type Luria Broth medium (Miller's LB Broth, Sigma–Aldrich) and incubated at 37 °C by constant agitation under aerobic conditions during 20–24 h. After this, the basal equilibrium of bacteria was reached, yielding a concentration of 10^9 colony formation units (CFU) per millilitre. Optical density measurements performed after the strain incubation at 600 nm determine this equilibrium concentration. No centrifugation is needed to prepare *E. coli* suspensions due to the very low concentration used in all experiments of the present work, 10^4 CFU/mL. This concentration was chosen because it is the typical concentration of the natural ground water and, as mentioned in Section 1, the final application of this research is aimed to provide drinking water and to treat natural water contamination in isolated areas of developing countries, neither industrial wastewater nor sewages.

For the overall experiments, the suspension of bacteria in distilled and autoclaved water was made directly in the tank of the photoreactor by inoculation of 110 μL from concentrated culture in 11 L of total volume of water. Determination of bacteria concentration of the several samples was performed in the following way. Serial dilutions of the samples in autoclaved and distilled water were carried out when necessary and samples plated on LB

agar (Sigma–Aldrich, USA). Every sample was plated 16 times (10 μL inoculation volume) on Petri dishes containing LB agar, in order to minimise the quantification error of the results. If the concentration of any sample was expected to be lower than 200 CFU/μL, 250 mL volume of the sample was spread out over the agar for better determinations of concentration. The inoculated samples were incubated at 37 °C for 20–24 h before colony counting, afterwards the statistical data processing was carried out, being the error which yields a confidence level of 95% of the mean value from the 16 results.

2.4. Methodology of photocatalytic disinfection

For all the photocatalytic experiments the *E. coli* suspensions were prepared in darkness (black cover over the solar collectors) with an initial concentration of around 10^4 CFU/mL, checked by sampling (called *Control* 1). After this, a given quantity of TiO_2 is added to the reactor and mixed by pumping in darkness during 15 min to homogenise perfectly the final suspension. Then, a new sample (*Control* 2) is taken to determine the adsorption or inactivation of bacteria in presence of TiO_2 in the dark. Both control samples were kept in darkness at the laboratory and inoculated at the end of every experiment with the objective of determining the stability of the bacteria in the presence or absence of catalyst. Once the suspensions were prepared, the cover was removed and the solar photocatalysis experiment started and the samples were taken at pre-determined times. The protocol followed during the experiments with supported photocatalyst was the same than that followed during suspended catalyst experiments, with the exception that addition of the photocatalyst was not necessary, since it was in the photoreactor (immobilised TiO_2) at the beginning of all runs. In such case, only one control sample is kept in the lab to monitor the bacteria survival.

Blank experiments were performed in the photoreactor only with bacteria suspensions in darkness (black cover) or in the presence of sunlight and without catalyst, depending on the purpose of the test. The water used in the experiments was obtained from the PSA Distillation Plant (conductivity < 10 μS/cm, $Cl^- = 0.2$ mg/L, $NO^{3-} = 0.5$ mg/L, organic carbon (OC) < 0.5 mg/L) and it was autoclaved before any experiment.

2.5. Experimental parameters

This work evaluates the role of several experimental parameters like the flow rate, the catalyst concentration, the light intermittence and the irradiated surface. Three flow rates were evaluated: 22.5, 13.0 and 5.0 L/min in the presence of natural solar light with and without TiO_2. Several geometric configurations of solar irradiation were also tested. For this purpose, some parts of the solar collector were covered to control the irradiated surface and the frequency of the light and dark periods. Thus, the solar radiation effect was studied under four irradiated surface values and intermittence conditions, as shown in Section 3. The study of the role of the catalyst is evaluated with four TiO_2 concentrations: 25, 50, 200 and 500 mg/L and with supported TiO_2 on a glass fibre support supplied by Ahlstrom.

3. Results and discussion

3.1. Natural solar light at CPC reactor

The effect of sunlight on *E. coli* suspensions was analysed in several experiments in bacteria inactivation with four different photoreactor configurations (Fig. 3, right) in which surface areas and intermittence of irradiation differed (Fig. 3, left). A blank run with no irradiation, labelled "no sunlight" in Fig. 4, was also carried out.

Fig. 4 shows the evolution of the normalised concentration of bacteria against irradiation time (left side) and the $\log_{10} C$ versus Q_{UV} (Eq. (3)) for the same experimental results (right side), where C is the bacteria concentrations, in CFU/mL units, at any given moment during the experiment.

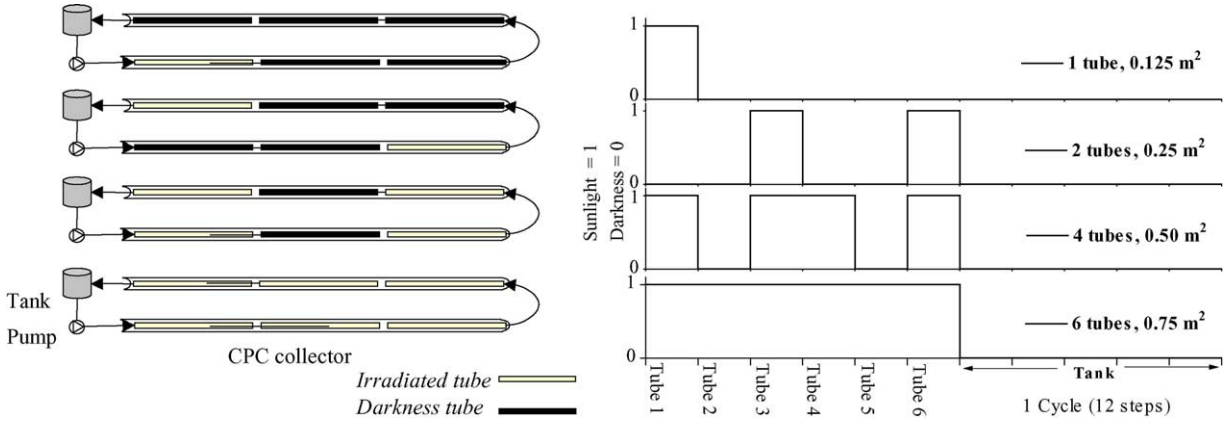

Fig. 3. Experimental configurations of four irradiated surfaces (left side). Sunlight irradiation time for each configuration during one cycle of all the fluid throughout the solar photoreactor (right side).

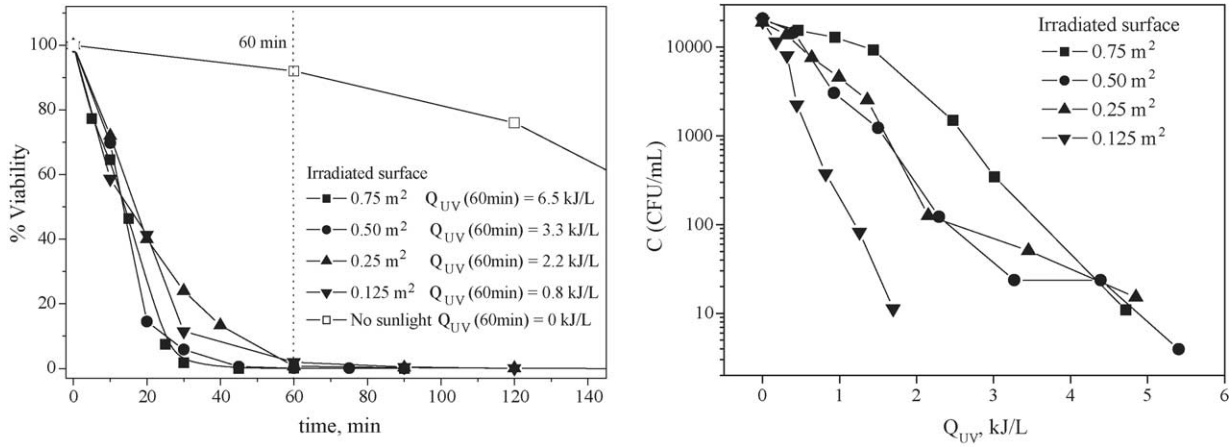

Fig. 4. *Escherichia coli* inactivated under sunlight in the CPC solar photoreactor with four irradiated surfaces (0.75, 0.50, 0.25 and 0.125 m^2) and a dark control run. The graph on the left shows the percentage of bacteria survival vs. time and the one on the right the $\log_{10} C$ vs. Q_{UV}.

As also reported by other researchers [2,9,19,20], bacteria are clearly inactivated by sunlight, as detected in the difference between the results obtained in the experiments with and without light.

Bacteria deactivation with several different irradiated surfaces shows a decreasing exponential tendency and no significant differences (Fig. 4, left), which is inconsistent with the idea that the more radiation, the more deactivation. The two different plots for the same results demonstrate that the experiment performed with the largest irradiated collector surface yields the lowest efficiency, and the lowest irradiated surface produces the highest bacteria inactivation rate. This paradoxical result is proven in the graph on the right in Fig. 4. Indeed, Fig. 4 (left) indicates that for a given time, e.g. 60 min, survival is similar for all the irradiated surfaces with different UV energy accumulated in the photoreactor Q_{UV} (kJ/L) for each (Fig. 4, inset). No differences in bacterial performance are detected with differing intermittence either, probably due to the fact that the flow rate is too high (22.5 L/min) to establish differences between light and dark residence times. Further experiments might be designed to test such possible effects, as found by Rincón and Pulgarín [7].

The effect observed in this figure could be attributed to several reasons. On one hand, the small energy contribution of the solar irradiation – compared to the Q_{UV} values at any given time in all of the systems – might be enough to cause *E. coli* inactivation, since regardless of the incoming radiation, behaviour was similar in all of the configurations. Another possible reason that could be responsible for the results is the combined effect of the accumulated incoming UV energy in the reactor over experimental time. These assumptions had to be verified with additional experiments.

To test the first possibility, two additional experiments were carried out. One of them consisted of shortening the solar illumination time (5 min) and lengthening the runtime in the dark (85 min), and the second, of two periods of solar irradiation (15 min each), with a dark period of 90 min between them and again at the end. Bacteria deactivation

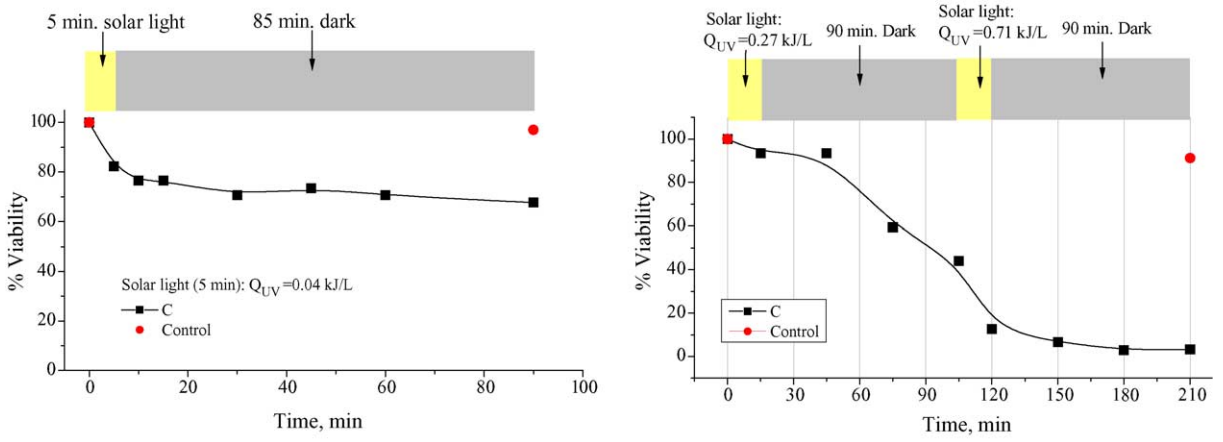

Fig. 5. *Escherichia coli* deactivation under sunlight in the CPC solar photoreactor (0.25 m^2) and a dark control run. Left graph: 5 min solar irradiation and 90 min in the dark. Right graph: two 15 min periods of solar irradiation and 90 min in the dark.

observed in the two runs is presented in Fig. 5. The graph on the left shows that very low UV energy in the photoreactor (0.04 kJ/L) is not enough for total bacteria deactivation. The graph on the right proves that more accumulated UV energy per litre (0.27 kJ/L) is not enough either, although it has a retarded effect on the bacteria during the dark period. Furthermore, another short period of light was necessary for total deactivation of the *E. coli*.

The temperature increase inside the photoreactor was discarded as a factor causing bacteria deactivation, since it was measured between 15.8 and 36.5 °C, at which *E. coli* suspensions are stable. It is also important that *E. coli* deactivation by sunlight in absence of TiO_2 is not a photocatalytic process and has no bactericidal consequences, because regrowth of bacteria was detected in all cases.

3.2. Flow rate experiments

The flow rate selected to carry out the experiments with bacteria could be an important factor because stress from movement of the fluid in which the bacteria are dispersed could cause their inactivation. Therefore, three different flow rates, 22.5, 13.0 and 5.0 L/min, were evaluated. The experiments were carried out in the reactor with 0.25 m^2 of irradiated surface area and compared to results in the dark, in order to differentiate the stress due only to flow rate or to flow rate plus sunlight. The results of these experiments are presented in Fig. 6, as the percentage of viable bacteria over time.

As observed in this graph, the effect of the stress produced by the different flow rates is only significant when the bacteria suspensions are inactivated in the presence of solar radiation. Even though the lowest flow rate produces the highest residence time of bacteria under solar radiation, the efficiency of bacteria inactivation is still the lowest. This may be attributed to the capacity of bacteria to adapt to stressful conditions such as incoming solar photons inside the reactor, which could easily be developed if residence time were long enough. This could explain how 22.5 L/min yields higher efficiency than 13.0 L/min, which is higher than 5.0 L/min.

The bacteria regrowth detected in all the experiments carried out without the catalyst justifies the use of titanium dioxide as a photocatalyst to disinfect water containing *E. coli* 10^4 CFU/mL. The following results (Fig. 7) were obtained with 50 mg/L of TiO_2 Degussa P25 in the solar photoreactor with 0.25 m^2 of irradiated surface.

As Fig. 7 shows, *E. coli* inactivation produced by solar photocatalysis is between three and six times faster in the presence of TiO_2. In agreement with the literature, in this figure the great importance of the role of the photocatalyst is clear [3,9,21–23]. As photocatalytic activity depends on the quantity of UV photons (wavelength < 390 nm) or the intensity of UV radiation, the results are shown against the incoming UV energy accumulated in the collector per unit of volume, Q_{UV} (Eq. (3)). The points plotted in the negative region of Q_{UV} in Fig. 7 are only values corresponding to dark conditions, since negative values of accumulated UV energy is pointless. Moreover, the process strongly depends on the flow rate, probably due to two factors: (1) the stress produced by mechanical agitation inside the reactor (Fig. 6) and/or (2) the possibility of bacteria being adsorbed or reaching the catalyst. The adsorption of *E. coli* on the catalyst in darkness seems to be quite strong. This process has to be studied further to determine the extent of this effect, however, bacteria deactivation or killing was verified by regrowth tests made in all experiments 24 h after inactivation on samples stored in the lab at room temperature. Regardless of the adsorption or desorption of colonies on the catalyst surface, no regrowth was detected when TiO_2 was employed.

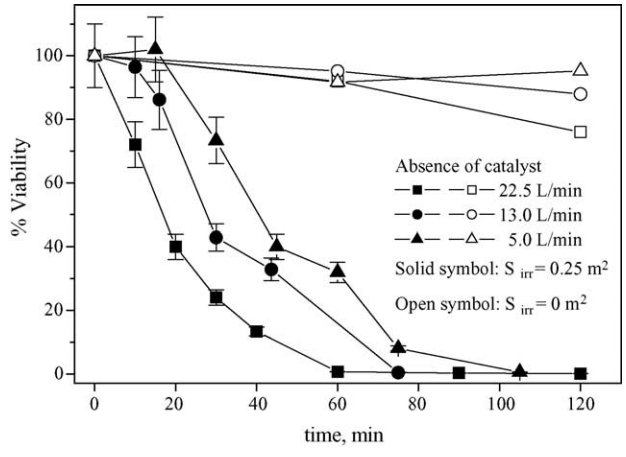

Fig. 6. Inactivation of *Escherichia coli* in absence of catalyst under sunlight for 0.25 m^2 irradiated surface and flow rate: 22.5, 13.0 and 5.0 L/min (solid symbols). Experiments performed in the dark in absence of catalyst with the same flow rates (open symbols).

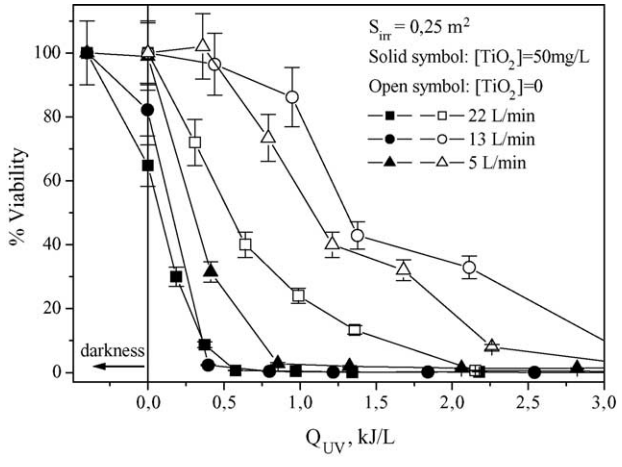

Fig. 7. Inactivation of *Escherichia coli* under solar photocatalysis [TiO_2] = 50 mg/L, 0.25 m^2 of irradiated surface and three flow rates: 22.5, 13.0 and 5.0 L/min (solid symbols) and for experiments made without catalyst under solar radiation (open symbols).

3.3. Photocatalyst concentration

Titanium dioxide Degussa P25 (a cheap photocatalyst) has been tested with a large number of hazardous compounds and with various types of bacteria and microorganisms [3,8,24]. However, while there are several studies on bacteria killing with supported photocatalyst and electro-photocatalysis [21,22], other studies report optimised efficiency of disinfection with a TiO_2 slurry [8], which has been proven to be much more effective for a wide range of chemical contaminants.

The experiments performed with $[TiO_2]$ = 0, 25, 50, 200 and 500 mg/L and 22.5 L/min showed total bacteria deactivation and no regrowth in any case in the treated water (Fig. 8). The ''blank'' experiments performed with neither catalyst nor radiation produced a decrease in bacteria survival of about 10%. According to other authors [2], the deactivation of the bacteria suspension depends slightly on the catalyst concentration for the evaluated range. For high concentrations of catalyst, this can be explained by the attenuation of light by titanium dioxide, which avoids the photoexcitation of all the available sites on the surface of the catalyst for generating hydroxyl radicals and $^{\bullet}OH$ in presence of UV and thus produces similar photocatalysis efficiency at different catalyst concentrations.

3.4. Supported photocatalyst

Although the experiments performed with fibreglass paper as the supported photocatalyst (KN47) yielded inactivation of pure *E. coli* suspensions, the bactericidal efficiency of TiO_2 is more than twice as high when in a slurry (Fig. 9). This was expected from previous results on supported photocatalysis with organic pollutants [25] or even with bacteria [2].

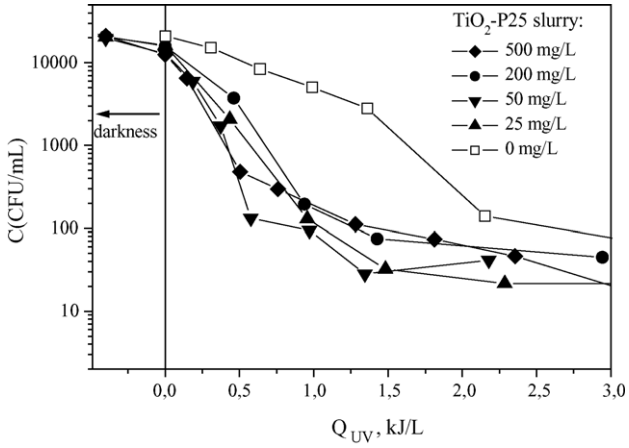

Fig. 8. *Escherichia coli* deactivation ($C_0 = 10^4$ CFU/mL) vs. Q_{UV} in the CPC solar photoreactor with 0.25 m^2 irradiated surface area for $[TiO_2]$ = 0, 25, 50, 200 and 500 mg/L.

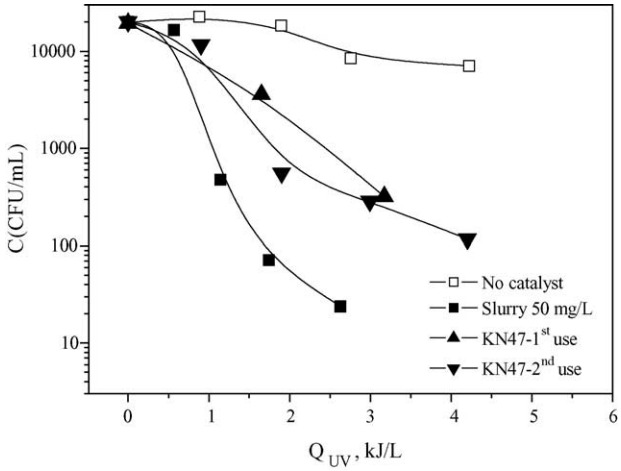

Fig. 9. *Escherichia coli* deactivation ($C_0 = 10^4$ CFU/mL) vs. Q_{UV} in the CPC solar photoreactor. Comparison of TiO_2 slurry, 50 mg/L (solid squares), with supported photocatalyst KN47 (19.3 g TiO_2/m^2) in two photocatalytic uses (solid triangles) and only solar light (open squares).

4. Conclusions

A CPC solar photoreactor has been demonstrated to be efficient for bacteria disinfection by solar photocatalysis with TiO_2 slurries and supported TiO_2 during treatment periods of 30–60 min. 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 TiO_2 in suspensions and or by supported TiO_2.

However, while sunlight deactivates *E. coli* suspensions, it does not completely deactivate them, since bacteria regrowth was detected. This method of disinfection has to be improved by the photocatalytic action of TiO_2 under solar radiation to kill bacteria completely, which was proven successful and efficient enough to persevere in work in photocatalytic applications for drinking water disinfection.

The irradiated area in the CPC collector plays a key role in the bacteria inactivation by solar irradiation. There is also a synergistic effect of the experiment runtime and the irradiated collector surface due to stress from flow rate on the bacteria suspensions.

Compared to the supported TiO_2 as a photocatalyst, the slurry TiO_2 behaves more efficient for bacteria deactivation. Under our experimental conditions, the disinfection rate is independent of catalyst concentration, although not as high as those reported in the literature. Results found with supported TiO_2 on fibreglass paper (Ahlstrom ©) are promising.

Acknowledgements

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