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PULSED POWER SUPPLY AND COAXIAL REACTOR APPLIED TO *E. coli* ELIMINATION IN WATER BY PDBD

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Key words: *Escherichia coli*, pulsed dielectric barrier discharge, pulsed power supply, reactor discharge, plasma

ABSTRACT

The design and instrumentation intended for ATTC8739 *Escherichia coli* (*E. coli*) bacteria elimination in water, based on non thermal plasma generation at room pressure have been carried out by means of dielectric pulsed discharges. The latter have been produced by a power supply capable of providing voltages up to the order of 45 kV, 1-500 μs pulse widths and variable frequencies between 100 Hz to 2000 Hz. This supply feeds a coaxial discharge reactor of the simple dielectric barrier type. The adequate operation of the system has been tested with the elimination of *E. coli* at 10^4 and 10^6 bacteria/mL concentrations, leading to reductions up to 85.3 % and 95.1 %, respectively, during the first 30 min of treatment.

Palabras clave: *Escherichia coli*, descargas de barrera dieléctrica pulsada, fuente de alimentación pulsada, reactor de descarga, plasma

RESUMEN

Se presenta el diseño y la instrumentación para la eliminación de la bacteria *Escherichia coli* (*E. coli*) ATTC 8739 presente en el agua, basada en la generación de plasma no térmico a presión atmosférica, obtenido mediante descargas pulsadas de barrera dieléctrica. Estas descargas son generadas por una fuente pulsada que puede proporcionar voltajes hasta del orden de los 45 kV, anchos de pulsos desde 1 hasta 500 μs operando a frecuencias variables desde 100 hasta 2000 Hz. El rector de descargas es del tipo coaxial de barrera dieléctrica simple. El funcionamiento adecuado del sistema se comprobó la eliminación de la bacteria *E. coli* para concentraciones de 10^4 y 10^6 bacterias/mL, obteniéndose una reducción en la concentración de hasta un 85.3% y 95.1 % respectivamente para los primeros 30 min de tratamiento.

INTRODUCTION

The presence of a high bacterial concentration often makes impossible the use of water resources in order to satisfy the basic human needs unless a previous treatment process is applied (Escott *et al*. 2000). The stability of these microorganisms in chemically degrading habitats and under the action of conventional treatments, has promoted the development of more effective elimination methods. These are mostly based on the use of active species (OH, H_2O_2, O_3, etc) such as advanced oxidation process (AOP), or the use of UV radiation (Sunka *et al*. 1999), among others. One common characteristic of the non conventional methods is the avoidance of chlorine (Cl_2) based chemicals such as chlorofluorocarbons, perfluorinated compounds, chloramines (NH_2Cl), trihalomethanes ($CHCl_3$) and hypoclorous acids (ClOH) (Smith *et al*. 2010). AOP requires a technology capable of generating highly oxidizing species which can be implemented by the application of non thermal plasmas at room pressure to the control of volatile organic compounds (Julien and Vervisch 2006), NO_X elimination (Kin and Kin 2003), food conservation and sterilization (Fang *et al*. 2006), control of air pollution (Chang 2008), treatment of biological and chemical hazardous residuals (Tedder and Pohland 2002) as well as the elimination of water born microorganisms (Hernández-Arias *et al*. 2012). In the latter case, plasmas can be generated either by AC or pulsed dielectric barrier discharges (PDBD).

The ions and free electrons that move in the PDBD electrode gap are accelerated by the electric field. In the event of inelastic collisions, the electrons ionize the reactor gas molecules producing more free electrons. In the end, a streamer takes place reaching electron densities ~$10^{20}/m^3$ at energies between 1 and 10 eV. As these electrons are responsible for the chemical reactions that generate oxidizing species (Yang *et al*. 2012), PDBD in water constitute a feasible technological alternative in the elimination of microorganisms. Several parameters characterize the pulsed power supplies (PPS) capable of producing this kind of plasma discharges: rising time (t_r), pulse width (t_w) repetition rate (f), as well as voltage magnitude and polarity (U) (Schoenbach *et al*. 2002). Typical voltage/frequency/pulse width fixed values are: 500 kV/120 Hz/1.5 µs (Slac *et al*. 1998), 10-35 kV/1000 Hz/50-150 µs (Yan *et al*. 2006), 120 kV/50 Hz/600ns (Ghazala *et al*. 2002), while some variation ranges are 1-30 kV/100-2000 Hz/1-50 µs (Rodríguez-Méndez *et al*. 2008). Naturally, some other kinds of PPS are possible which provide from hundreds of kV (Hegeler *et al*. 2011) to several MV (Mesyats *et al*. 2001) with pulse widths between picoseconds (Mesyats *et al*. 1998) and microseconds (Rodríguez-Méndez *et al*. 2008), having been accomplished in point-plane (Sunao *et al*. 2002) and coaxial (Hernández-Arias *et al*. 2012) configurations. In the latter one, the discharge occupies the reactor quite uniformly, providing a better distribution of the generated species (Gouri *et al*. 2009). The production of oxidizing agents can be controlled by the adjustment of the t_w, f, t_r and U electric parameters, with diverse effects on the pollutants (Schoenbach *et al*. 2002).

This paper describes the design and implementation of a pulsed high voltage supply characterized by a 1-45 kV output, pulse width between 1 and 500 µs as well as a 100-2000 Hz frequency, coupled to a cylindrical reactor designed and constructed with a 12.5 mm electrode gap. A central anode is covered with an aluminum oxide layer. The external electrode is made of stainless steel. This reactor enabled to verify the correct operation of the power supply during the elimination process of ATCC 8739 *E. coli* at 10^4 and 10^6 bacteria/mL concentrations, typically found in septic system effluents and residual untreated waters (Crites and Tchobanoglous 2000).

SET UP

Pulsed power supply (PPS)

A pulsed energy system has been designed and constructed as a combination of a pulsed power supply and a cylindrical reactor, as shown in **Fig. 1**, in order to carry out pulsed dielectric barrier discharges applied to the elimination of bacteria in water. The pulsed power supply was implemented according to **Fig. 2**, on the basis of three pulsed power network (PPN) elements, using a 220V (AC) input (1) and a variable transformer (2) operated by means of an interrupter (3) connected to full phase rectifying

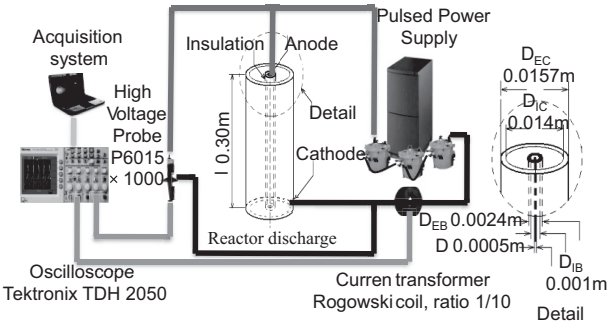

Fig. 1. Pulsed power energy system for elimination of *E.coli* in water

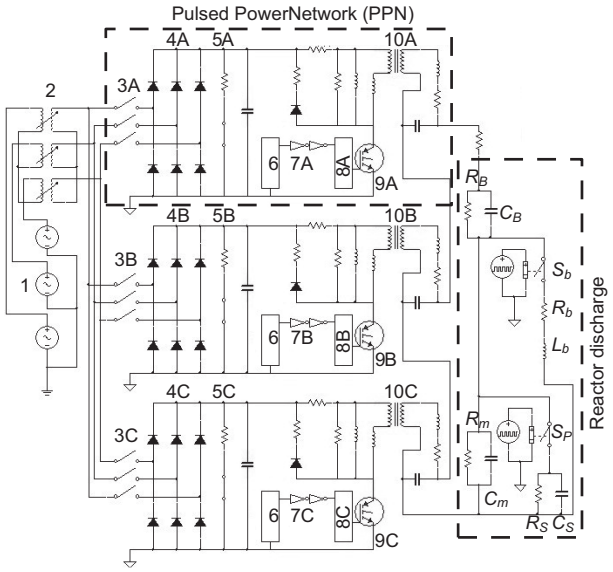

Fig. 2. Pulsed power supply

circuit (4) that provides ~300 V (DC). A discharge circuit (5) is integrated to main one. The three PPN operation is controlled by a SG3524N oscillator circuit (6) whereas the output of this circuit is coupled by means of TTL circuits (7) for the operation of three M57962L drivers (8). Finally, these circuits are coupled by switching the HGTG27N120BN IGBT transistor (9), which work with an inductive load (10); the transformer relation is 1:50. The PPN can operate either independently or in a coordinate manner. In the first manner the PPN switches to ~300 V, achieving an output of ~15 kV; in second manner the transformer is connected in serial and the transformation relation is 1:150, such as the output voltage is in the order of 45 kV.

Reactor discharge

The reactor designed and constructed is constituted of a stainless steel cylinder (cathode) and a wire of nickel and copper alloy (anode) covered with aluminum oxide (Al_2O_3) as insulator. The dimensions are showed in **Fig. 1**. The PPS network and the electrical model of the reactor are presented in **Fig. 2**. It consists of four stages: 1) dielectric barrier, 2) physical characteristics, 3) pre-breakdown, 4) breakdown onset.

The dielectric barrier step is formed by R_B and C_B, where R_B corresponds to the dielectric resistance expressed by (Oh *et al*. 1999):

$$R_B=(\sigma_A \bullet e)^{-1} \tag{1}$$

where σ_A is the electric conductivity of the barrier (~1×10^{-11} S/m), and e refers to its thickness (~0.0014 m) whereby R_B ~140 MΩ. And for the dielectric layer entails a capacitance given by (Oh *et al*. 2003):

$$C_B=\frac{\varepsilon_r \bullet \varepsilon_0 \bullet A_r}{\delta} \tag{2}$$

where ε_r is the absolute permittivity of the material (~8.8541×10^{-11} F/m), A_r the area (~1.885 m^2) that represents the ratio between the diameters of the barrier that covers the anode (~0.876), therefore C_B ~190 pF.

The elements of the physical characteristics section depend on the reactor geometry, material and content, given by R_m and C_m. R_m is a function of water conductivity (Ceccato *et al*. 2010):

$$R_m=\frac{\ln\left(\frac{D_{IC}}{D}\right)}{2\bullet\pi\bullet\sigma_w\bullet l} \tag{3}$$

where l is the reactor length (0.3 m), D_{IC} is the internal cathode diameter (0.0254 m), D is the diameter of the anode (0.0005 m) and σ_w is the prevailing conductivity (0.0005 S/m in the case of sweet water). Then, it follows from equation (3) that R_m ~4.17 kΩ. On its part, C_m represents the capacitance of the reactor, given by (Ceccato *et al*. 2010):

$$C_m=\frac{2\bullet\pi\bullet\varepsilon_w\bullet l}{\ln\left(\frac{D_{IC}}{D}\right)} \tag{4}$$

where ε_w is the permittivity of the medium where the discharge takes place, which, for water, is ~7.08334×10^{-10} F/m. Then, given equation (4), C_m ~340 pF.

The pre-breakdown section is characterized just for a time interval before the break-up (t_p), simulated by the S_P switch shown in **Fig. 2** and determined by Jones and Kunhardt (1996):

$$t_p \approx \frac{\upsilon_d}{d_g} \tag{5}$$

where υ_d is the drift velocity of charge on the environment (~30×10^3 m/s) (William 1999), so t_p is ~425 ns. It can be seen that this time depends on the distance between the electrodes (d_g ~0.01245 m), and the mobility of the charges present in the conduction channel. R_S depends on: electron density, charge mobility, the cross section area of the conduction

channel between the electrodes (streamer) and the applied voltage. Hence, the resistance in the pre-breakdown stage has been estimated by Jones and Kunhardt (1996) as:

$$R_S = \frac{d_g}{n \bullet q \bullet \mu \bullet A_S} \tag{6}$$

where q is the elementary charge (~1.60256×10^{-19} C), n is the electron density, namely ~1×10^{26} particles/m^3 (Jones and Kunhardt 1996), and μ is the mobility (~1×10^{-5} m$^2\times$V$^{-1}\times$s^{-1}). Thus, if d_g ~0.01245 m and the streamer area is A_S ~3.1416×10^{-8} m^2 then, given equation (6), R_S ~2473 Ω. Now, C_S is a capacitive value which depends on the distance of the *gap* and break voltage expressed as (Jones and Kunhardt 1996):

$$C_S = \frac{\rho_d}{U_m}(d_g) \tag{7}$$

where U_m is the maximum voltage applied, corresponding to ~45 kV, ρ_d is the streamer charge ~1×10^{-6} C/m, therefore C_S ~277 fF.

Finally, step four, the breakdown onset (Lisitsyn *et al*. 1998) begins when the conduction channel has been formed at the time delimited by the S_b switch shown in **Fig. 2**. The elements in this section depend on the cross-section radius of the formed streamer and the vacuum magnetic permeability. Each streamer formed in a cylindrical channel of conductivity σ_c, length l and radii r_s assume a R_b resistance determined by (Lisitsyn *et al*. 1998):

$$R_b = \frac{d_g}{\sigma_c \bullet \pi \bullet r_s^2} \tag{8}$$

where r_s is ~1×10^{-4} m (Aka and Beroual 2006) and σ_c =$1.5\times10^3\ T^{3/2}$ Ω^{-1}cm^{-1} (William 1999), here T is the temperature (~2 eV) (William 1999), and, therefore, σ_c ~4242.7 S/m, whereas, from Eq. (8), R_b ~94 Ω. L_b designates the inductive value of the streamer and is given by (Fofana and Beroumal 1996):

$$L_b = \frac{\mu_0 \cdot d_g}{2 \cdot \pi}\left[\frac{1}{4} + \ln\left(\frac{d_f}{r_s}\right)\right] \tag{9}$$

where μ_0 is the vacuum permeability ~$4\bullet\pi\bullet10^{-7}$ N/A^2 and d_f is the *gap* between streamer and the null electric field point. Then, $d_f \sim l$, and, L_b ~20.56 nH.

Experimental process with *E. coli* ATTC 8739

The *E. coli* populations required by the study were developed as samples of ATCC 8739 strain inoculated in 5 mL of Luria-Bertani (LB) broth and incubated for 24 h at 37 ºC, in order to promote bacterial growth. After the incubation, a dilution was performed at a 1:100 ratio. Placing 100 μL of it in a Neubauer chamber subjected to a count up process, a concentration of ~1.6×10^7 bacteria/mL is revealed. Once the initial concentration is known, successive dilutions at a 1:10 ratio are carried out until the work final concentration is attained. 100 μL samples of the suspension containing concentrations of 10^4 and 10^6 bacteria/mL are placed in Petri dishes containing agar solid culture medium are then planted in triplicate.

In the experimental phase with the electric discharges, 45 mL of a suspension were used in concentrations of 10^4 and 10^6 bacteria/mL, carrying out two independent PDBD processes for 5 minute periods with the following electric parameters: (1) 18 kV/1000 Hz/30 μs, (2) 25 kV/1000 Hz/40 μs and (3) 28 kV/500 Hz/30 μs. At the end of 5, 10, 15, 20,

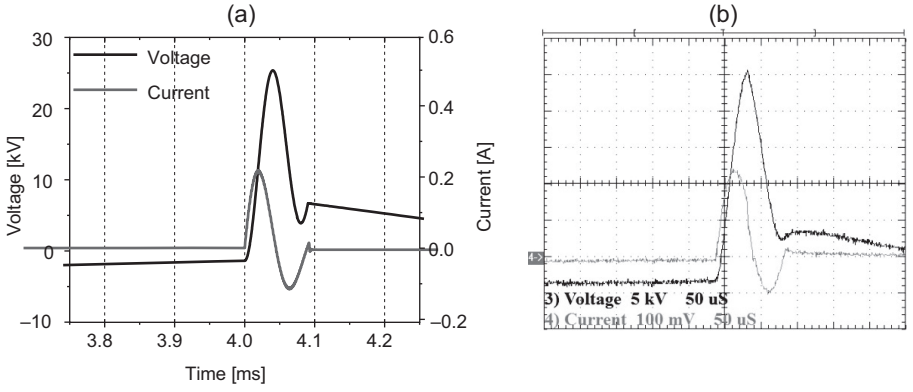

Fig. 3. Current and voltage waveforms according to (a) the simulation and (b) the experiment

25 and 30 minutes of treatment, the suspension was removed from the reactor and three samples of 100 µL are planted in a nutrient medium. These crops were placed in the incubator for 24 hours at 37 ºC and finally, after the incubation time, are forming colonies units (CFU) are determined as number of CFU/mL. The plotted data (**Fig. 4-7**) are obtained by multiplying the value of the dilution by the respective CFU and then multiplying the value obtained by 10, so as to account for the 100 µL.

RESULTS AND DISCUSSION

A simulation of the experimental phase of the system has been carried out given the electrical parameters 28 kV/500 Hz/30 µs leading to plots shown in **Fig. 3a**, to be compared to their experimental counterparts in **Fig. 3b**. The plasma discharges were conducted in the coaxial reactor containing water with bacteria in 10^4 and 10^6 bacteria/mL concentrations. The current was measured by means of a 1/10 ratio Rogowski coil (see **Fig. 1**) reaching ~240 mA whereas the voltage was established with a Tektronix P6015 oscilloscope (see **Fig. 1**) as ~28 kV. It is found that the experimental parameter values and the current and voltage waveforms approach quite closely their respective theoretical predictions, which suggest that the physical assumptions made by the model are widely applicable to the experimental phase.

The system was tested with the elimination of the *E. coli* bacteria. The kinetics of bacterial inactivation was estimated from the survivability percentage of *E. coli* bacteria versus the total plasma treatment time. **Figure 4** exhibits the inactivation process results at 10^4, 10^6 CFU/mL without any O_2 flow. In the first case, **Fig. 4a** shows a decline from 1.6×10^6 CFU/mL to a $\sim1\times10^5$ CFU/mL concentrations amounting to a ~ 95.16% elimination. Likewise, the plot in **Fig. 4b** exhibits a 1.6×10^4 CFU/mL concentration on the decrease until a $\sim1.5\times10^3$ CFU/mL value is attained. This represents a 85.34 % reduction of the bacterial population. Both results were achieved within 30 minutes of treatment.

By changing the electrical parameter values to 18 kV/1000 Hz/30 µs while preserving the concentrations, the plots of **Fig. 5** are obtained. In the case of 10^6 CFU/mL, the elimination was ~25.3% and ~29.3% in the case of 10^4 CFU/mL. It follows that the bias voltage seems to bear a decisive influence on the process. This is even more evident on **Fig. 6** with 10^6 bacteria/mL and the parameter values 18 kV/1000 Hz/40 µs and 25 kV/1000 Hz/40 µs resulting in ~25.3 % and ~95 %, elimination rates, respectively. In other words, a higher applied voltage increases the bacterial elimination rate.

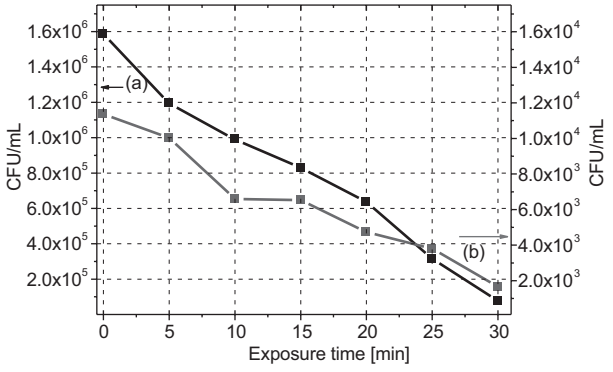

Fig. 4. *E. coli* elimination plots with (a) 104 and (b) 106 CFU/mL concentrations without any O2 flow. The discharge parameters are: 28 kV/500 Hz/30 µs

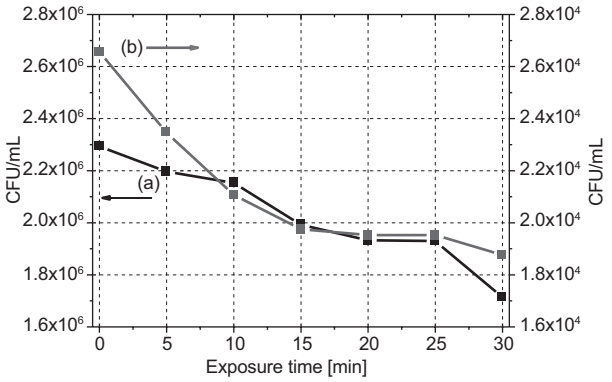

Fig. 5. *E. coli* elimination plots with (a) 106 and (b) 104 CFU/mL concentrations without any O2 flow. The discharge parameters are: 18 kV/1000 Hz/30 µs

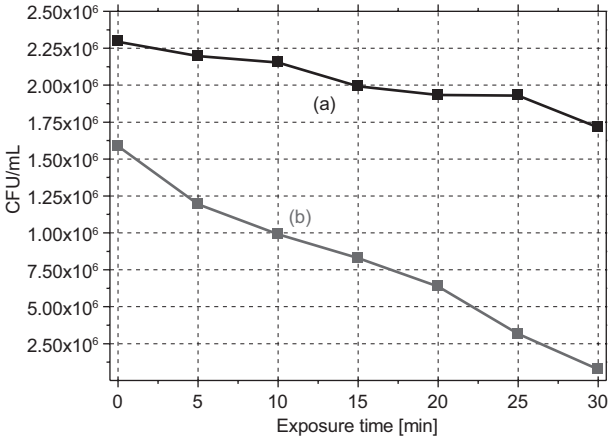

Fig. 6. *E. coli* elimination plots with a 106 CFU/mL concentration without any O2 flow. The discharge parameters are: (a) 18 kV/1000 Hz/30 µs and (b) 25 kV/1000 Hz/40 µs

The experiment was additionally refined by injecting an oxygen flow to the reactor during its operation in order to enhance the oxidation process. The results are presented in **Fig. 7**. Initial 10^4 bacteria/mL concentrations were used along with the 28 kV/500 Hz/30 μs characteristics. **Figure 7a** shows the response without any oxygen injection as a reference. Then, **Fig. 7b** reveals the reaction to a 0.5 litre/minute. Thus, the first process achieved only a ~25.7% rate whereas with O_2 flow the rate grew to ~96.8%. Clearly, the addition of oxygen enhances the generation of reactive species (ROS) such as O_3, OH and H_2O_2 capable of damaging by oxidation the cell membrane and DNA of the target microorganism.

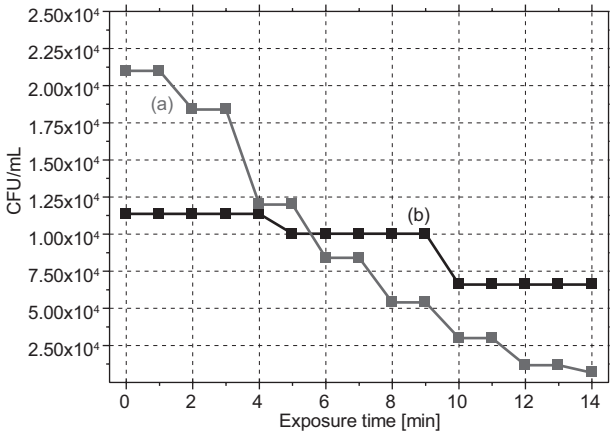

Fig. 7. *E. coli* elimination plots with the discharge parameters 28 kV/500 Hz/30 μs at the 104 CFU/mL concentration when (a) oxygen is added at a 0.5litres/minute flow rate and when (b) no oxygen is added

CONCLUSIONS

A pulsed energy system has been designed and constructed as a combination of a pulsed power supply and a cylindrical reactor to carry out dielectric barrier pulsed discharges in bacteria contaminated water. The pulsed power supply provides an output in the 1-45 kV range with 1 to 500 μs pulse widths at 100-2000 Hz frequencies. The mathematical model of the dielectric barrier pulsed discharge was developed taking into consideration voltage input, physical characteristics of the reactor, nature of the dielectric and the evolution of the start up and breakdown phases. The wave forms obtained are in full agreement with those obtained experimentally. Likewise, the model provides an effective tool to evaluate the discharge behavior.

The pulsed energy system has been experimentally tested by conducting discharges characterized by the following parameters: (1) 18 kV/1000 Hz/30 μs, (2) 25 kV/1000 Hz/40 μs and (3) 28 kV/500 Hz/30 μs applied to the coaxial reactor designed and constructed whose content was 45 mL of water contaminated with ATTC 8739 *E. coli* at concentrations of 10^4 and 10^6 bacteria/mL. The experiments were carried out with and without a 0.5 liters/minute oxygen flow. The greater elimination efficiency was achieved using the higher bias voltages and in the presence of oxygen going from a ~42 % to a ~97 % elimination rate within the same exposure period to PDBD. This occurs because the presence of oxygen favors the generation of highly oxidizing species, also the existence of an intense electric field during the process causes harmful effects to the cellular structure of the bacteria. Finally, with the set-up presented herein, it is possible to carry out experimental studies on the bacteria removal using pulsed dielectric barrier discharge, to perform advanced oxidation processes, which can be applied to treatment of bacteria contaminated waters.

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