PHOTOCATALYTIC DESTRUCTION OF WATER POLLUTANTS USING A TIO2 FILM IN PET BOTTLES

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ABSTRACT

The performance of the PET bottles coated with a TiO_2 was studied in this work. These bottles can be used for solar water disinfection because TiO_2 combines the action of solar disinfection (SODIS) and advanced oxidation process improving the effectiveness of these technologies (Litter et al., 2005). The effect of different parameters such as initial contamination, solar radiation, etc. is analyzed using kinetics models (Salih, 2003; Horie et. al, 2005) that are validated by experiments. The contaminants studied are bacteria (E. coli) and arsenic. The behavior of the PET bottles performance is simulated with actual weather data from remote sites in Peru.

1. INTRODUCTION

Water, although vital for supporting life, is a major threat to human health. According to the World Health Organization, 1.8 million of people die because of diarrhea per year and 88% of these cases are related to the ingestion of contaminated water, lack of sanitation and bad hygiene practice (World Health Organization [WHO], 2006.) Furthermore, almost 1/6 of World's population do not have access to safe drinking water, most of them in the third World. Meanwhile the construction of water purification plants is not suitable sometimes in the case of developing countries; developed countries can suffer natural disasters that can cut the drinking water supply to a lot of people. For these reasons, scientists and engineers have been investigated several water purification methods.

Litter et al. (2005) have developed a technique that combines both SODIS and photocatalyst methods in order to use their advantages. Meanwhile SODIS contributes with its simplicity; the advanced photooxidation process provides its effectiveness in the degradation of organic and chemical pollutants.

The approach of this technology consists in the use of recycled PET (Polyethylene Terephthalate) bottles that are coated with a film of TiO2. These bottles are used as photocatalytic batch reactor where the water can be treated in the sun for some hours to improve its quality (Litter et al. 2005).

Although this new method is an important contribution, the influence of different factors on its performance needed some investigation (temperature, solar radiation, ambient temperature), the PET leaching effect to the water after some hours of exposition to the sun, the effect of this technique on the resultant water (taste, smell, appearance, etc.), the proper water storage time without recontamination, and the actual feasibility of using and manufacturing the bottles in villages by local people.

In order to investigate these factors, the University of Massachusetts Lowell, with its project Village Empowerment, has performed some experiments in isolated Peruvian villages in the Andes. Bottles coated with TiO2 were tested in the project trip to Peru in June, 2006, and the results have been considered in the development of this work. In addition, mathematical models of water disinfection methods that use TiO2 as catalyst (Chang et al., 2000; Li et al., 1996.) are used in order to analyze the individual effect of several factors and predict the performance of this method under real conditions. Finally, the mathematical model has been validated with experiments performed at the University.

2. SOLAR DISINFECTION TECHNOLOGIES

2.1. SODIS

Solar Disinfection (known as SODIS) is an ancient technology that uses the effect of solar UV radiation for the inactivation and destruction of microorganisms in drinking water. The technology is as simple as filling a plastic bottle with water and exposing it to solar radiation for a determined time.

Other advantages of this technologies are the low cost (it can use recycled bottles) and the avoidance of chemicals, such as chlorine. Although its effectiveness has been proved in different places (EAWAG & SANDEC, 1998), this method is sensitive to the volume of water to be treated, initial concentration of pollutants, water turbidity, solar radiation, the amount of oxygen dissolved in the water and the container used.

Polyethylene terephtalate (know as PET) is a material used in the manufacturing of plastic bottles and contains stabilizers that protect the plastic against the UV effects. Due to its low weight, transmissivity of UV-A radiation and chemical stability, it is preferred as the material for SODIS treatment. PET can be used as bottles or as plastic bags.

2.2. Advanced Oxidation Process-TiO2

Titanium dioxide (TiO_2), also know as titania, is a catalyst used in the advanced oxidation process in water disinfection. It has been tested for the reduction of hazardours organic chemicals and some inorganic pollutants (Bissen, Vieillard-Baron, Schindelin and Frimmel, 2001). It is available in three different crystalline structures: anatase, rutile and brokite. For photocatalytic purposes just anatase and rutile are used because of their optical properties, opacity and durability. Although rutile TiO_2 has been considered as photocatalytically inactive, it has been found that Degussa P-25, a commercial photocatalyst that has a proportion anatase/rutile of 3-4, has the best performance in photocatalysis (Sun and Smimioti,2003). In the case of bacteria inactivation, It seems that the different characteristic of TiO2, such as the surface area and the particle size, do not influence their bacterial inactivation efficiencies (Gummy, Rincon, Hadju and Pulgarin, 2005).

2.3. TiO2 Photocatalytic Reactions

When a photon is absorbed by a material, an electron is released. In order to do that, the energy of the photon should be greater than the bandgap of the material. Then, if the resultant electrons and positive holes react with the surrounding molecules causing chemical reactions, a photoreaction takes place (Goswami et al., 2000).

With quantum energy in Planck's equation, the energy of a photon is described by:

$$\varepsilon = h \cdot \frac{c}{\lambda} \qquad (1)$$

Where ε is energy in Joules, c is speed of light (3 X 10^8 m/s), λ is wavelength and h is Planck's constant (6.625 X 10^{-34} J-s). Using equation (1) and knowing that anatase TiO2 has a bandgap of 3.25 eV (Syarif et al., 2002), we find that the maximum wavelength that would produce an effect on TiO2 is 382 nm.

Now when the catalyst is activated by photons, negative electrons and positive holes are formed:

$$TiO_2 + hv \rightarrow TiO_2 \cdot \left(e_{cb}^{-} + hole_{vb}^{+}\right) \qquad (2)$$

The most common reaction is the recombination of electrons and holes on the surface of the catalyst, reducing the quantum efficiency of the process (Bandala, Arancibia-Bulnes, Orosio & Estrada, 2004).

$$TiO_2 \cdot \left(e_{cb}^{-} + hole_{vb}^{+}\right) \xrightarrow{recomb} TiO_2 + heat \qquad (3)$$

Other electrons can react with reducible species such as oxygen molecules. Positives holes can oxidize species reducing the possibility of recombination (Son, Lee, Cho and Lu; 2004)

The reactions that yield additional hydroxyl radicals may continue with other reactions. The hydroxyl radical is a very powerful oxidizing agent which can oxidize organic pollutants, directly or through intermediate compounds (Goswami; 2001).

2.4. Factors Affecting UV Inactivation

2.4.1. pH and Temperature

For bacteria, the rate of disinfection is considered independent from pH values, if it does not change drastically during the process (Rincon & Pulgarin; 2003). Cho, Chung, Choi and Yoon (2004) established that when pH varies from 5 to 9, reaction rates could be considered independent of this characteristic. In the case of the effect of temperature effects on bacteria disinfection, it was found that they are more susceptible to oxidative radicals at higher temperatures (Cho et al.; 2004), where each microorganism has its own resistance to the chemicals.

Temperature has also its own germicidal effect by itself. Fujikawa, Ushioda and Kudo (1992) assumed that the effect of temperature over bacteria can be described for a first order expression:

$$-\frac{dC}{dt} = k_{(T)} \cdot C \qquad (4)$$

$k_{(T)}$ is constant rate that is function of temperature.

2.4.2.Dissolved Oxygen

Bactericidal action of sunlight on water is dependent on the amount of molecular oxygen (O_2) dissolved in water. TiO_2 photoreacts with oxygen contented in O_2 and H_2O, therefore the lack of sufficient supply of oxygen would reduce the reaction rates (Sagawe, Brandi, Bahnemann & Cassano,2005); although photoreactions have been observed under absence of dissolved oxygen .

According with Herrera et al. (1999) to swirl or shake water would be enough to provide enough oxygen to improve the efficiency of the reactions. After the reactions start and dissolved oxygen is consumed, the needed molecular oxygen can be absorbed from the surrounding air that is in contact with the water.

In order to analyze de photoreaction rate of dissolved oxygen, the Langmuir- Hinshelwood model can be used:

$$-r = \frac{a \cdot b \cdot C_{DO}}{(1 + bC_{DO})} \qquad (5)$$

where r is the reaction rate, a and b are equation constants and C_{DO} the concentration of dissolved oxygen

2.4.3. Turbidity

Turbid water attenuates more radiation, diminishes the amount of dissolved oxygen, and increases the amount of organic elements that are affected by the oxidative radicals. Temperature has the main germicidal effect on high turbidity water. EAWAG and SANDEC (1998) recommend the use of sedimentation or filtration to separate solids from the water when turbidity is higher than 30 NTU.

3. MATHEMATICAL MODEL

There have been several attempts to create a kinetics model of organic pollutants destruction. In order to be accurate and more general, models should take into consideration pH, oxygen concentration, temperature and any factor that could interfere with the catalyst reaction.

3.1.Langmuir-Hinshelwood Model

The Langmuir-Hinshelwood model has been used to describe several kinetic processes with good accuracy, especially with chemical pollutants. This model has the following relationship (Goswami, 2000):

$$-\frac{dC}{dt} = \frac{K_1 \cdot K_2 \cdot C}{(1 + K_2 C)} \qquad (6)$$

Where C is the concentration of contaminant solutate in water, K_1 is the reaction constant, and K_2 is the equilibrium adsorption constant. When C tends to be 0 (low concentration), the expression (6) can be simplified as:

$$-\frac{dC}{dt} = K_1 \cdot K_2 \cdot C = K \cdot C \qquad (7)$$

Where K can be understood as a total reaction constant that is proportional to the intensity of the radiation

$$K = k \cdot I \qquad (8)$$

and finally the following expression is obtained:

$$\frac{C}{C_0} = e^{-k \cdot D} \qquad (9)$$

Where k is the new reaction constants, I is UV radiation intensity, t is time of exposition, D is UV dose and Co is the initial solute concentration. The expression (9) has been used to describe disinfection processes in municipal wastewater with good accuracy (Li et al., 1996).

3.2. Multitarget Single Hit (MSTH) Model

The Langmuir-Hinshelwood model loses its accuracy when deals with microorganisms such as *E. coliform* or *S. cerevisiae*. When the disinfection process starts, microorganisms have an initial resistance against the germicidal effect of UV; the higher the concentration, the greater the resistance. In 1955 Douglas Lea develop a model for cell killing by radiation called “target theory of cell killing” that was experimentally validated for solar disinfection by Salih in 2000. Later, Salih (2003) came up with an equation that would describe in a better way the behavior of microorganisms such as *E. coli* under the effects of UV radiation. MTSH can be expressed in the following way:

$$\frac{C}{C_0} = 1 - \left(1 - e^{-Kx_1'}\right)^{x_2} \quad (10)$$

where x_2 is the target number of viable cells (cells that should be inactivated) and x_1' can be expressed as:

$$x_1' = D \times e^{-\frac{\mu}{\rho}\cdot\frac{d}{\rho}} \quad (11)$$

where D is UV dose and the expression $\frac{\mu}{\rho}\cdot\frac{d}{\rho}$ represents the attenuation of the environment that transmits UV radiation. EAWAG and SANDEC (1998) presents a table with the reduction of UV-A as function of turbidity.

3.3.Series Event Model

This model has been developed to analyze the effect of TiO2 in water disinfection from bacterial contaminants. In accordance with this model, the following assumptions were taken.

The cell deactivation obeys a second-order reaction between cells and oxidative radicals, and the death of a cell is caused by n times reactions. The concentration of oxidative radicals is constant under quasi-steady state. Horie, David, Taya and Tone (1996) proposed the resultant equation:

$$\frac{C}{C_o} = \exp(-k'\cdot t)\cdot\sum_{i=0}^{n-1}\frac{(k'\cdot t)^i}{i!} \quad (12)$$

where k' can be expressed as follows

$$k' = k_r C_{OX} \quad (13)$$

where k_r is a rate constant and C_{0X} is concentration of radicals. Furthermore, C_{0X} can be expressed in the following equation (Horie et al., 1996):

$$C_{OX} = k_\phi \cdot a_{T,0} \cdot \frac{A \times I}{k_{OX} \cdot V_L} \quad (14)$$

where k_ϕ is a quantum yield per specific area, $a_{T,0}$ is specific area, A is area exposed to radiation , I is radiation, k_{OX} is the apparent decomposition rate constant of oxidative radicals and V_L is volume of test solution.

4. RESULTS

4.1 Destruction of Bacteria

Initial bacteria (*E. coli*) concentration was set above 10 cell/ml using Hach's sulphur bacteria incubation test (HACH Cat. 261076). The tests were performed at different times at University of Massachusetts Lowell on the roof where water samples were taken, and radiation, temperature and time were registered. PET bottles of 600 ml were coated over half the area from top to bottom with TiO_2 according to the procedure of Litter et al. (2005) wherein $HClO_4$ was used to acidify the coating and help it adhere to the bottle wall.

In order to compare the performance between coated and non-coated PET bottles, both bottles were tested at the same time under the same conditions. To determine the water sample recollection time, *E. coli* disinfection simulation was used, selecting the time when the predicted bacteria concentration reaches 10, 1 and 0.1 cells/ml. These bacteria concentrations were chosen because of the dilutions of samples that were used (1, 1/10, 1/100, 1/1000) to test presence of bacteria.

4.1.1. Effect of UV Radiation Intensity

Because the test was performed using natural UV radiation and the data presented are average values of the data obtained during September and October 2006 at the University in Lowell from 12:00 p.m. to 4:00 p.m.
The following data was found when coated and non-coated bottles were used in bacteria disinfection experiments.

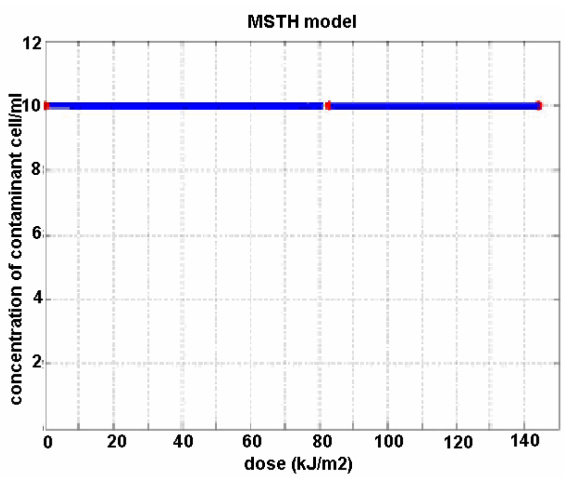

Fig. 1 E. coli concentration after using non coated bottles (*) data (-) model

From figures 1 it is obvious that the concentration of bacteria has not decreased. It is important to point out that the UV radiation and the time of exposure are less than the recommended values for solar disinfection.

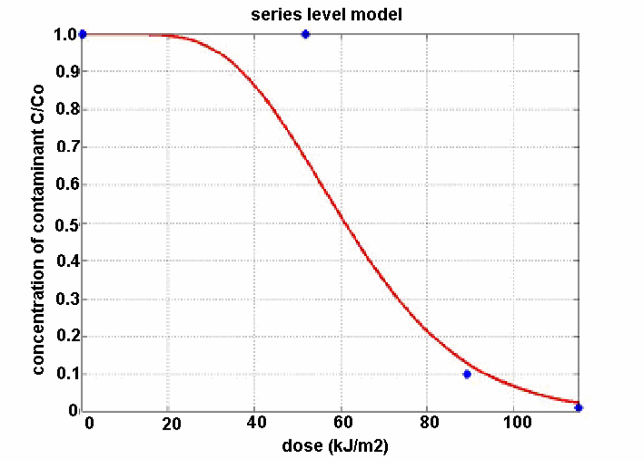

Fig. 2 *E. coli* concentration after using coated bottles with TiO_2 film (*)data (-) model

Bottles that were coated with TiO_2 film present better performance destroying bacteria than ones that are not coated. The final concentration of bacteria that was found is 10 cell/100ml, although it does not imply that all bacteria cells were destroyed, this value is used as disinfection lower limit because of the threshold of the bacteria presence test that were used (test strips).

4.1.2 Effect of Initial Time of Exposure

To analyze the effect of increasing and decreasing radiation, water disinfection tests were performed at different starting hours, testing some bottles in the morning and some in the afternoon. The analyzes was performed only for bottles that were coated with 0.04 g of TiO2 and that were filled with water with initial *E. coli* concentration of 10 cell/ml.

4.1.2.1 Morning Test

The tests and simulations were performed in the morning from 10 a.m. to 12 p.m.

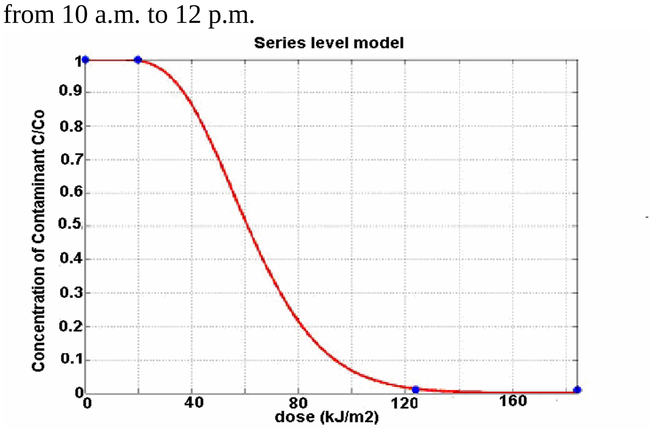

Fig. 3 Destruction of bacteria in the morning (*) data (-) simulation

4.1.2.2 Afternoon Test

The tests and simulations were performed in the afternoon from 12 p.m. to 4 p.m.

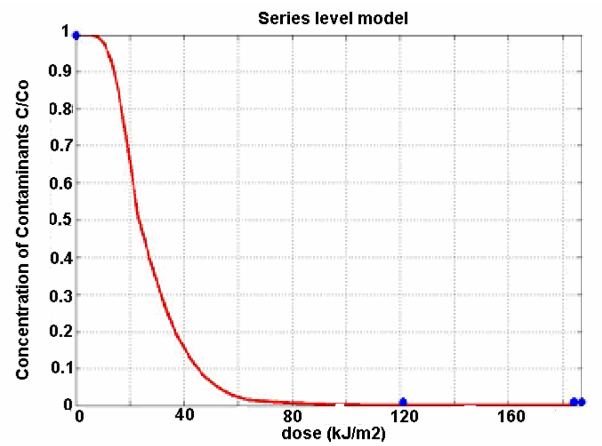

Fig 4 Destruction of bacteria during afternoon (*) data (-) simulation

The dose accumulation and the disinfection rate are slower in the morning as a consequence of the lower radiation that reaches the bottle; after that, when it is 12:00 p.m., the radiation increases, helping the final stage of the disinfection. In the other hand, the dose accumulation has the inverse behavior during the afternoon, so the rate of disinfection gets slower which makes the time that is required for disinfection longer

4.1.3 Effect of Initial Bacteria Concentration

The difficulty of experimentally obtaining high bacteria concentrations was overcome using mathematical simulation.

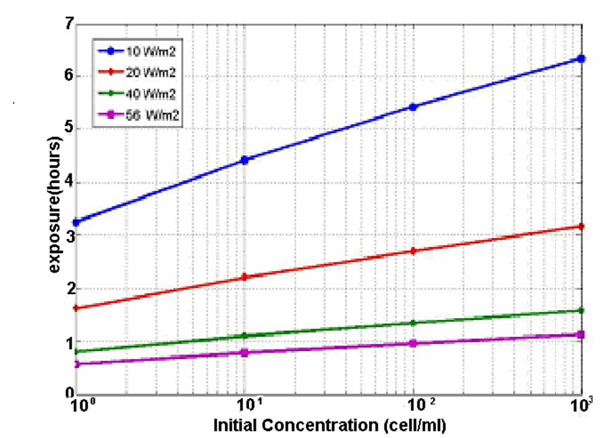

Fig. 5 Required time for disinfection of water contaminated with *E. coli* at different UV radiation levels

According to figure 5, effect of initial bacteria concentration is greater when the irradiation is low, because bacteria have higher resistance at lower radiation. At the same time, the required time of exposure has a logarithmic dependency of the initial bacteria concentration.

The curve that represents time of disinfection at 56 W/m^2 of UV radiation, which is the one that correspond to 1000 W/m^2 of total solar radiation, shows that the time needed to disinfect water that contains 1 cell of *E. coli* per milliliter is approximately 30 minutes, which is still a long period if we consider the low bacteria concentration and the high UV radiation.

4.1.4 Effect of TiO2 Concentration in Coating Film

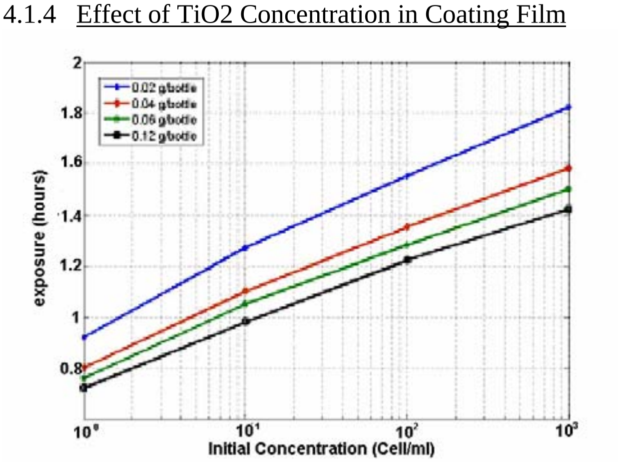

Fig. 6 Required time for total water disinfection at different TiO_2 concentration

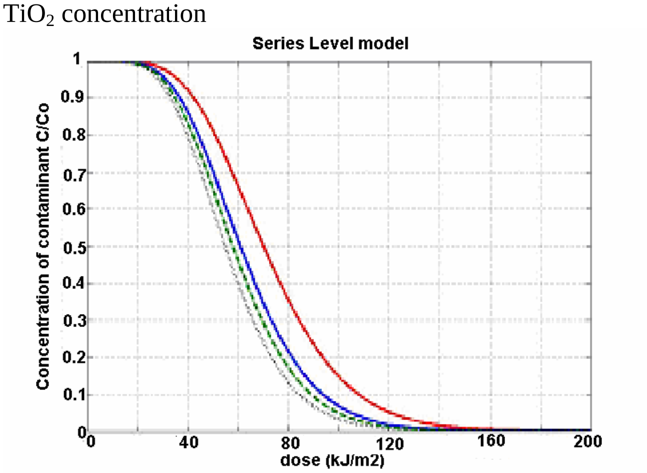

Fig. 7 Disinfection of *E. coli* with different TiO_2 concentrations (-) 0.02 g/bottle (-) 0.04 g/bottle (--) 0.06 g/bottle (..) 0.12 g/bottle.

The effect of TiO_2 concentration is presented in the Figs. 26 and 27. If TiO_2 concentration is increased from 0.02 g/bottle to 0.12 g/bottle, the time of exposure is reduced by 24 minutes at initial bacteria concentration of 1000 cell/ml. This little increment in the efficiency of the coated bottles is a consequence of the way that TiO_2 is used. Because TiO_2 is fixed to the bottle's wall and the photocatalytic oxidation occurs near to the TiO_2 particles, the increment of TiO_2 concentration only affects the portion of contaminated water that is in contact with the TiO_2 film.

4.1.5 Storage Time and Recontamination

In order to test the storage time for this technology, the same water sample with high bacteria concentration (+ 1000/100ml) was tested in two different bottles: one was coated with TiO2 and the other one was not coated. The bottles were exposed to low solar radiation (winter season – cloudy day) for few hours. After both bottles were tested, it was found that both sample contaminations were below the threshold of the test (1000/100ml).After two days of dark period (no exposure to sun), the results showed that the bottle with TiO2 was keeping the low bacteria concentration meanwhile the concentration of bacteria in the non-coated bottle went back over the threshold (+ 1000/100ml).

4.2 Simulation of Bacteria Destruction in Cochapeti

The Village Empowerment Peru Project (U Mass Lowell) has worked in Cochapeti since 1999, in the Peruvian Andean Mountains at 3400 meter above sea level at 9.98° S. latitude and longitude 77.65 ° W.

Radiation data was collected by the Peru Project team and it was used in the following simulation. This data was obtained by data logger system that saved hourly average values of total radiation (measured on the roof of the medical clinic in Cochapeti), ambient temperature, and information of the solar systems installed in the clinic. This information corresponds to a period of two years.

The following assumptions were taken:

- The contaminant to be simulated is *E coli.*
- The bottle will be placed in a horizontal surface that will not receive shadows from the surroundings.
- The effect of solar radiation is accumulative on the process of killing bacteria.
- Initial bacteria concentration of 1000 cells/ml.
- The water temperature will be approximately the same as the water temperature obtained under the same ambient conditions. This assumption allows using the data obtained in Lowell during fall (10-17 °C ambient temperature) in Cochapeti simulation during daytime (8-20 °C ambient temperature). Therefore, during warmer seasons, the model is conservative but it would be able to determine the minimum time to obtain safe water at given conditions

According with the data, the month with lowest radiation is March. The month with the highest radiation is October.

We obtain the following simulation results for March:

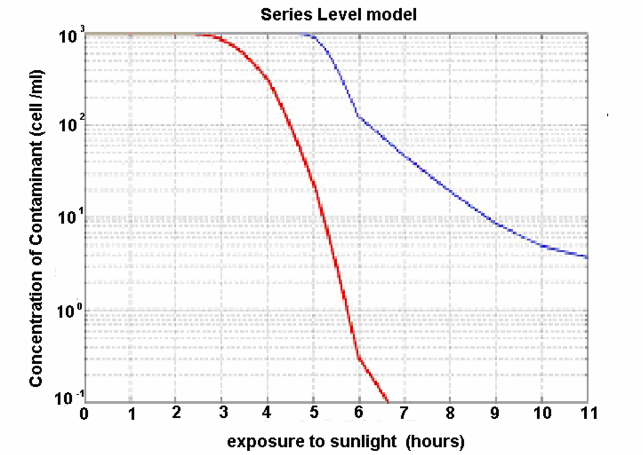

Fig. 8. Destruction of bacteria in Cochapeti in March with (-) coated bottles (-) non coated bottles

Fig. 8 shows that simple solar disinfection takes more than one day in order to reach contamination levels of *E. coli* 10 cells/100ml. In contrast the bottle coated with TiO_2 takes 7 hours if the exposure begins at 8:00 a.m.

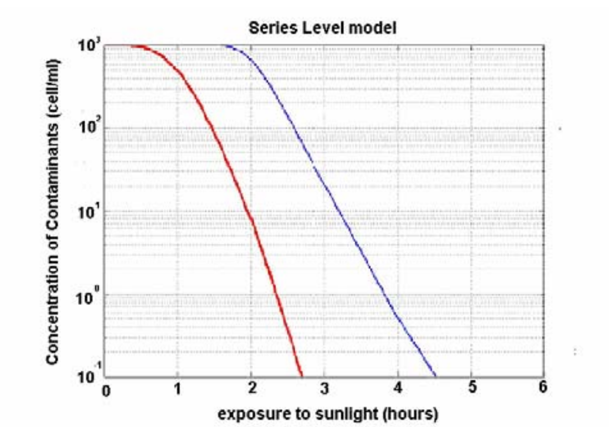

Fig. 9 Destruction of bacteria in Cochapeti in October (-) TiO2 film (-) SODIS

During the high radiation season, simple solar disinfection takes more 4 and a half hours, while disinfection using coated bottles takes almost 3 hours making the process faster by more than 90 minutes. Although it means a saving of time, the required time for disinfection is still long if it is compared with the use of chlorine or other water purification methods. However, other contaminants can be removed with the photocatalyst such as arsenic.

The simulations were performed using 8.00 a.m. as the initial time for exposure, which means that during the first hours, the accumulation of UV dose was slow. If the exposure starts at noon, the time will be reduced to an hour because of disinfection will take the advantages of the higher radiation and the consequent faster UV dose accumulation.

5. OTHER RESULTS AND DISCUSSION

The coating process used is very simple and results in TiO_2 coated bottles that can be used several times (at least for six times without obvious degradation of TiO_2 film, although more tests should be performed to study just this characteristic). The performance and the quality of the TiO_2 film obtained depend on the care shown in the coating process. For that reason care is needed. Locally made vinegar is not a substitute for the $HClO_4$ acid.

The simulation results establish that under low radiation in cloudy conditions (in Cochapeti), the time for water disinfection using TiO_2 is still very long (7 hours). The increment of disinfection rate depends mainly on TiO_2 concentration and initial contaminant concentration. Theoretically, the concentration of TiO_2 can be increased as much as it is needed, but this concentration would depend on the solution used to create the film (% W/V and repetitions of coating) and how much TiO_2 can be attached to the PET walls.

The time of exposure needed for purification depends on the initial time of exposure, and it is recommended to start in the morning when the advantages of increasing radiation can be used.

The use of TiO_2 film has advantages over SODIS although still it may be less efficient than suspended TiO_2 because when we fix the TiO_2, we reduce the area of catalyst that is in contact to the contaminants.

TiO_2 shows advantages in reducing the disinfection time for bacterial contaminants and eliminating chemicals that simple solar disinfection does not degrade.

According to the results obtained in the storage time test, recontamination in simple SODIS bottles was observed during dark conditions when water disinfection occurs under low radiation. It means that effective bacterial inactivation was not reached. In the other hand, TiO2 coated bottles do not present recontamination signal as stable bacteria concentrations were observed during more than a week.

Safe handling of $HClO_4$ and the tedious work required to coat the bottles (in order to avoid the formation of bubbles on the film surface) makes this process, although simple, not recommended for non trained people. This process could be recreated in the villages by medical technicians who have some chemical handling training or by the villagers, after safety rules training. Futures research should contemplate the use of other acids in the coating process that are less dangerous to handle.

Before the use of SODIS or TiO2 in any location, measurement of contaminants concentrations is needed to ensure that they can be removed with the process to be utilized. In the case of arsenic, it is important to know if the water contains enough Iron (III) to react with arsenate.

The quality and durability of the TiO2 on the bottles depend on quality of the work performed in the coating process. Some bottles have not shown signs of TiO_2 film deterioration after several uses (more than six times), others after three uses. In general, TiO2 coated bottles can be used until they lose their film.

Although water purified by TiO_2 has not shown signs of recontamination in more than a week, it is recommended to drink water soon anyway. Bad handling of the bottles (open the bottles and drink some water or touch the interior of the bottle with hands) can definitely change this time. As a general recommendation, if the water is not going to be consumed immediately, keep the exposure to solar radiation until the day or night it is going to be consumed. Further research is needed on safe storage times with this method.

In general, the TiO_2 approach is very promising for remote areas for destruction of a variety of contaminants in, and storage of, drinking water. It appears that the coating process can be undertaken locally with some training and that recycled bottles can be used. Additional research is needed to refine the process and to test for maximum storage times.

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