RESEARCH ARTICLE

Solar light (*hv*) and H₂O₂/*hv* photo-disinfection of natural alkaline water (pH 8.6) in a compound parabolic collector at different day periods in Sahelian region

J. Ndounla[1,2] · **C. Pulgarin**[1]

Abstract The photo-disinfection of natural alkaline surface water (pH 8.6±0.3) for drinking purposes was carried out under solar radiation treatments. The enteric bacteria studied were the wild total coliforms/*Escherichia coli* (10^4 CFU/ml) and *Salmonella* spp. (10^4 CFU/ml) naturally present in the water. The photo-disinfection of a 25-l water sample was carried out in a solar compound parabolic collector (CPC) in the absence and in the presence of hydrogen peroxide (H_2O_2). The addition of H_2O_2 (10 mg/L) to the sample water was sufficient to enhance the photo-disinfection and ensure an irreversible lethal action on the wild enteric bacteria contents of the sample. The inactivation kinetic of the system was significantly enhanced compared to the one carried out without H_2O_2 addition. The effect of the solar radiation parameters on the efficiency of the photo-disinfection were assessed. The pH has increased during the treatment in all the photo-disinfection processes (*hv* and H_2O_2/*hv*). The *Salmonella* spp strain has shown the best effective inactivate time in alkaline water than the one recorded under acidic or near-neutral conditions. The evolution of some physico-chemical parameters of the water (turbidity, NO_2^-, NO_3^-, NH_4^+, HPO_4^{2-}, and bicarbonate (HCO_3^-)) was monitored during the treatment. Finally, the possible mechanistic process involved during the enteric bacteria inactivation was suggested.

Keywords Solar photo-disinfection · H_2O_2 · Alkaline water · Enteric bacteria · Inactivation · Compound parabolic collector

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Introduction

Pathogenic enteric bacteria are related to the outbreak of several water-borne diseases (e.g., diarrhea, cholera, dysenteries, and typhoid) in developing countries. This microbial contamination of water sources by farming, breeding, reduces the amount of potable drinking water and increases waterborne diseases outbreak. The use of solar radiation to disinfect drinking water has been successfully evaluated by several authors under the solar disinfection (SODIS) process (Sommer et al. 1997; Sobsey 2002; Boyle et al. 2008; Marques et al. 2013). Burkina Faso, like many other developing countries in Sub-Saharan Africa, is situated in the latitude lines of 30° N and 30° S and receives about 2,000 to 3,000 h of solar illumination annually. This energy could be productively used to improve the solar disinfection of drinking water. SODIS implies the synergistic effect of sunlight and temperature (Wegelin et al. 1994). Clinical field trials on the evaluation of the efficiency of SODIS towards the reduction of occurrences of diarrhea have been conducted in Kenya (Conroy et al. 2001; Du Preez et al. 2011), India (Rose et al. 2006), Iran (Mahvi 2007), and Cambodia (McGuigan et al. 2011). The development of an enhanced solar disinfection process in the Sahelian region could be useful to efficiently solve the problem of potable drinking water scarcity.

The enhancement of the SODIS efficiency has been reported by several authors, with the aim to develop a low-cost process capable of producing a larger volume in less time than

the 1–2 L in 6 h proposed by SODIS references (Reed 2004; Rincon and Pulgarin 2006; Ubomba-Jaswa et al. 2010; Marques et al. 2013). SODIS enhancement with hydrogen peroxide addition in water (10 mg/L of H_2O_2) or by photo-Fenton (hv/H_2O_2/Fe^{2+}) process has been considered by some authors (10 mg/L of H_2O_2 and 0.6 mg/L of added iron (Spuhler et al. 2010)) to be an affordable and efficient process which could be used to speed up the SODIS process and to increase the volume of water produced (Sciacca et al. 2010; Spuhler et al. 2010; Rodriguez-Chueca et al. 2012). H_2O_2 can directly attack the cellular membrane, initiating lipid peroxidation chains that increase membrane permeability and affect the viability of the cells (Halliwell and Chirico 1993; Spuhler et al. 2010). Fenton and related systems encompass reactions of peroxides (usually H_2O_2) with metal ions leading to the formation of reactive oxygen species (ROS) and reactive radical species (Eq. 1). These metal ions are mostly transition metals which could be found naturally or due to industrial activities in natural waters such as manganese, zinc, chromium, copper, iron, etc. In most of the research carried recently on Fenton and photo-Fenton, the metal ion involved in the reaction is iron (Safarzadeh-Amiri et al. 1996; Cho et al. 2004; Bandala et al. 2012; Rodríguez-Chueca et al. 2013). Therefore, the catalytic Fenton reaction in the dark, generally active at acidic pH, is known to favor the generation of the hydroxyl radicals ($^{\cdot}$OH) and Fe^{2+} ions. OH$^{\cdot}$ radical is highly oxidant and has a lethal action on the enteric bacteria.

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH^- + ^{\cdot}OH \qquad (1)$$
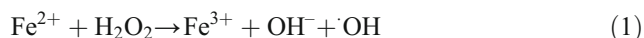
Surface water contains a large amount of natural organic matter (NOM). This NOM can form photo-active Fe^{3+} complexes even at neutral and basic pH. In the presence of soluble Fe^{3+}-organo-complex, the solar light enables the formation of Fe^{2+} required for the catalytic cycle and an oxidized organo-complex. NOM is also able to interact as a photosensitizer, leading to the production of ROS ($^{\cdot}$OH, $HO_2^{\cdot}$, $O_2^{\cdot-}$) (Canonica et al. 1995). The photo-Fenton process was firstly more efficiently been used under acidic conditions (pH 2.5–3) for biorecalcitrant chemical compound degradation (Herrera et al. 1998; Sarria et al. 2005; Kenfack et al. 2009; Malato et al. 2009). After the first records of the efficiency of photo-Fenton disinfection at near-neutral pH (Rincon and Pulgarin 2006), several authors have conducted investigations to evaluate it in natural water and some at neutral pH (Moncayo-Lasso et al. 2009; Sciacca et al. 2010; Ndounla et al. 2013). The photochemical reduction of nitrite or nitrate and the chemical oxidation of ammonia could lead to OH$^{\cdot}$ radical generation (Eqs. 2–3) (Kotzias et al. 1987; Fanning 2000; Brito et al. 2010). The decomposition of ammonia can occur through direct oxidation with the hydroxyl radical so that forming various compounds of nitrogen among them, the nitrogen gas, nitrogen Oxides (NO_X) and ionic compounds such as nitrite and nitrate (Huang et al. 2008). Therefore, the redox activities of the nitrogen compounds in the water during photo-disinfection could significantly affect the rate of the photo-inactivation.

$$NO_3^- + H_2O + hv \rightarrow NO_2^- + OH^- + OH^{\cdot} \qquad (2)$$

$$NO_2^- + H_2O + hv \rightarrow NO + OH^- + OH^{\cdot} \qquad (3)$$
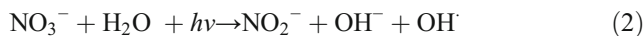
Inorganic ions can have an interfering effect on the Fenton reagent. Depending on their concentrations, Fenton and photo-Fenton oxidations of organic compounds are inhibited in varying degrees by inorganic ions (e.g., phosphate, sulfate, chloride) (Pignatello et al. 2006). Phosphate has a doubly detrimental effect by precipitating iron and by scavenging hydroxyl radicals (Malato et al. 2009).

Domestic, agricultural, and industrial activities favor the introduction in the water cycle of some inorganic ions such as bicarbonate (HCO_3^-), carbonate (CO_3^{2-}), SO_4^{2-}, S^{2-}, F^-, HPO_4^{2-}, NO_2^-, NO_3^-, and NH_4^+. These ions can either lead to the precipitation of iron scavenging of $^{\cdot}$OH or coordinate to dissolve Fe^{3+} and Fe^{2+} in more or less unreactive complexes (Pignatello et al. 2006). These reactions, when they occur, can affect the photo-inactivation process.

The current study is the first conducted on alkaline surface water with adding H_2O_2 at field scale in a compound parabolic collector (CPC) solar reactor. Its aim is to evaluate the efficiency of the H_2O_2-enhanced photo-disinfection (possible photo-Fenton) treatment in theoretically unfavorable alkaline conditions. The pH evolution during photo-disinfection and the effect of the solar radiation parameters (day period of illumination, irradiance and dose) on the efficiency of the photo-disinfection is assessed. The impact of some inorganic ions present in the natural water sample on the efficiency of the photo-disinfection process is also evaluated.

Materials and methods

Physico-chemical measurements and chemical reagents

The HACH DR/2000 spectrophotometer methodologies used in this study to characterize some physico-chemical components of the water sample (turbidity, iron, nitrite, nitrate, phosphate, sulfate, fluoride, sulfide, and ammoniac) follow the guidelines of the Standard Methods for Examination of Water (HACH 2001). The HACH methods used for the determination of each component and its detection limit are presented in Table 1. However, the bicarbonate and carbonate ion concentrations were determined by titration. A universal meter WTW 340i equipped with a WTW SenTix 41-3 probe was used to measure the pH and temperature. The H_2O_2 concentration was followed during the experiments by a Peroxide Merckoquant (Merk) test with a detection limit of around 0.5 mg/L. Microbiology Chromocult® (Merck KGaA) was used for bacterial plating. Growth media were poured into a pre-sterilized Petri dish,

Table 1 Summary of the HACH analytical methods used to characterize some components of the water sample (HACH 2001)

Components	HACH DR/2000 methods/programs	Detection ranges
Turbidity	Program 750 (wavelength 450 nm)	0–450 NTU (nephelometric turbidity units)
Total iron	FerroVer Method, program 265 (Powder Pillows, wavelength 510 nm)	0–3.00 mg/L
Nitrate	High range (HR), program 355 or the cadmium reduction method (wavelength 500 nm)	0–30.0 mg/L NO_3^--N
Nitrite	Low range (LR), program 371 or diazotization method (wavelength 507 nm)	0–0.300 mg/L NO_2^--N
Ammonia	Nessler method, program 380 (wavelength 425 nm)	0–2.50 mg/L NH_3-N
Phosphate	PhosVer 3 (ascorbic acid) method, program 490 (Powder Pillows, wavelength 890 nm)	0–2.50 mg/L PO_4^{3-}
Sulfate	SulfaVer 4 method, program 690 (Powder Pillows, wavelength 450 nm)	0–70 mg/L SO_4^{2-}
Fluoride	SPADNS method, program 190 (wavelength 580 nm)	0–2.00 mg/L F^-
Sulfide	Methylene blue method, program 690 (wavelength 665 nm)	0–0.600 mg/L S_2^-

92×16 mm (Sarstedt AG). Hydrogen peroxide, 30 % (AnalaR Normapur, VWR), was used to prepare the Fenton reagent, and hydrochloric acid fuming (HCl), 37 % (Fluka Analytical, Sigma-Aldrich®), was used for glass reactor cleaning.

Characteristics of the water sample

The water used during the experiments was collected from April to May 2011 (dry season) at dam 3 in Ouagadougou, Burkina Faso. Ouagadougou is located at 12° 21′ 26″ latitude north and 1° 32′ 7″ longitude west and receives approximately 2,500 h of solar radiation per year. The experiments were conducted under direct solar exposure at the International Institute for Water and Environmental Engineering (2iE), Ouagadougou. The water sample is locally used by part of the local population for household purposes (cooking, drinking, and washing) and has a pH 8.6±0.3. Its physico-chemical parameter concentrations are presented in Table 2. The enteric bacteria concentration in the water were approximately 10^4 CFU/mL for each entity involved in

Table 2 Physico-chemical characteristics of the water sample measured before and after the photo-Fenton disinfection treatment

Parameters	Before the treatment	After the treatment
Temperature (°C)	28–29.5±0.5	–
pH	8.6±0.3	–
Turbidity (NTU)	8±3	8±3
Total Iron (mg/L)	0.10±0.05	0.11±0.06
Nitrite (NO_2^-) (mg/L)	0.011±0.003	0.012±0.002
Nitrate (NO_3^-) (mg/L)	3.26±0.2	4.02±0.3
Ammonia (NH_4^+) (mg/L)	0.11±0.05	0.17±0.04
Sulfate (SO_4^{2-}) (mg/L)	12±1	12±1
Sulfide (S^{2-}) (mg/L)	0.007±0.002	0.008±0.001
Fluoride (F^-) (mg/L)	0.50±0.02	0.50±0.03
Phosphate (PO_4^{2-}) (mg/L)	0.07±0.01	0.15±0.02
Bicarbonate (HCO_3^-) (mg/L)	148.10±0.05	137.86±0.04
Carbonate (CO_3^{2-}) (mg/L)	3.8±0.1	3.7±0.2

this study (total coliforms/*Escherichia coli* and *Salmonella* spp.). The sampling collection was performed 1 h before the experiment in plastic jerricans (20 L).

Bacterial strain and growth media

The wild bacterial strain monitored in this study was the fecal indicator bacteria coliforms/*E. coli* and *Salmonella* spp. Microbiology Chromocult® (Merck KGaA) was used for bacterial plating. Chromocult is a selective and differential growth media. It selectively inhibits growth of the non-enteric bacteria. As experiments were conducted with natural water, considering their initial enteric bacteria concentration, no dilution was realized during the bacterial plating. Sample water (100 μL) was inoculated into the growth medium. Considering the selectivity of Chromocult, the detection limit of enteric bacteria was zero colony growths observed in the plate. The differential nature of the medium permits the distinction of *Salmonella* spp. (colorless), *E. coli* (purple and pink), and the blue- and salmon-colored colonies of other coliform bacteria. However, in order to emphasize the decrease of the total coliforms, all the *E. coli* observed and other coliforms counted are presented together in this study as total coliforms/*E. coli*.

The experiments in the compound parabolic collector

All the experiments (solar radiation and photo-Fenton) were conducted under direct sunlight in a CPC (Ndounla et al. 2014). The CPC is a SOLARDETOX ACADUS-2003 batch photoreactor device model delivered by Ecosystem SA (Barcelona, Spain). Twenty-five liters of surface water was disinfected during each treatment at constant flow (2 L/min). Preliminary experiments were performed to evaluate the efficient exposure duration (4 and 2 h, respectively) which could be proposed to the population concerned if the implementation of the photo-treatment is taken into account. Afterwards, the photo-disinfection was carried out in the CPC during six different time intervals: (i) 8 am to 12 pm (8–12 h), (ii) 10 am

to 12 pm (10–12 h), (iii) 12 pm to 2 pm (12–14 h), (iv) 1 pm to 3 pm (13–15 h), (v) 2 pm to 4 pm (14–16 h), and (vi) 3 pm to 5 pm (15–17 h) for both processes (direct solar radiation and solar light enhanced with H_2O_2). The evaluation of the influence of direct solar radiation parameters (irradiance and cumulated dose) on the efficiency of both photo-disinfection processes is reported. The solar UV radiation was then reported during the experiments by a UV-A radiometer ACADUS 85 UV fixed on the CPC photoreactor. Solar irradiance intensity per square meter ($W.m^{-2}$) was then monitored between 300 and 400 nm. During the exposure, pre-sterilized glass flasks of 100 mL were used at regular time intervals (0, 10, 20, 30, 45, 60, 90, 120, 150, 180, and 240 min) to collect the treated water samples to be analyzed. One hundred microliters was taken with a micropipette from the flask and poured into a Petri dish plate containing growth media (Chromocult agar). Plates were incubated for 18–24 h at 37 °C and the colonies counted with a colony counter (Stuart SC6 Colony Counter). To check the durability of photo-disinfection after exposure, all the flasks were further kept in the dark for post-irradiation controls after 24 h. Dark control tests were conducted simultaneously in the dark on 100 mL of samples containing 10 mg/L of H_2O_2. The concentration of some physico-chemical parameters of the water (HCO_3^-, CO_3^{2-}, SO_4^{2-}, S^{2-}, F^-, HPO_4^{2-}, NO_2^-, NO_3^-, NH_4^+, turbidity, and the total iron content) was evaluated before and after the treatment. The pH and temperature evolution during the treatments were successively recorded. Each experiment was repeated three times to ensure reproducibility. The Wolfram Mathematica 8.0 and MS Excel programs were used for data analysis and graph fitting.

Results and discussion

Evolution of some physico-chemical characteristics of the water sample

The surface water used in this study was collected from Ouagadougou's dam 3 during the dry season. Table 2 presents its main physico-chemical characteristics before and after photo-treatment. Water composition has a great influence on the photo-disinfection treatment, and light penetration is minimal in highly turbid water (Joyce et al. 1996; Kehoe et al. 2001). To realize an efficient photo-disinfection, it is recommended to conduct it in water with less than 30 NTU turbidity (Byrne et al. 2011). The water treated in this study was clear with only 8±3 NTU. The initial water temperature ranged between 28 and 29.5±0.5 °C, and its pH was 8.6±0.3. The impact of the alkalinity of the natural water treated in this study on the efficiency of the photo-disinfection process enables us to produce the record of the first data in such conditions. The Sahelian African soils are ferruginous, which leads permanently to natural iron contents in the water. Considering the presence of this iron and other metal ions such as copper in the sample water, after the addition of H_2O_2 (10 mg/L), it was possible to the photo-Fenton reaction to take place in the system during the exposure to solar radiation.

The concentration of mineral nitrogen compounds (nitrate, nitrite, ammonia) in the surface water used in this study was far below the World Health Organization (WHO) norms for drinking water which is 3 mg/L for nitrite and 50 mg/L for nitrate. No guideline is given for ammonia (WHO 2011). The variations of the concentration of these nitrogen compounds during the photo-treatment were not relevant as their increased has remained far below the WHO restriction. The concentration of sulfate, sulfide, fluoride, and phosphate was under the restriction of the WHO guidelines for drinking water (WHO 2011). Photo-disinfection did not significantly affect their variation during the treatments (Table 2). Rincon and Pulgarin (2007) have reported that the mixture of these components (sulphate, sulfide, fluoride and phosphate) at high concentrations can positively influence the kinetic of the photo-disinfection, while when individually taken into account, they have a negative impact on the photocatalytic process (Pignatello et al. 2006; Rincon and Pulgarin 2007).

The HCO_3^- concentration was extremely high in the surface water treated in this study, and the presence of CO_3^{2-} ions was also recorded (Table 2). It should be taken in account that the carbonates are generated from bicarbonates in water, when its pH is greater or equal to 8.3. As a buffer complex, HCO_3^-/CO_3^{2-}, the bicarbonate and carbonate ions could greatly influence the photocatalytic process (Pignatello et al. 2006; Rincon and Pulgarin 2007) by their quenching effect on the hydroxyl radicals in a H_2O_2/light system (Kochany and Lipczynskakochany 1992). Relevant differences were not noticed in their concentration before and after photo-disinfection. Due to their buffering effect, they have also a great impact on the pH variation during photo-disinfection.

Enteric bacteria inactivation in alkaline water by solar light ($h\nu$), $H_2O_2/h\nu$ system, and dark control (H_2O_2/obs)

The inactivation of wild enteric bacteria contents of the natural surface water in the CPC during the photo-disinfection treatment by solar radiation ($h\nu$), $H_2O_2/h\nu$, and dark control (H_2O_2/obs) is presented in this paper. The evaluation of the iron content of the natural water before the exposure has led to the determination of an initial concentration of 0.10±0.05 mg/L. The H_2O_2 addition in the sample water for the system $H_2O_2/h\nu$ before the exposure to solar radiation was 10 mg/L. The open symbols represent the total coliforms/*E. coli* and the full symbols are for *Salmonella* spp. The traces (△) and (▲) represent Fenton evaluation conducted in 100-mL glass flasks kept in the dark. The traces (○) and (●) illustrate the enteric bacteria decreased under solar radiation, and (□) and (■) present the inactivation under photo-Fenton treatment. The decrease of both enteric bacteria under both photo-disinfection methods follows the first-order kinetic (McGuigan et al. 1998).

Solar radiation system

The generation of the ROS from NOM, which intervene together with the direct action of photons in the lethal attack of the bacteria, is highly influenced by the light intensity or irradiance (W m^{-2}) (Reed 2004; Ndounla et al. 2014). However, the solar inactivation of wild enteric bacteria of natural surface water at different daytime intervals is not only influenced by average irradiance (AVI) but also by the temperature increase recorded during each exposure period in Figs. 1, 2, and 3, trace (○) and (●). It is noticeable that the synergy between the temperature rise and the AVI and not the cumulated dose significantly affected the photo-disinfection process. Indeed, during the exposure period, 15–17 h, in the presence of very low AVI of 8 W m^{-2} for a cumulated dose of 21 Wh m^{-2} and a temperature of less than 40 °C (Fig. 3f′), none of the enteric bacteria in the water were inactivated under direct solar radiation (Fig. 3f).

During the periods 8–12 h (Fig. 1a) and 14–16 h (Fig. 3e), the temperature increased to approximately 45 °C, and the AVI were 17 and 20 W m^{-2}, respectively. These conditions have led to the total inactivation of both enteric bacteria. One hundred fifty minutes (period 8–12 h) and 90 min (period 14–16 h) were required for total coliform/*E. coli* strain inactivation. Two hundred forty minutes (period 8–12 h) and 120 min (period 14–16) were recorded for *Salmonella* spp. strain inactivation. The high dose required during the period 8–12 h for *Salmonella* spp. total inactivation (180 Wh m^{-2}) proved that the availability of high doses does not lead to high inactivation kinetics. Only 80 Wh m^{-2} was required during the exposure period 14–16 h for both enteric bacteria's total inactivation. The dose recorded for the successive exposure periods 10–12 h (Fig. 2b), 12–14 h (Fig. 2c), and 13–15 h (Fig. 2d) was, respectively, 90, 50, and 50 Wh m^{-2} for both enteric bacteria's inactivation in

approximately 90 min in all the cases. This result again confirms the previously recorded observations about the irrelevant impact of the dose in the photo-disinfection of drinking water sources.

The AVI recorded during the successive daytime periods 10–12, 12–14, and 13–15 h was, respectively, 29, 27, and 28 W m^{-2}. These high AVI have approximately the same order of magnitude as the ones recorded in the Sub-Sahelian region during the dry season (Kenfack et al. 2009). Associated to the temperature rise of more than 40 °C during the first hour of exposure, in almost all the cases, they have led to the total inactivation of both enteric bacteria strains in approximately 90 min. Therefore, high AVI induced a similar required time (and dose) for total inactivation. The total coliform/*E. coli* strain was the only one to be inactivated in 60 min during the exposure period 13–15 h. The temperature increase at the beginning of the exposure has certainly significantly impacted the high inactivation kinetic of the total coliform/*E. coli* strain observed. However with only one bacterial strain inactivated, the treated water remain unsafe for drinking purposes and should go under improved photo-disinfection or exposure under high solar radiation to be safe.

It is generally accepted that the photonic flux greatly affects the ROS production and the concomitant oxidative stress in the bacteria, leading to inactivation or death, while the increased temperature inactivates the enzymes which were supposed to protect them from this stress (Cabiscol et al. 2000). As neither the temperature increase nor the ROS generation through the photonic action of irradiance was sufficiently available during the exposure period 15–17 h, the steady state of the active enteric bacteria recorded in the water at the end of the exposure could be attributed to the absence of lethal oxidative stress. These results highlight the fact that it is the synergetic effect of the photonic and the thermal parameters of the solar radiation (intense solar radiation coupled to increased temperature
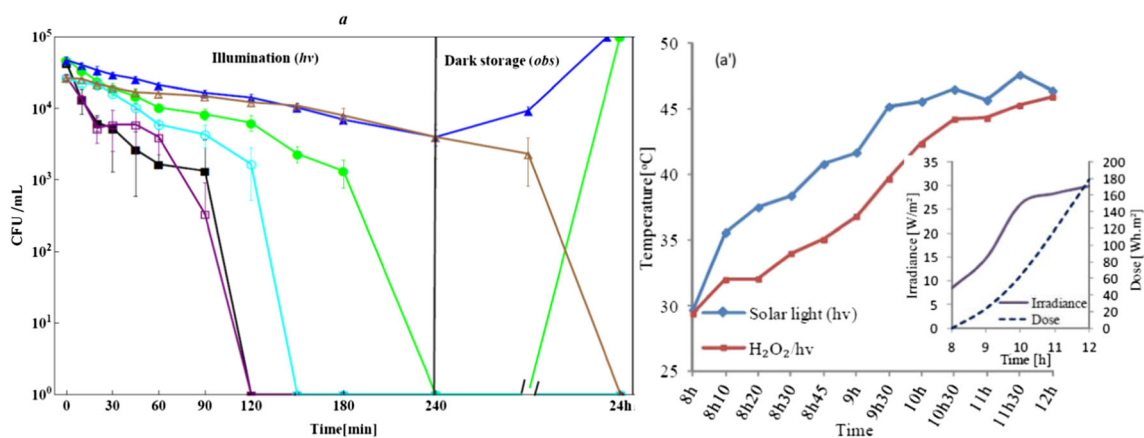

Fig. 1 (*a*) Inactivation of wild enteric bacteria in natural surface water carried out from 8 to 12 h under direct solar illumination (*hv*). Post-irradiation events (24-h dark storage). pH 8.6±0.3, natural iron content (Fe): 0.10±0.05 mg/L, addition of H$_2$O$_2$ (10 mg/L) in the water for the enhanced photo-disinfection process. Total coliforms/*E. coli* (□) and *Salmonella* spp. (■) under enhanced photo-disinfection (H$_2$O$_2$/*hv*), total coliforms/*E. coli* (○), and *Salmonella* spp. (●) under direct solar radiation (*hv*), total coliforms/*E. coli* (△), and *Salmonella* spp. (▲) in the dark control system (H$_2$O$_2$/*obs*). (*a′*) Evolution of the water temperature [*T*, °C] during both treatments (*hv* and H$_2$O$_2$/*hv*). *Inset*: solar irradiance [W m^{-2}, (—)] and cumulated total dose [Wh m^{-2}, (---)] available during the experiment
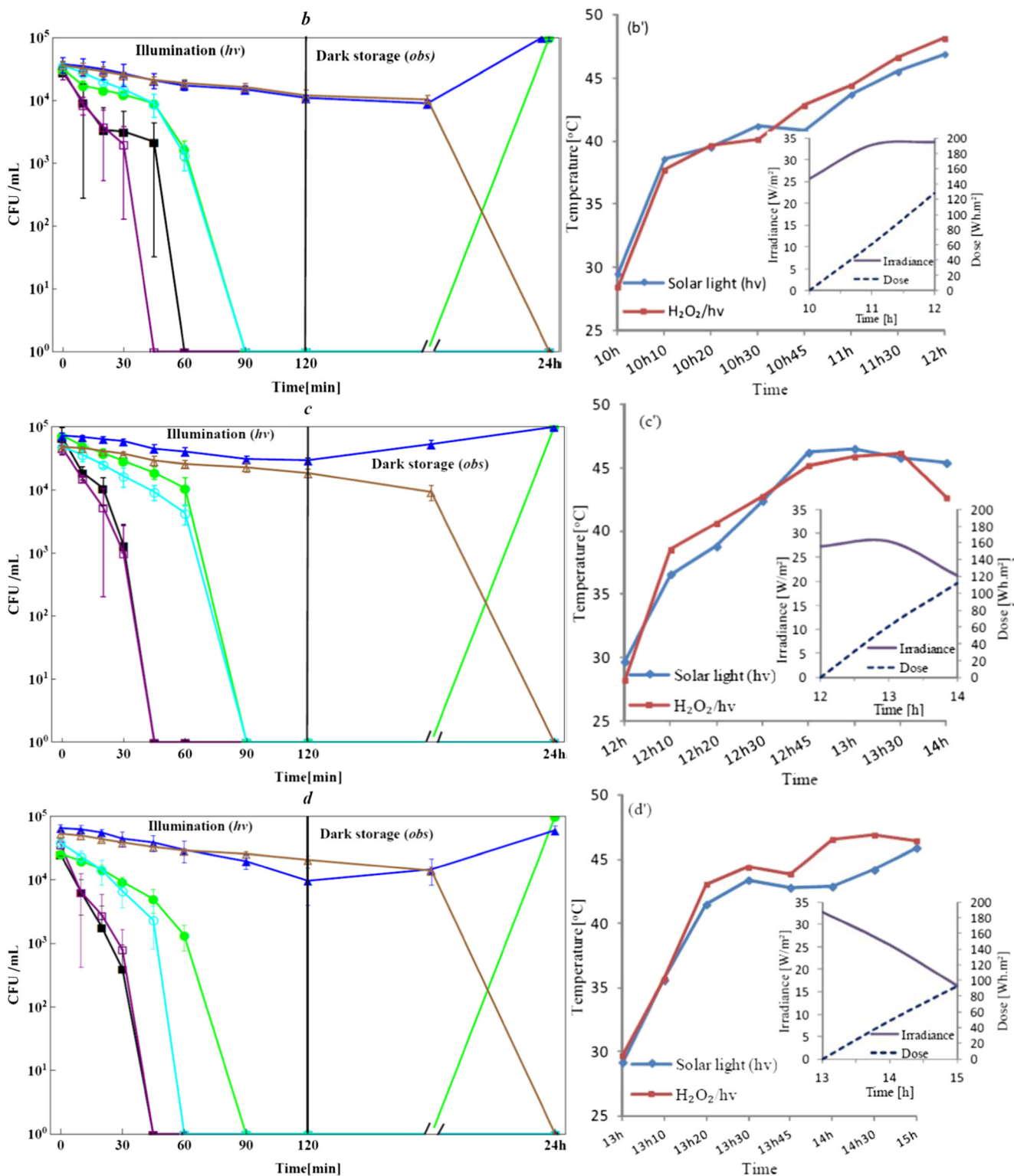
Fig. 2 (*b–d*) Inactivation of wild enteric bacteria in natural surface water carried out from 10–12, 12–14, and 13–15 h, respectively, under direct solar illumination (*hv*). Post-irradiation events (dark storage during 24 h). pH 8.6±0.3, natural iron (Fe) content: 0.10±0.05 mg/L, addition of H_2O_2 (10 mg/L) in the water for the enhanced photo-disinfection process. Total coliforms/*E. coli* (□) and *Salmonella* spp. (■) under enhanced photo-disinfection (H_2O_2/hv), total coliforms/*E. coli* (○), and *Salmonella* spp. (●) under direct solar radiation (*hv*), total coliforms/*E. coli* (△), and *Salmonella* spp. (▲) in the dark control system (H_2O_2/obs). (*b'–d'*) Evolution of the water temperature [*T*, °C] during both treatments (*hv* and H_2O_2/hv). *Inset*: solar irradiance [W m^{-2} (──)] and cumulated total dose [Wh m^{-2} (---)] available during the experiment
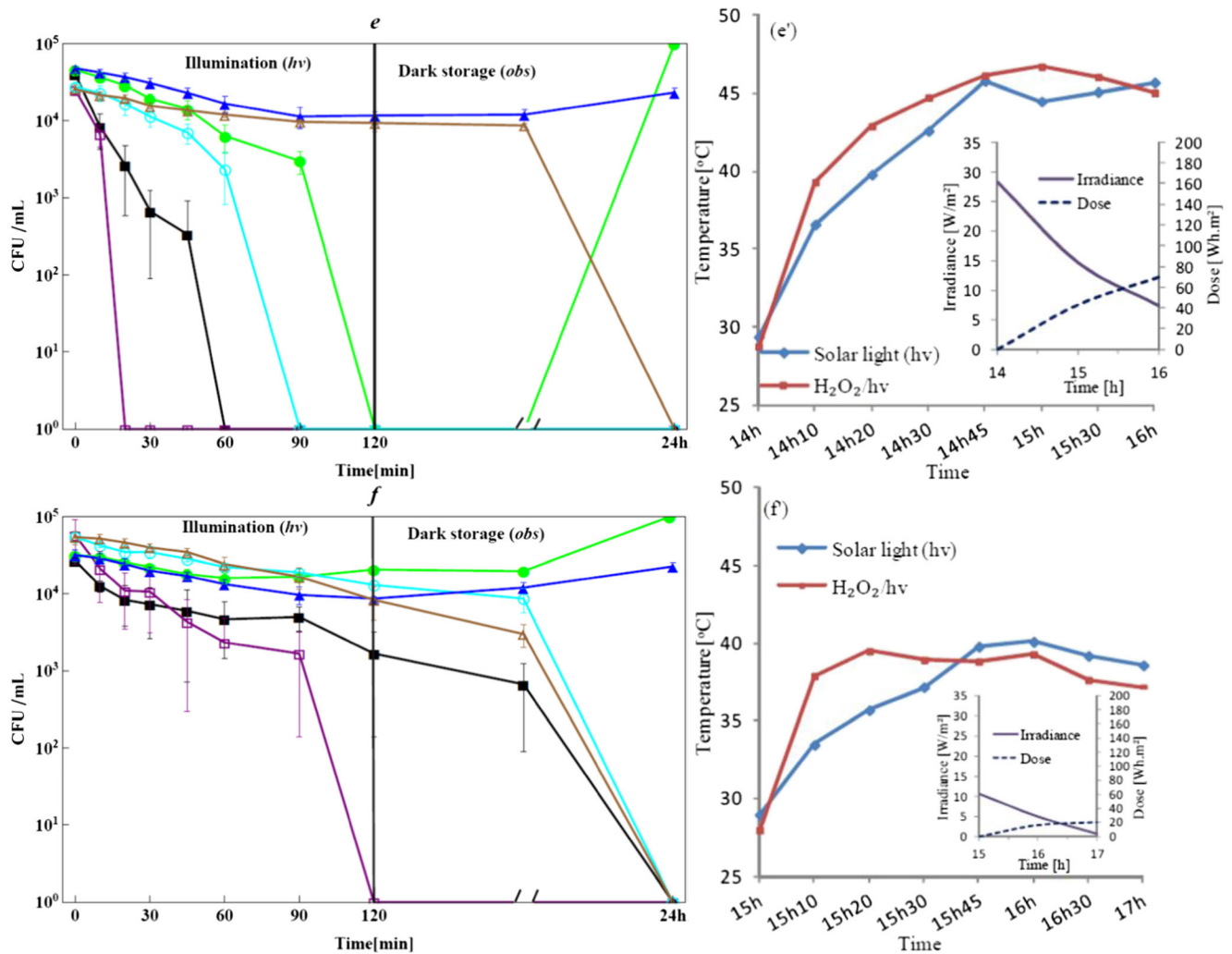
Fig. 3 (*e*, *f*) Inactivation of wild enteric bacteria in natural surface water carried out from 14–16 and 15–17 h, respectively, under direct solar illumination (*hv*). Post-irradiation events (dark storage during 24 h). pH 8.6±0.3, natural iron (Fe) content: 0.10±0.05 mg/L, addition of H_2O_2 (10 mg/L) in the water for the enhanced photo-disinfection process. Total coliforms/*E. coli* (□) and *Salmonella* spp. (■) under enhanced photo-disinfection (H_2O_2/*hv*), total coliforms/*E. coli* (○), and *Salmonella* spp. (●) under direct solar radiation (*hv*), total coliforms/*E. coli* (△) and *Salmonella* spp. (▲) in the dark control system (H_2O_2/*obs*). (*e′*, *f′*) Evolution of the water temperature [*T*, °C] during both treatments (*hv* and H_2O_2/*hv*). Inset: solar irradiance [W m^{-2} (——)] and cumulated total dose [Wh m^{-2} (---)] available during the experiment

to approximately 45 °C), which lead to the enteric bacteria inactivation during the solar disinfection (Wegelin et al. 1994; Sommer et al. 1997; McGuigan et al. 1998). Unfortunately, during the post-irradiation storage in the dark, the *Salmonella* spp. strains recovered their viability and grew to more than their initial level in all the treated water. This regrowth of *Salmonella* spp. is the negative side of the bare solar disinfection. It revealed that their inactivation was not irreversible. It will be necessary to evaluate whether the enhanced solar disinfection with H_2O_2 addition (H_2O_2/*hv*) could ensure the irreversibility of all the enteric bacteria inactivation.

Enhanced photo-disinfection system (H_2O_2/hv)

Fast inactivation kinetics were recorded in all the enhanced by H_2O_2 addition systems (H_2O_2/*hv*) (Figs. 1, 2, and 3; trace (□)

and (■)). Comparatively to the situation observed under direct solar radiation (only *hv*) in the previous section, it could be assumed that the generation of highly oxidant hydroxyl radical •OH and other ROS has occurred in the system during the exposure. This ROS (•OH, HO$_2$•, O$_2$•⁻) generation from photosensitized NOM (Canonica et al. 1995) and Fe^{2+} regeneration from photo-active Fe^{3+}-organo-complex (Pignatello et al. 2006) has certainly contributed to enhancement of the inactivation kinetics observed here at alkaline pH. Considering the natural iron content of the sample water and the photo-Fenton generation of •OH in the medium could be part of the mechanistic pathway involved during the photo-treatment. Knowing that H_2O_2 is relatively stable (unlike •OH) and uncharged (unlike O2•⁻), it can penetrate the cell membranes and diffuse into cells (Jang and Imlay 2010; Spuhler et al. 2010). After crossing over the cell cytoplasm and penetrated inside

the cell, the H_2O_2 can react with the intracellular free or loosely bound iron through intracellular Fenton reaction and generate $^{\bullet}OH$. It can also oxidize the iron sulfur clusters ([4Fe-4S]) and the release Fe^{3+}, which if reduced to Fe^{2+}, can contribute to intracellular Fenton reactions (Jang and Imlay 2008; Spuhler et al. 2010). However, it can be assumed that the oxido-reduction of the nitrogen components of the sample water during the irradiation has generated also additional $^{\bullet}OH$ as described by Fanning (2000) and Brito et al. (2010) which may greatly enhanced the inactivation kinetic of the process.

The high photon-flux generated by the AVI available during the exposure periods 10–12, 12–14, and 13–15 h, which was, respectively, 29, 27, and 28 W m^{-2}, associated to the temperature rise of more than 40 °C during the first hours of exposure led to a drastic inactivation of both enteric bacteria in approximately 45 min in all cases (Fig. 2, trace (□) and (■)). However, during the exposure period 10–12 h, the *Salmonella* spp. total inactivation took a little more time. The effect of the low solar radiation available in the morning and afternoon was highlighted by the recorded irradiance: 17 and 20 W m^{-2}, respectively, for the exposure periods 8–12 and 14–16 h (Figs. 1, 2, and 3). A relevant inactivation kinetics was, however, recorded in both cases. The total inactivation was achieved for both enteric bacteria after 120 min during the exposure period 8–12 h. The fast inactivation in 20 min of total coliforms/*E. coli* strain was recorded for the exposure period 14–16 h, while that of *Salmonella* spp. was achieved after 60 min.

The inactivation of the weakened strains of total coliforms/*E. coli* was noticed at the end of the enhanced photo-disinfection (H_2O_2/hv) process, during the exposure period 15–17 h, in contrast to the negative results recorded for the same period under bare solar radiation (Fig. 3, trace (□) and (■)). Even though the AVI was very low during this period (8 W m^{-2}), no regrowth was recorded after the dark post-irradiation storage (24 h). As noticed in the previous section, the cumulated dose of the solar radiation did not significantly influence the inactivation process. The dose recorded for the total inactivation of both enteric bacteria during the exposures was approximately 50 Wh m^{-2} for the periods 12–14, 13–15, and 14–16 h. It was 120 and 60 Wh m^{-2}, respectively, for the exposure periods 8–12 and 10–12 h. It is important to notice the residual effect of the H_2O_2 in the dark control system (H_2O_2/obs) during the storage. However, the remaining H_2O_2 amount (3–4 mg/L) in the treated water disappears completely from the water after 2 or 3 days of storage. This H_2O_2 depletion was also reported in our previous paper (Ndounla et al. 2013).

Dark control system (H_2O_2/obs)

As can be observed in all the traces (△) and (▲) in Figs. 1, 2, and 3, neither the total coliform/*E. coli* strain nor that of *Salmonella* spp. decreased of one magnitude order was noticed during the exposure to the Fenton system. However, it is surprising to notice that the Fenton process (Eqs. 1–2) led to the total inactivation of the weakened total coliform/*E. coli* strain during the subsequent 24-h dark storage. In contrast, the more resistant strain of *Salmonella* spp. (Berney et al. 2006) attained a higher active bacteria population than that present at the beginning of the treatment during the same dark storage time.

Post-irradiation events

Post-irradiation events after solar radiation disinfection

The *Salmonella* spp. conversion from nonculturable to culturable strains was noticed in all the waters which were disinfected for 2 h under direct solar radiation. After the recovery of their culturability during the dark storage, they increased to more than their initial contents. These *Salmonella* spp. strains' recovery during favorable conditions leads to the assumption that the effect of solar radiation was bacteriostatic and not bactericidal (Rincon and Pulgarin 2007). A longer exposure time is therefore needed to ensure the bactericidal effect of the bare solar radiation on *Salmonella* spp. strains to ensure its irreversible inactivation. The total coliform/*E. coli* strain was not inactivated during the exposure period 15–17 h. However, after being weakened by illumination, it was totally inactivated during the subsequent 24 h of dark storage. In contrast, the remaining *Salmonella* spp. strains took advantage of the favorable conditions of the dark storage to recover their capacity for growth. Such capacity is part of the resistance of *Salmonella* spp. to photo-inactivation (Berney et al. 2006).

Post-irradiation events after enhanced with H_2O_2 photo-disinfection

None of the total coliform/*E. coli* and *Salmonella* spp. strains inactivated under the enhanced photo-treatment (H_2O_2/hv) have recovered viability during storage (Figs. 1, 2, and 3, trace (□) and (■)). No regrowth of any of the strains was observed after the 24 h of dark storage. Considering this irreversible inactivation, it can be assumed that this point of use drinking water treatment process could be efficiently used to produce larger volumes of water in shorter time than required by SODIS bottle system.

pH evolution during the experiments

The pH increases, recorded in all the photo-disinfection (*hv* and H_2O_2/hv) processes as presented in Fig. 4, are in contrast with the decrease report by several authors during the photo-catalytic treatment with TiO_2 (Rincon and Pulgarin 2007; Malato et al. 2009). This increase is probably due to (i) the high concentration of the HCO_3^-/CO_3^{2-} recorded in the natural water treated in this study (Table 2) could significantly
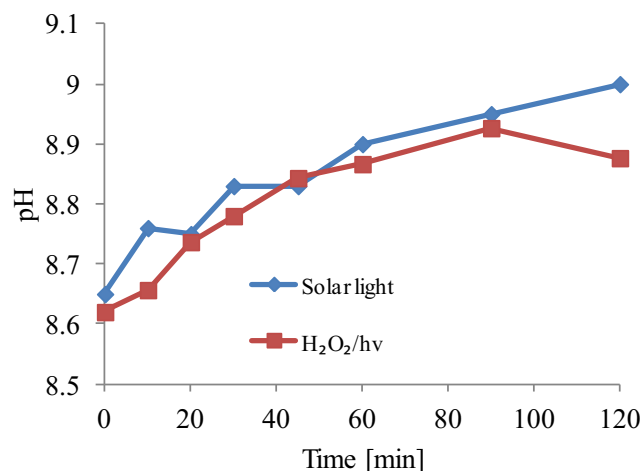
Fig. 4 Evolution of pH during the photo-disinfection treatments: (♦) under solar radiation treatment without H_2O_2 addition (*hv*), (■) under enhanced photo-treatment (H_2O_2/*hv*) with H_2O_2 (10 mg/L)

induce the buffer action of the complex HCO_3^-/CO_3^{2-} which maintains the solution in the alkaline region; (ii) the degradation of the nitrogen component of the water during their photo-degradation, leading to the generation of the OH^- and consequently increased alkalinity (Kotzias et al. 1987; Fanning 2000). However, this pH increase has not negatively affected the inactivation kinetic of the system.

Comparative evaluation of the enteric bacteria inactivation by enhanced photo-disinfection processes at alkaline or near-neutral pH in natural waters

Table 3 presents the initial concentration of some relevant physico-chemical parameters and the microorganisms of the well water used in this study. The presence of several iron species in natural water: soluble (Fe^{2+} or $^{3+}$) and solids (e.g., iron oxides) have lead to heterogeneous conditions for photo-Fenton process. This heterogeneous conditions could take advantage of several pathways: (i)

Table 3 Some characteristics of the well water sample used during the experiments

Parameters	Contents
Turbidity	5±3 NTU
pH	5.4±0.1
Temperature	29±0.1 °C
Dissolve total iron	0.07±0.02 mg/L
Solids total iron	0.23±0.01 mg/L
Wild *E. coli*	10^4 CFU/mL
Wild *Salmonella* spp.	10^5 CFU/mL

NTU nephelometric turbidity units, *CFU/mL* colony-forming unit per milliliter, *°C* degree Celsius, *mg/L* milligram per liter

the high adsorption of iron oxides by bacteria (Spuhler et al. 2010), (ii) the effect of the bacteria siderophores which increases iron dissolution (Stintzi et al. 2000) and leads to high photo-Fenton activity, (iii) the enhancing semi-conductor effect of some types of solid iron oxides on photo-disinfection (Moncayo-Lasso et al. 2008; Mazille et al. 2010), or (iv) the NOM or humic substance complexation with iron to maintain their solubility in solution (Pignatello et al. 2006; Lipczynska-Kochany and Kochany 2008). Ayodele et al. (2012) have efficiently optimized the degradation kinetic of phenolic coumpounds with heterogeneous photo-Fenton process at alkaline pH. This efficiency of the photo-Fenton process at alkaline pH for phenol degradation, bring out the opportunities to involve more research on its efficiency in the disinfection of bacteria in natural alkaline water sources in contrast to the preceding studies who were mostly carried in acid medium (Huang et al. 2008). Cabiscol et al. (2000) reported that the oxidative stress is highly lethal for enteric bacteria, therefore in presence of ·OH radical the optimization of the bacterial inactivation is significantly enhanced leading to the observed results recorded in this study. The ·OH radical occurrence in the water treated in this study has probably follows this two pathways: either through the oxido-reduction of the nitrogen components of the water in the medium or the homogenous (Fe-org) and heterogeneous (Fe oxides) photo-Fenton in alkaline or near-neutral water sources. Figure 5 shows the inactivation curves of the total coliforms/*E. coli* and *Salmonella* spp. by photo-Fenton in well water (W, pH 5.4±0.1) or in surface water (S, pH 8.6±0.3). The solar radiation parameters (irradiance/dose) and temperature rise during the experiments were similar to those presented in the previous section at the same time intervals for both water sources. In contrast to the fact regularly observed at acidic or near-neutral pH, *Salmonella* spp. strains seem to be less resistant to photo-disinfection at alkaline pH. Their total inactivation occurred under several exposure periods in this study at the same time as that of total coliforms/*E. coli* (Fig. 5a–c). The delay (15 min) noticed between the inactivation kinetics of *Salmonella* spp and that of total coliforms/*E. coli* during the exposure period 10–12 h is not worth recording as a relevant fact. However, the *Salmonella* spp strain has shown in general best effective inactivate time in alkaline water than the one recorded under acidic or near-neutral conditions. Knowing that alkaline pH levels (>8) can render free residual chlorination less effective, leading to a significant decrease in bacterial disinfection by chlorination (Marois-Fiset et al. 2013), the opportunity to efficiently disinfect natural alkaline water by photo-disinfection could be a positive alternative for household water disinfection in such cases.
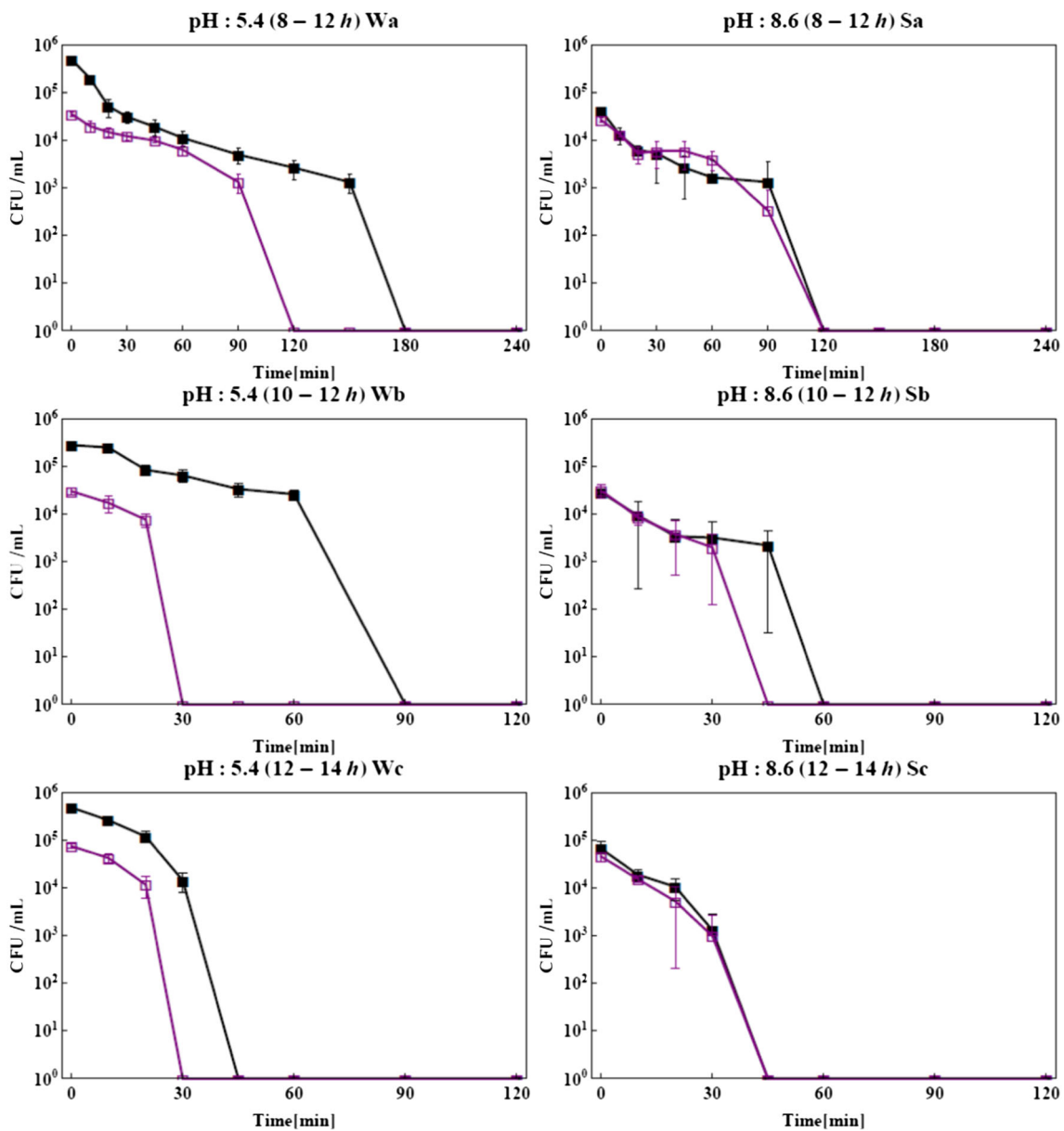
Fig. 5 Wild enteric bacteria inactivation by enhanced photo-disinfection/ photo-Fenton (natural $Fe^{2+/3+}$/H_2O_2/hv) in natural wells (*W*) and surface (*S*) water under direct solar radiation at different time intervals of the day (8–12, 10–12, and 12–14 h). (□) total coliforms/*E. coli*, (■) *Salmonella* spp. Some parameters of the sample water are presented here: the surface water contains 0.10±0.05 mg/L natural iron (Fe) and pH 8.6±0.3, while the well water contains natural iron (dissolved, 0.07±0.02 mg/L of $Fe^{2+, 3+}$ and solid iron oxides (0.23±0.01 mg/L), pH 5.4±0.01. H_2O_2 (10 mg/ L) was added in each sample to initiate the enhanced process

Inactivation mechanisms

Inactivation mechanisms under solar radiation treatments (*hv*)

Dissolved oxygen in the water seems to have a direct impact on the bactericidal action of solar disinfection (Reed 1997). The water recirculation in the CPC during the photo-disinfection process is subjected to high fluctuation following the oxygen availability at different point of the reactor. The NOM present in natural water acts as photosensitizers. Under irradiation, the photosensitizers become electronically excited and react with O_2, leading to ROS such as singlet oxygen ($^1O_2$), superoxide ($HO_2^{\cdot}$/$^{\cdot}O_2^-$), H_2O_2, and $OH^{\cdot}$ radicals (Canonica et al. 1995; Reed 1997). The catalases and other enzymes which should protect the cells from oxidative stress are photo- and thermosensitive. The temperature increase of up to 45 °C during photo-disinfection inactivates these enzymes, leaving the cells susceptible to internal ROS attack and subsequent inactivation. $OH^{\cdot}$ radicals are the more bactericidal ROS (Jang and Imlay 2010; Spuhler

et al. 2010). A delay in the photo-disinfection kinetic is observed when several environmentally unfavorable factors are present (Rincon and Pulgarin 2007), such as the low temperature and solar radiation available during the exposure period 15–17 h. This unfavorable condition has significantly influenced the reactivation of the *Salmonella* spp. Strain after the photo-treatment.

Inactivation mechanisms under enhanced photo-treatments (H_2O_2/hv)

Natural organic matter contains functional groups which can form complexes with Fe^{3+} or Fe^{2+}. These complexes not only increase the solubility of iron over the natural pH range but can also considerably contribute to the photo-Fenton reactions via a LMCT under solar radiation. The positive effect of NOM constituents (e.g., carboxylic acids) on photo-Fenton process, which allow us to work at near-neutral pH and, as noticed here, at alkaline pH (pH 8.6±0.3) too, has recently been reported by several authors (Georgi et al. 2007; Lipczynska-Kochany and Kochany 2008; Vermilyea and Voelker 2009). The Fe^{2+} generation from the Fe^{3+}-organo-complex in the enhanced photo-disinfection treatments carried out in this study reacts with the H_2O_2 added to the water and leads to the generation of lethal $^\bullet OH$ radical (Pulgarin et al. 1995; Pignatello et al. 2006; Malato et al. 2009). The association of the ROS production with the reaction between the photosensitized NOM and dissolved O_2 supplied by the water recirculation in the CPC and the high generation of •OH by photo-action (photo-Fenton) and nitrogen components oxido-reduction in the water cause high oxidative stress in the enteric bacteria. In a normal situation, the cells can escape oxidative stress by producing catalase enzymes to inactivate them (Cabiscol et al. 2000). It is known that the enzymes are photo- and thermosensitive and are inactivated with increased temperatures (Ghadermarzi and Moosavi-Movahedi 1996; McGuigan et al. 1998). The simultaneous temperature increase and photonic action during the ROS production have inactivated the enzyme production. In the absence of the protective enzymes, the self-defense mechanisms of the cells are inhibited, thus favoring the H_2O_2 penetration into the cell through membrane peroxidation and the intracellular production of the OH• and other ROS ($^1O_2$, $HO_2^\bullet$, $^\bullet O_2^-$, H_2O_2) leading to irreversible cell inactivation. The post-irradiation events show no enteric bacteria recovery, thus confirming their irreversible inactivation.

Conclusions

The photo-disinfection treatment was efficiently enhanced by H_2O_2 addition at alkaline pH. Solar irradiation is the key factor for both photo-disinfection processes (hv and H_2O_2/hv).

The enhanced photo-disinfection via the enhancement of internal and external photo-Fenton at alkaline pH is efficient if conducted under irradiances superior to 12 W m^{-2} and a temperature higher than 40 °C. None of the enteric bacteria strains (total coliform/*E. coli* and *Salmonella* spp.), totally inactivated under enhanced photo-disinfection treatment, have succeeded in recovering the culturability during the subsequent 24 h of dark storage. The resistivity of the *Salmonella* spp. strains noticed at near-neutral pH in our previous study was not confirmed at alkaline pH. These strains were highly sensitive at alkaline pH, and their total inactivation was recorded at the same time as those of the total coliforms/*E. coli*. The enhanced photo-disinfection treatment ensures the irreversible inactivation of the enteric bacteria, while under bare solar radiation exposure of 4 or 2 h, the inactivated *Salmonella* spp. strains have recovered their viability during the 24 h of subsequent dark storage. To efficiently disinfect the water under uniquely solar radiation, it is important to expose it for more than 4 h under intense solar irradiance and impose a temperature increase of up to 45 °C to ensure the simultaneous irreversible inactivation of total coliform/*E. coli* and *Salmonella* spp. Considering the enhancement ability of the H_2O_2/hv system and its irreversible lethal action on enteric bacteria, we recommend additional research into improvements to ensure the safety of the treated water before it implementation at point of use level.

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