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Original Research Article

Disinfection of Domestic Wastewater by Solar TiO_2 Photocatalysis Using CPC Solar Reactor: A Case Study in Bursa[a]

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ABSTRACT

This study intends to evaluate the applicability of solar TiO_2 photocatalysis, which is one of the solar-based advanced oxidation processes, for domestic wastewater disinfection. Studies were performed under natural sunlight with a CPC solar reactor and with beakers in summer and autumn months (July-October). Different flow rates (0.21-15 L min -1) and TiO_2 concentrations (1-300 mg L -1) were applied in CPC solar reactor and optimum conditions were detected to reach the highest *E.coli* removal. Optimum flow rate and TiO_2 concentration that provided the highest *E.coli* removal (2.97-log) after 5 hours were 10 L min -1 and 200 mg L -1, respectively. It was observed that the removal efficiencies decreased at TiO_2 concentrations above 200 mg L -1. Higher removal ratios were obtained at higher flow rates. This study shows that the application of solar TiO_2 photocatalysis using CPC solar reactors for domestic wastewater disinfection in Bursa city appears to be feasible as an environmentally friendly, economical and non-toxic alternative.

Keywords: Solar/TiO_2, Wastewater disinfection, Natural sunlight, CPC solar reactor, *E.coli*

INTRODUCTION

In recent years, photocatalytic processes have been applied in many different fields of environment and energy (Nakata and Fujishima 2012). Titanium dioxide (TiO_2) is the most widely studied and used semiconductor in photocatalytic processes. The usability of TiO_2 together with the sunlight has provided a safe, non-hazardous, non-by-product forming and environmentally friendly process (Gamage and Zhang 2010, Nakata and Fujishima 2012).

TiO_2 is a preferred semiconductor due to its characteristics like high oxidation potential, low cost, non-toxicity, chemical stability, super hydrophilicity and reusability (Gamage and Zhang 2010, Nakata and Fujishima 2012). TiO_2 can be used as suspended, i.e. slurry or fixed on TiO_2 supported materials (Sichel *et al.* 2007).

TiO_2 photocatalysis is based on the formation of hydroxyl radicals (•OH) by reactions occurring on the semiconductor surface (i.e.TiO_2) under sunlight at the wavelenghts below 385 nm (near UV region) (Rincón and Pulgarin 2003). Matsunaga *et al.* (1985) first reported the photocatalytic damage to the bacteria with TiO_2. There are studies in the literature on the use of TiO_2 photocatalysis under natural or simulated solar light for the destruction of various bacteria, spores, yeasts, algae, fungi, protozoa, viruses and microbial toxins (Foster *et al.* 2011, Gamage and Zhang 2010). Disinfection with TiO_2 photocatalysis process depends on different factors such as temperature, catalyst physicochemical characteristics and concentration, microorganism type and concentration, light intensity, pH, flow rate, contact time and presence of organic material and/or inorganic ions (Rincón and Pulgarin 2003, Sichel *et al.* 2007, Herrmann 2005, Malato *et al.* 2009).

Different reactor types have been developed for the application of photochemical and photocatalytic processes: parabolic-trough concentrators (PTCs), one-sun collectors (flat systems) and compound parabolic reactors (CPCs). CPCs have the advantage of both PTCs and flat systems. They receive both direct and diffuse sunlight and as a result, they are preferred as the best option for solar-based processes (Malato *et al.* 2002, Fernández *et al.* 2005).

Studies directed to the treatment and disinfection of water and wastewater by solar-based processes have been conducted in different parts of the world such as Spain, Egypt, Morocco, Mexica, Peru, Tunisia, Switzerland, Argentina, Greece and France (Gamage and Zhang 2010). Our country is fortunate in terms of solar energy because of its geographical location (Varınca and Gönüllü 2006). It has a high potential for the implementation of solar-based applications in different areas.

Bursa is located in the Marmara Region, northwest of Turkey, between 40º 11' N latitude and 29º 04' E longitude. Although Marmara Region receives less sunlight than the southern parts of Turkey, it is a region where solar-based applications can be carried out according to the Solar Energy Potential Atlas (GEPA) prepared by the General Directorate for Renewable Energy of the Ministry of Energy and Natural Resources in 2008.

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According to GEPA (2008) data, global radiation has its maximum value in June and minimum value in December in Bursa City. The daily sunshine duration reaches its maximum value of 10.78 hours in July and minimum value of 3.40 hours in December.

This work aimed to determine the capability and effectiveness of using solar TiO_2 photocatalysis for domestic wastewater disinfection in Bursa city. Experiments were performed with continuous flow (CPC solar reactor) and batch reactors (beakers) under natural sunlight. Optimum flow rate and TiO_2 concentration were determined using CPC solar reactor. Thus, the applicability of solar-based processes in Bursa city has been investigated.

MATERIALS and METHODS

Preparation of Synthetic Wastewater

Synthetic domestic wastewater was prepared according to OECD (2001) with a COD concentration of 10000 mg L -1 to be diluted to a desired COD level for the experiments.

Reactor for Batch Experiments

Beakers were used for batch experiments under natural sunlight in the field study. The beakers used had a height of 95 mm and a diameter of 80 mm. The height of water in a beaker was 65 mm.

Reactor for Continuous Flow Experiments

CPC solar reactor was used for continuous flow experiments under natural sunlight in the field study. Reactor consisted of a solar collector, a water tank, a control panel and a circulation pump (Figure 1). The solar collector had 6 tubes made of quartz glass. The illuminated length, outer and inner diameter of the tubes were 960 mm, 29 mm and 26 mm, respectively. Tubes were connected to each other serially by plastic linkages; thus, the circulation of water between the tubes and the water tank was provided. U-shaped aluminum reflectors were placed under the tubes. A stirrer was mounted to the lid of the tank. Water flow rate was adjusted by a flowmeter. Reactor was tilted according to the local latitude of Bursa (40°) and located in the east-west direction.

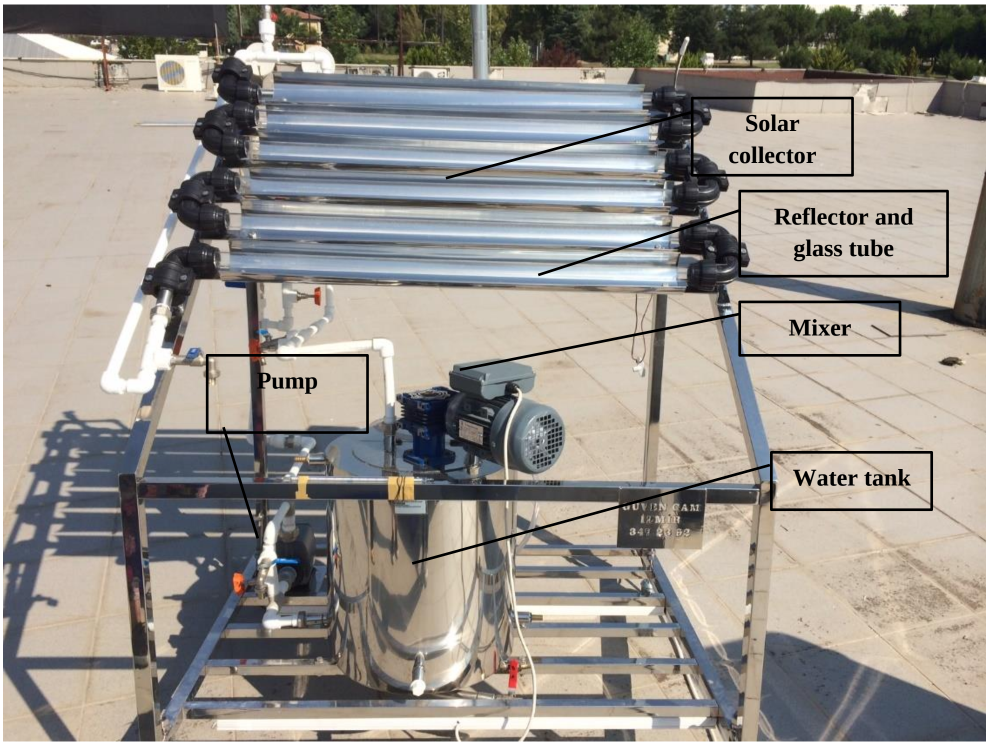

Figure 1. CPC solar reactor.

Preparation and Enumeration of *E.coli*

E.coli (ATCC 25922) was purchased from American Type Culture Collection (ATCC). According to ATCC recommendations, *E.coli* was inoculated in Tryptic Soy Broth (TSB; Merck, Germany) and grown overnight to be activated at 37°C with constant shaking in an orbital incubator. A loopful of bacteria was transferred on Tryptic Soy Agar (TSA) slants and after incubation, slants were stored at 4 °C as stock culture. *E.coli* was subcultured on fresh TSA slants monthly.

Colonies on TSA slants were inoculated in 100 mL TSB and cultured at 37°C for 20 h in an orbital incubator. *E.coli* growth was measured by optical density at 595 nm (OD_{595}). Bacteria at the stationary phase were harvested by centrifugation at 5.000 rpm for 10 min and washed twice with ringer solution (Merck). Bacteria were resuspended in ringer solution and stored at 4°C. Bacterial concentration of this stock solution was approximately 10^8 CFU mL -1.

Bacterial colonies were enumerated by pour plate method on plate count agar (PCA) (Giannakis *et al.* 2015). Cream-colored colonies on PCA were counted after incubation at 37°C for 20 h.

Experimental Procedure for Batch Reactors and CPC solar reactor

The field study with batch and continuous flow reactors were carried out on top of a building located in the Uludağ University, Görükle Campus. Aeroxide TiO_2 P25 (Evonik Industries AG, Germany) was used for solar/TiO_2 experiments.

Experiments with beakers were performed at neutral pH with synthetic domestic wastewater (COD:90 mg L -1) containing *E.coli* at the concentrations of 1 and 100 mg L -1 TiO_2. Experiments were performed between 11:00-14:00. Samples were taken at 0, 60, 120 and 180 min.

Field study with CPC solar reactor was carried out between 10:30-15:30 with synthetic domestic wastewater (COD: 90 mg L -1) at neutral pH (pH 7). The pH of wastewater was adjusted, *E.coli* was added and the first sample was taken to determine the initial microorganism concentration. The circulation pump was started and the water was circulated several times in the CPC solar reactor and the full flow of the tubes was provided. The flow rate was set to the desired value. TiO_2 was then added to the wastewater to provide the desired concentration and several more circulations of water were provided to ensure homogeneity. Microbiological, physical and chemical analyses were carried out on samples taken at 0, 60, 120, 180, 240 and 300 min.

Determination of physical and chemical parameters

OD_{595} was measured by a Hach Lange DR5000 spectrophotometer. pH, water temperatures were measured by a Hach Lange HQ40d portable multiparameter water analyzer before and after the experiments. The solar UVB (280–315 nm) and UVA (315–400 nm) irradiances were measured using Delta Ohm Multifunction Datalogger (DO9847) equipped with appropriate probes. The global radiation measurement (295–3000 nm) was performed with a pyranometer (Delta Ohm LPPYRA02).

Determination of cumulative UVA energy

The cumulative total UVA energy reaching the water during the experiment was calculated according to the following equation (Eq.1) (Malato *et al.* 2009, Polo-López *et al.* 2010):

$$Q_{UV,n} = Q_{UV,n-1} + \Delta t_n \overline{UV_{G,n}} \frac{A_{CPC}}{V_t}$$

$\Delta t_n = t_n - t_{n-1}$ (contact time between the times n and n-1, min)
$\overline{UV_{G,n}}$: the average incident radiation on the irradiated area during t_n (W m -2)
V_t: total water volume in the reactor (L)
A_{CPC}: illuminated surface area of the reactor (m^2)
$Q_{UV,n-1}$: the UV energy accumulated per litre at time n-1 (kJ L -1)
$Q_{UV,n}$: the UV energy accumulated per litre during the experiment,i.e.at time n (kJ L -1)

RESULTS AND DISCUSSION

Experiments with batch reactors

Prior to the experiments with CPC solar reactor, batch system field studies were done using beakers to assess the disinfection efficiency in these reactors and to compare with continuous flow reactors. The air was cloudy, occasionally sunny during the beaker experiments and air temperature was 36-37 °C. The light intensities reaching the beakers changed between 4.28-19.15 W m -2 for UVA, 0.358-1.431 W m -2 for UVB and 278-1191 W m -2 for global radiation. For beaker experiment at a low TiO_2 concentration (1 mg L -1), initial bacterial concentration of 10^6 CFU mL -1 drop to the concentration of 6.2 x 10^4 CFU mL -1 after 60 min (31 kJ m -2) (0.81 kJ L -1). After 120 min, bacterial concentration reached to zero (71.8 kJ m -2) (1.87 kJ L -1) and 6-log removal was obtained. The removal of bacteria decreased with the increase in TiO_2 concentration from 1 to 100 mg L -1. The initial bacterial concentration of 10^6 CFU mL -1 reduced to the concentration of 1.72 x 10^4 CFU mL -1 after 180 min (116.4 kJ m -2) (3.03 kJ L -1) with TiO_2 concentration of 100 mg L -1. The bacterial concentration reached to zero (6-log removal) in 120 min despite overcast weather conditions with 1 mg L -1 TiO_2 as compared with 100 mg L -1 TiO_2 (only 1.76-log removal in 180 min). This result showed the negative effect of turbidity produced by TiO_2 on the removal of bacteria in batch reactors.

Experiments with CPC solar reactor

Field experiments with CPC solar reactor were carried out between July and October. It was aimed to determine the optimum TiO_2 concentration and flow rate using the CPC solar reactor.

Variations in air and water temperatures and light radiation values for CPC solar reactor experiments are given in Table 1. During the experiments, air temperatures changed between 21-43 °C. Water temperatures varied between 24-31 ºC at the beginning of the processes and 36-52 ºC at the end of the processes. The increase in water temperatures at the end of the processes was caused by infrared rays from the sun. The light intensities reaching the reactor changed between 3.66-27.9 W m -2 for UVA, 0.276-2.08 W m -2 for UVB and 158-1150 W m -2 for global radiation depending on weather conditions and hour of the day.The total cumulative UVA energy reaching the reactor during the experiments changed between 4.88 and 8.55 kJ L -1.

Table 1. Variations in water and air temperatures and light radiation values for CPC solar reactor experiments.

	EXPERIMENTS*										
	(1)	**(2)**	**(3)**	**(4)**	**(5)**	**(6)**	**(7)**	**(8)**	**(9)**	**(10)**	**(11)**
Weather conditions	Partly cloudy	Clear, sunny	Clear, sunny	Clear, sunny	Clear, sunny	Clear, sunny	Clear, sunny	Sunny, time to time cloudy	Clear, sunny	Sunny, time to time cloudy	Partly cloudy
Air temperature ranges (°C)	32-37	32-40	32-43	35-43	35-39	37-40	34-38	29-36	33-37	31-36	21-29
Water temperature ranges (°C)	29-36	31-37	29-40	30-41	30-38	29-39	27-38	31-47	29-52	26-51	24-44
UVA intensity (W m -2)	4.66-24.4	3.86-21.5	5.43-23.8	6.32-21.9	3.66-23.3	9.06-22.5	4.52-27.9	4.88-22.0	5.45-23.4	4.55-22.6	4.91-23.0
UVB intensity (W m -2)	0.413-1.606	0.276-1.488	0.473-1.67	0.772-1.238	0.285-1.479	0.624-1.604	0.391-2.08	0.383-1.384	0.441-1.507	0.374-1.622	0.391-1.616
Global radiation intensity (W m -2)	209-1041	264-947	494-953	559-890	158-965	740-964	231-1150	230-1034	596-985	397-1048	187-1128
Cumulative UVA dose (kJ L -1)	4.88	5.96	8.29	7.80	8.3	7.50	8.55	7.40	8.24	7.6	5.6

***(1)** Flow rate: 4.65 L min -1 TiO_2: 1 mg L -1
(2) Flow rate: 4.65 L min -1 TiO_2: 50 mg L -1
(3) Flow rate: 0.21 L min -1 TiO_2: 1 mg L -1
(4) Flow rate: 0.21 L min -1 TiO_2: 20 mg L -1
(5) Flow rate: 0.72 L min -1 TiO_2: 5 mg L -1
(6) Flow rate: 5.65 L min -1 TiO_2: 5 mg L -1
(7) Flow rate: 5.65 L min -1 TiO_2: 100 mg L -1
(8) Flow rate: 15 L min -1 TiO_2: 100 mg L -1
(9) Flow rate: 10 L min -1 TiO_2: 100 mg L -1
(10) Flow rate: 10 L min -1 TiO_2: 200 mg L -1
(11) Flow rate: 10 L min -1 TiO_2: 300 mg L -1

Figure 2 shows logarithmic *E.coli* removals. TiO_2 concentrations between 1 and 300 mg L -1 and flow rates between 0.21 and 15 L min -1 were studied to determine the optimum catalyst concentration and flow rate. As can be seen from Fig.2, low TiO_2 concentrations and low flow rates did not provide an effective *E.coli* removal [experiments (1), (2), (3), (4) and (5)]. The log removals for experiments (1), (3), (4) and (5) were below 0.5-log at the end of 300^{th} min. Only for experiment (2), the removal reached to 0.69-log.

Low (5 mg L -1) and high (100 mg L -1) TiO_2 concentrations were used at a flow rate of 5.65 L min -1 for experiments (6) and (7). The log removal reached to 0.78-log after 300 min with the application of 5 mg L -1 TiO_2. The effect of increment in TiO_2 concentration on log removals was clearly seen, even at 120^{th} min. When TiO_2 concentration increased from 5 mg L -1 to 100 mg L -1, *E.coli* log removals for experiment (7) increased to 0.87-log at 120^{th} min and 1.36-log at 300^{th} min, respectively.

Higher flow rates (10 L min -1, 15 L min -1) and higher TiO_2 concentrations (100 mg L -1, 200 mg L -1, 300 mg L -1) were applied for experiments (8), (9), (10) and (11). The log removals for these experiments varied between 2.06-log and 2.97-log at the end of 300 min. TiO_2 concentrations above 100 mg L -1 provided to reach higher log removals in shorter times. While the log removals of *E.coli* were 0.58-log and 0.10-log, respectively, for experiments (8) and (9) at 120th min, the removals increased to 2.19-log and 2-log for experiments (10) and (11).

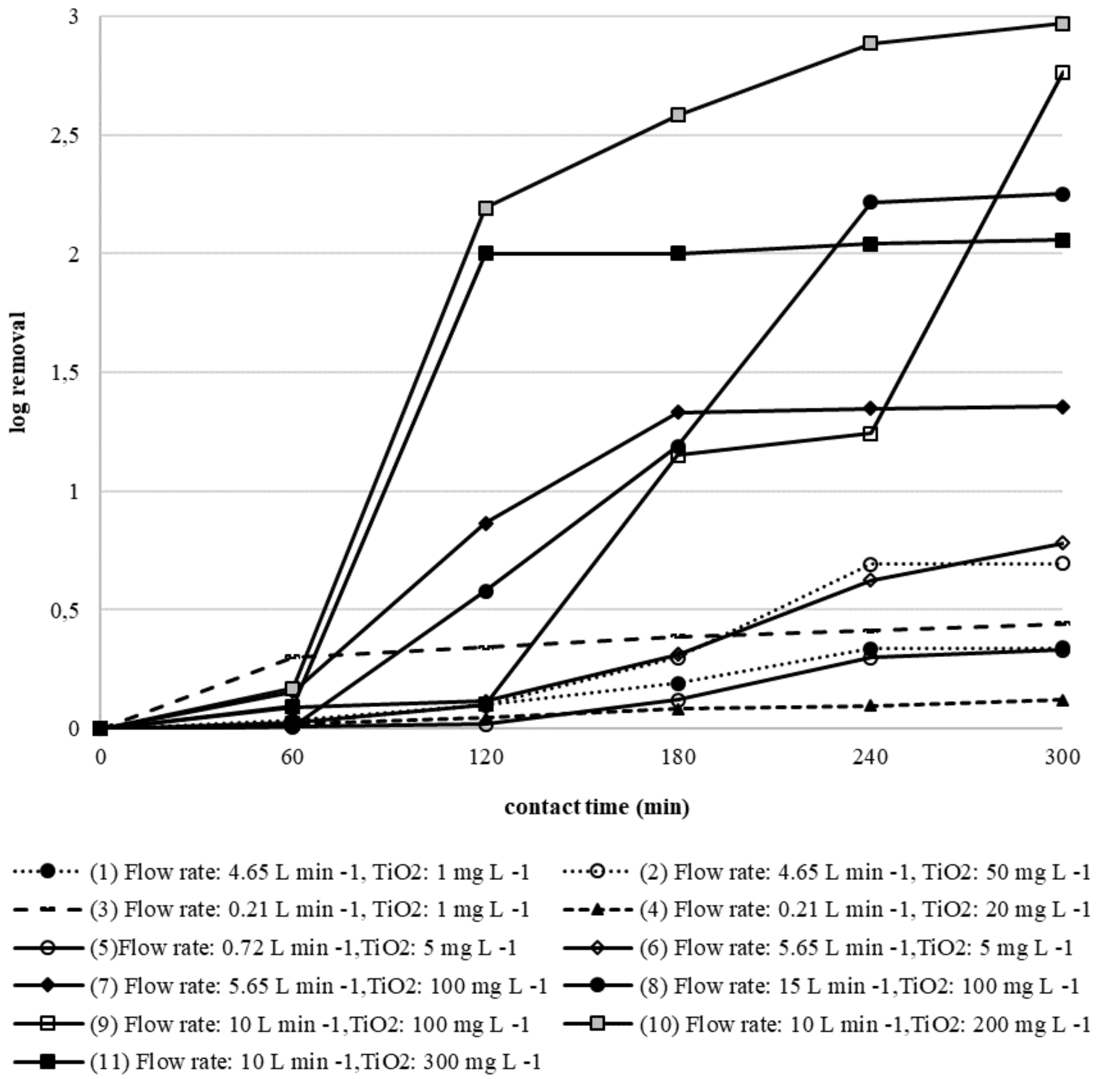

Figure 2. *E.coli* removals obtained by solar TiO_2 photocatalysis process with CPC solar reactor.

The determination of optimum TiO_2 concentration and flow rate was significant to obtain best disinfection efficiencies. The optimum TiO_2 concentration and flow rate were determined as 200 mg L -1 and 10 L min -1 (experiment 10). The log removals reached to 2.19-log and 2.97-log at 120th and 300th min, respectively.

In photocatalytic disinfection processes, when the catalyst concentration is increased to a certain value (optimal catalyst concentration), the rate of inactivation increases and at higher catalyst concentrations, the rate of inactivation remains constant or decreases due to the effect of light screening. Examination of the Fig.2 indicated that scattering effect might have appeared with the application of 300 mg L -1 TiO_2 and resulted in lower log removals. Many scientific studies aimed at determining the optimal catalyst concentration have reported that different results are obtained depending on the light intensity and the photoreactor geometry (Malato *et al.* 2009). In the literature, concentrations of 100 mg L -1 and above are generally applied in wastewater disinfection studies with TiO_2 photocatalysis under natural sunlight. Agulló-Barceló *et al.* (2013) applied the TiO_2 photocatalysis process to real wastewaters and worked at a concentration of 100 mg L -1 TiO_2. Lydakis-Simantiris *et al.* (2010) examined the disinfection of wastewater treatment plant effluents and reported that they worked at concentrations of 0.5-1 g L -1 TiO_2.

It is important to optimize flow rate for photocatalytic disinfection efficiency. Slow water flow may result in the precipitation of bacteria and TiO_2 particles whereas fast water flow may prevent the light to reach the bacteria and the surface of TiO_2 particles. According to Fig.2, the log removals varied between 0.12-log and 1.36-log with the application of flow rates of ≤5.65 L min -1. The removals did not change significantly at the flow rate of 15 L min -1 (experiment 8) when compared to the removal at the flow rate of 10 L min -1 (experiment 9) for the same TiO_2 concentration (100 mg L -1). Consequently, 10 L min -1 was selected as optimum flow rate. Fernández-Ibánez *et al.* (2009) conducted photocatalytic disinfection experiments with different concentrations of TiO_2 in CPC solar reactors and investigated the removal of *F.solani* spores in pure water. As a result of fungal spores and catalyst interactions, the weight of the aggregates increases and low flow rates are not sufficient to suspend them. Loss of catalyst occurs and the amount of hydroxyl radicals formed also decrease resulting in lower removal efficiencies.

The initial and output values for some important parameters for CPC solar reactor experiments were also assessed. pH values changed between 6.99-7.40 at the beginning of the process and 6.86-7.65 at the end of the process. pH values tend to increase at the end of the process in general.

Comparison of batch and continuous flow systems

Significant differences may occur in terms of removal efficiencies in batch and continuous flow reactors. Some of the selected studies for comparison of batch and continuous flow systems are presented in the table below (Table 2).

Table 2. Comparison of batch and continuous flow systems.

	UVA intensity (W m -2)	UVB intensity (W m -2)	Global radiation intensity (W m -2)	UVA dose (kJ L -1)	Contact time (hour)	TiO_2 concentration (mg L -1)	Log removal
Batch system							
Beaker	4.28-19.15	0.358-1.431	278-1191	1.87	2	1	6-log
Beaker	4.28-19.15	0.358-1.431	278-1191	3.03	3	100	1.76-log
Continuous flow system							
CPC solar reactor (exp. 9)	11.32-23.4	0.623-1.507	855-985	6.02	3	100	1.15-log
	5.45-23.4	0.441-1.507	596-985	8.24	5	100	2.77-log
CPC solar reactor (exp. 10)	4.55-22.6	0.374-1.622	397-1048	7.6	5	200	2.97-log

According to Table 2, it was observed that complete bacterial inactivation (6-log) occurred in beaker with the low TiO_2 concentration (1 mg L -1) in 2 hours. Only 1.76-log removal was obtained with 3.03 kJ L -1 UVA dose for the beaker containing high TiO_2 concentration (100 mg L -1) in 3 hours contact time. For batch systems, due to the low TiO_2 concentration and low turbidity, the microorganisms did not hide behind TiO_2 aggregates and were not protected from light and exposed to more light. Additionally, because of the continuous illumination of the beaker, the defense mechanisms of bacteria that exposed to high UVA, UVB intensities were prevented and thus, disinfection efficiency reached its maximum. At high TiO_2 concentration, because of high turbidity and static conditions, the light reaching the bacteria was reduced seriously.

CPC solar reactor (experiment 9) study that had the concentration of TiO_2 (100 mg L -1) provided 1.15-log removal in 3 hours contact time with 6.02 kJ L -1 UVA dose. For CPC solar reactor, UVA dose was doubled but lower removal was achieved when compared the beaker study containing the same TiO_2 concentration. It was thought that the interruption of light within the CPC solar reactor affected the bacterial removal. Since the wastewater was not in circulation in the beaker, the light reached to the bacteria without interruption resulting in higher bacterial inactivation. However, in the CPC solar reactor, the radiation was not continuous. The wastewater was circulating between the tank, pipes and hoses, which did not receive light, and the solar collector. The wastewater only received light when passing through glass tubes. The total illuminated volume of water (V_{ill}) passing through the tubes in the CPC solar reactor was calculated to be 3.1 L. The total water volume in the tank of CPC solar reactor was 20 L. Accordingly, when the water passed through the tubes of CPC solar reactor, only 3.1 L of 20 L of total water in the tank was exposed to the solar light. Remaining of 16.9 L of water filled the tank, hose and pump and the disinfection efficiency was reduced. Some researchers stated that the bacteria developed self-defense mechanisms against the oxidative stress and had time to recover by the interruption of the solar light in the tank (Bello Lamo *et al.* 2015, Rincón and Pulgarin 2003). The disinfection efficiency depends more on the cumulative UV dose reaching to the reactor without interruption (Ubomba-Jaswa *et al.* 2009).

The interruption of the solar light in the parts of the CPC solar reactor also increased the contact time required to reach full *E.coli* inactivation. Because the light was not continuous in the CPC solar reactor, only 2.77-log and 2.97-log removals after 5 hours were obtained in experiment (9) and (10), respectively.

CONCLUSIONS

In this study, the disinfection of domestic wastewater by solar TiO_2 photocatalysis using CPC solar reactor in Bursa was investigated. The optimum flow rate and TiO_2 concentration were determined as 10 L min -1 and 200 mg L -1, respectively, which provided 2.97-log *E.coli* removal within 5 hours. TiO_2 concentrations below 100 mg L -1 were not very efficient in the inactivation of *E.coli*. However, it is important to determine the upper limit for the TiO_2 concentration, since TiO_2 particles can block the entry of light to the reactor. High TiO_2 concentrations like 300 mg L -1 decreased the disinfection efficiency. Higher removal efficiencies were achieved at optimum flow rates. Lower log removals were obtained with the application of flow rates of ≤5.65 L min -1 due to the precipitation of bacteria and TiO_2. When the flow rate was increased to 10 L min -1, disinfection efficiency increased. However, it should be considered that the light may not reach to bacteria and TiO_2 at higher flow rates because of fast water flow.

In continuous flow reactors, the light is interrupted in some parts of the reactor. Within this period, bacteria gains time to improve their self-defense mechanisms causing lower removal efficiencies.

According to results of this study, an extensive evaluation should be made for disinfection with solar-based processes. Some factors like interrupted and non-interrupted UV, cumulative UV dose, UV intensity, reactor configuration, weather conditions, flow rate, catalyst concentration should be taken into account. Especially in circulating systems, appropriate operating parameters should be chosen not to allow the microorganisms to repair.

On the basis of the results obtained from this study, domestic wastewater disinfection with solar-based processes in CPC solar reactors can be considered as a good alternative for Bursa city.

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