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Ozonation of Wastewater by Dielectric Barrier Discharge and its Impact on the Elimination of Fecal Bacteria and Inorganic Micropollutants

Nadia Ramdani[1,*], *Ahmed. Hamou*[1], *Said. Nemmich*[2], *Amar Tilmatine*[2], *Mokhtaria. Yasmina Boufadi*[3] and *M. Lloréns pascual del Riquelme*[4]

[1] Department of physics, Laboratory of Environmental Sciences Study and Materials(LESEM), Faculty of Sciences, University of Oran 1, Ahmed Benbella ,Oran 31000, Algeria.
[2] Department of Electrotechnical, Laboratoire Applications of Plasma, Electrostatics, Electromagnetic– APELEC, University of Djillali. Liabes. Sidi-Bel-Abbès 22000. Algeria.
[3] Department of biology, Microorganisms benifical, fonctionnel food and health (LMBAFS), Faculty of Natural Sciences and life, University of Abdelhamid. Ibn. Badis. Mostaganem 27000, Algeria.
[4] Department of Chemical Engineering, Faculty of Chemistry, University of Murcia, 30100, Murcia, Spain.

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Abstract: The present paper investigates the wastewater treatment ozone process produced by dielectric barrier discharge (DBD) under high potential. Three (DBD) ozone generators of cylindrical form have been used for wastewater treatment plant (WWTP) of the city of Sidi-Bel-Abbes located at the West of Algeria. The experimental results show the efficiency of the treatment process by ozone based on the micro pollutant analysis (heavy metals) and microbial analysis (bacteria, viruses and parasites before and after ozonation treatment. The results show that 80% of micro pollutants are eliminated and 100% destruction of all bacteria which reveals the high efficiency of the process.

Keywords: Ozone, wastewater, treatment, DBD generator, elimination, micro pollutants, bacteria, heavy metals.

1 Introduction

It is well known that the wastewater contains a high number of substances issued from many sources. One may name a few such as residuals of household chemical products, corporal sanitary products, or medicinal wastes that are not sufficiently degraded or retained in conventional wastewater treatment stations equipped with a mechanical and biological phases. Due to the existence of modern analysis methods, a number of these types are detected in water under the form of micro-pollutants. Certain of these pollutants can have a negative impact on the biocenos is in the receiving environment or on the production and quality of potable water [1].

Currently many methods and techniques can be used to complement or substitute biological processes and give technically efficient wastewater treatment.

One of these techniques, which is the focus of this study, is the oxidation by electrical discharge [2].This technique is competitive and can be easily implemented for wastewater treatment without the addition of chemical agents. Many studies have shown the possible degradation of organic components by electrical discharge directly in water [3-4].

The produced electrical discharge in high potential field can generate reactive radicalar species in the presence of air or oxygen. One of the most important applications of the principle of this technique is the production of ozone for wastewater treatment [5-6].The dose of ozone applied for the treatment of wastewater is in the range of (1 to 10 mg O_3/l). This can only give a partial oxidation of the substances which can be transformed into biologically active sub-products. They can be in certain cases more toxic than the original substances such as the formation of bromate, formaldehyde or nitrosamines [7-8].These sub-products are usually easily biodegradable [7-8].

O_3 is an unstable gas produced by electrical discharge either in air or purer oxygen. This results from the dissociation of oxygen molecules into atomic oxygen and subsequent collision of atomic with molecular oxygen. The ozone is a strong disinfectant with higher oxidation power. However it is potentially toxic and explosive which requires on site generation, safety procedure and caution for use. The European countries were the first to use the ozone

*Correspondingauthore-mail:nadia_ramdani@ymail.com

for potable water. The United States used it in municipal wastewater facilities [9].

The disinfection mechanism depends on pathogen consideration. The inactivation of bacteria takes place through an oxidation reaction leading to the degradation of the membrane followed by cellular lysis. Ozone may also counter and disables enzymatic activity of bacteria through the action on sulphydryl groups. Concerning the viruses the targeted sites of action are the proteiccapcid used by the virion to fix on the cell surfaces and the nucleid acids [10].

The disinfection effectiveness of Ozone (O_3) is generally related to the contact time (t) and the disinfectant concentration (C) according to Chick's law (1908) as:

$$N(t)= No \exp (-kC''t) \qquad (1)$$

Where N(t) and No are the numbers of surviving microorganisms at times t and 0.K and n refer respectively to the coefficient of specific lethality and dilution. It has been shown that dosage of applied Ozone between 5 and 20 ppm and contact time of 5 to 20 minutes which yield 3 to 5log inactivation have been admitted for disinfecting municipal wastewater depending on the fixed microbial target [11]. It has been reported that with such doses the ozone oxidizes potential organic DBP persecutors and generate oxygenated DBP (organic acids) as pointed out by [12-13].

On the other hand, ozone's efficiency on bacteria and virus inactivation for primary and secondary effluents has been demonstrated, achieving reductions of fecal bacteria up to 2- to 3-log [14]. According to these authors, the efficiency of the disinfecting process depends on the specific microorganism, as well as concentration-contact time (CT) values [15].

From the economical point of view, due to the high investment costs of ozone generators, full attention must be given when designing and manufacturing them. Moreover, when determining the energy requirements for ozone generation, the energy for the feed - gas production (oxygen or compressed air) and preparation as well as the energy for the ozone generator must be considered [16-20]. Dielectric barrier discharge (DBD) reactors have been widely studied for optimizing the output generation rate of ozone, according to several parameters such as the high voltage level, the oxygen rate, the frequency of the voltage and other factors [21-27].

The objective of this work is the study of the elimination of organic micro-pollutants and bacteria in urban wastewater treated before their discharge by dielectric barrier discharge (DBD). This process is considered as a new technology in Algeria using a High Voltage DBD as ozone generator. The wastewater treated before and after ozonation has been undergone chemical and microbiological analyses for dosage of inorganic pollutants. The work carried out presents the motives, the experimental bench as well as the necessity to add a quaternary treatment at the wastewater purification station.

2 Materials and Methods

2.1. Sampling

The purification station (WWTP) of Sidi-Bel Abbes in the West of Algeria treats about 220 000 equivalent habitants (EH) with a classical chain of treatment. The process is composed of a pretreatment (sand and grid), primary decantation, a biological treatment by activated sludge followed by a secondary decantation. The primary and secondary sludge are dehydrated then evacuated in drying beds. In order to determine the efficiency of the treatment (WWTP), three samples are taken every day and through the week. The samples were taken before and after the treatment. All the samples were kept at 4°C.

2.2. Chemical analysis

The standard methods of wastewater analysis are described by [28] and also in catalogs of the equipment. The pH, conductivity and turbidity are measured by a pH meter (HANNA type HI 991001), conductimeter (HANNA type HI 8033) and a turbidimeter (LP2000 type) of laboratory respectively. The procedure uses the standardized method (NFT 90-101) to measure COD. The organic matters are oxidized with excess of potassium dichromate ($K_2Cr_2O_7$), in acidic medium (H_2SO_4) and in the presence of a catalyst (Ag_2SO_4). After two hours of heating at 150°C, the COD is determined by volumetric dosing. An OxiTop (VELP SCIENTIFICA) is used to analyze the BOD_5. A spectrophotometer DR2000 type has been used for the determination of phosphorous (PO_4^{3-}), the nitrates (NO_3^-) and the nitrites (NO_2^-).The dose of heavy metals contents (Cd, Cr, Pb, Ni, Cu, Fe, Mn and Zn).Upon arrival in the laboratory, samples were immediately acidified to pH 2.5 with 5 N HCl and filtered at 0.45 µm through glass fiber filters (type GF/F, Whatman), with an atomic absorption spectrophotometer M6 AA.

2.3. Microbial analysis

The detection of bacteria is determined by the classical methods of culture in medium liquid using the most probable number (MPN) technique [29].The instrument used for filtration is a stainless steel reservoir such as used in reference [28].The filtration membrane is made of cellulos is with pore diameter of 0.45 µm. The methods of analyses are given in table (1).

Table (1): Incubation conditions, culture medium and protocol of germs analysis.

Type bacterium	Culture medium	Incubation temperature	Analytical method
Total coliforms	gelosis Lactose : tergitol + TTC	T° = 37°C during 24h	Filtration Technique On Filtration membrane 0,45µm
Fecal coliforms	VRBL (violet red bile lactose)	T° = 44°C during 24h	
Fecal Streptococci	Rothe (presomptive) Litsky (confirmative)	T° = 37°C during 48h	
Escherichia coli	**Brain-Heart**Infusion Broth SS Environment	T° = 37°C during 24h	Enrichment and counting
Salmonella	Hektoen	T° = 37°C during 24h	Enrichment and counting

3. DBD generator

Fig. 1. shows the ozone generator used in this study, which is a dielectric barrier discharge type of reactor equipped with water cooling system. The inner cylindrical high voltage electrode is an adhesive aluminum sheath inserted and glued in a glass tube. The latter reacts as a dielectric barrier with 2.5mm in thickness, with an external diameter of 50 mm and a length of 300 mm. The grounded cylindrical electrode is a stainless steel tube of 240 mm in length and an internal diameter of 52 mm with discharge gap of1 mm in Fig. 2. The ozone generator is fed with 7Kv HT voltage with a frequency of 30 kHz via a voltage transformer from AC 220V and a frequency of 50Hz.

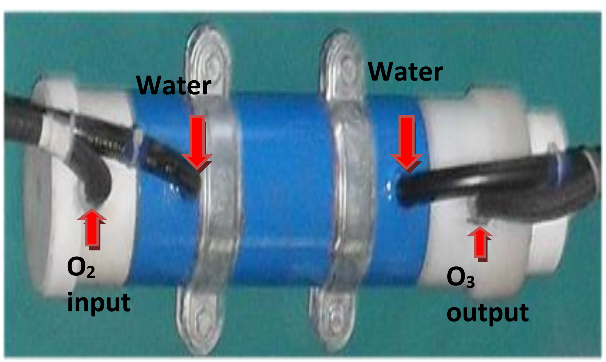

Figure 1: View of the new modified ozone generator with water cooling system

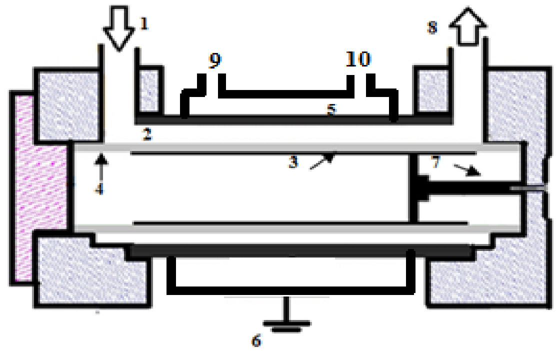

Figure 2: Longitudinal cross section of the developed ozone generator, (1) Oxygen inlet. (2) Gas gap for discharge. (3) Inner cylinder electrode (aluminum adhesive). (4) Dielectric barrier (glass). (5) Outer cylindrical electrode (6) Terminal to ground. (7) High-voltage terminal. (8) Ozone outlet. (9) Cooling water inlet. (10) Cooling water outlet.

3.1. Experimental setup of the ozone treatment system

The experimental setup used for wastewater treatment by ozone is described in Figure 3, 4 and 5. The contaminated water to be treated having a volume of 100 liters (5) is put in circulation by means of a water pump (4). A venture system (3) allows the injection of ozone in the water loop. The ozonized water is considered as treated water which will be reintroduced in the reservoir in a closed circuit. The ozone is produced by three generators developed at the Laboratory and assembled in series (1).They are fed by an oxygen concentrator (6) of medical Nuvo Lite Mark 5 type with a flow rate of 5 l/min.

The ozone concentration in water using the three generators is 6.25 (mg/l) measured with ozone measurement instrument into water (Eco Sensors/US). All the experiments were carried out in stable climatic conditions of temperature (20-25 °C) and humidity (60-70 %).

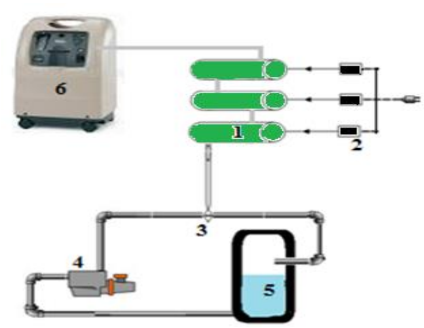

Figure 3: Representation of the experimental installation for the wastewater treatment process by ozone

Figure 4: Photograph of the experimental setup (Front view) 1. Three ozone generators connected in series ; 2- Transformer High-voltage ; 3- Venturi injector ; 4- Water pump; 5- Water reservoir; 6- Oxygen concentrator.

Figure 5: Photograph of the experimental setup (Back view)

4 Results and Discussion

4.1. Physicochemical analyses

The results are presented in such a manner to be easily treated to determine the residual pollution of the treatment station by the activated sludge. The daily evolution of certain physicochemical parameters of raw water and of the treated effluent during a period of three months is given in table 2.

Table (2): Summary of the average results of the physicochemical analyses of wastewater

Conventional parameters	**Raw wastewater**	**Wastewater after treatment**	**OMS Standards**
Temperature (°C)	12.45 (±4.2)	11.50 (±1.1)	30
pH	8.23 (±1.9)	8.31 (±1.2)	$6.5 \leq pH \leq 9$
COD (mg/l)	940 (±55.5)	210.29 (±23.3)	90
BOD_5 (mg/l)	397 (±23.3)	65.7 (±17.3)	30
Turbidity (NTU)	477 (±29.3)	14.59 (±2.3)	/
Conductivity (µs/cm)	1639 (±64.3)	1037 (±74.3)	1200
TSS (mg/l)	950.5 (±88.3)	42.4 (±9.3)	20
$N\text{-}NH_4$ (mg/l)	8.26 (±1.3)	2.57 (±0.9)	<0.5mg/l
$N\text{-}NO_3$ (mg/l)	0.09 (±0.02)	0.002 (±0.04)	1mg/l
$N\text{-}NO_2$ (mg/l)	0.03 (±0.01)	0.005 (±0.03)	
PO_4^- (mg/l)	3.3(±0.96)	1.10 (±0.93)	<20mg/l
Purification ratio (%)	85.53		-

The results show that, after the biological treatment with activated sludge in the WWTP, there is a noticeable reduction of the pollution parameters of organic types such as COD and BOD_5 but the values remain above the Standards [30].This allows concluding that the treatment in the aeration zone eliminate only 2/3 of the total soluble organic pollution. However they cannot be discharged in the environment. It has been observed that some values of physic chemical parameters are higher than the standard values such as:

- The presence of matter in suspension due to the urban nature of wastewater
- High BOD_5even with the biological purification which is explained by:
- Insufficient of aeration in the biological basin.
- Presence of oils on the surface of the water in the aeration basin which reduces the oxygen penetration.

The presence of heavy metals in the untreated wastewater is interpreted by the discharge from gasoline and cleaning stations of the city which do not respect the regulation of discharge in the wastewater network. After purification it is observed the presence of heavy metals. This is explained by the lack of chemical treatment in the purification stations. In this work it has been shown that the WWTP of the city of Sidi-Bel-Abbes do not have sufficient treatment techniques such as: chemical treatment and nonsufficient aeration in biological basin. The efficiency of the treatment depends on the time of stay of the wastewater in the basin and on the dropping speed f matter in suspension. The MES decantation drains with them micro-pollutants and micro-organisms [31].

According to the results obtained it has been observed that the wastewaters of the city are partially treated with a purification ratio of 85.53% then discharge in Oued Mekerra meeting the untreated waters which are not linked to the WWTP.

In summary, even with a reduction of micro-organisms and micro-pollutants during the biological aerobic treatment at activated sludge, the treated water coming from the WWTP are highly charged with pathogens. In order to solve the problem of pollution and its impact on the environment and human health, we suggest the addition of a quaternary ozone treatment at the station of purification.

4.2. Impact of ozonation on the effluent water quality

The results of the present study quantify the response of wastewater effluents through many key parameters of ozonation. The reductions of these parameters in the samples used for test with ozone have been carried out during a period of 30 min for the physicochemical parameters and during 5 min in ozonation for microbiological parameters. A series of analyses were carried out for treated wastewater in order to evaluating the effect of the treatment by ozone and optimizing the efficiency of the process.

4.2.1. Treatment by ozone effect on the BOD_5

The BOD_5 is the concentration of the diluted oxygen consumed by microorganisms for oxidizing the diluted organic substances or in suspension in the wastewater [32]. In this study BOD_5 is to quantify the biodegradable fraction measured of carbonized and organic pollutants in wastewater. It is noticed that the values of BOD_5 concentration varies from 65.7 à 29mg/l after 30min of ozonation treatment as shown in figure 6. The improvement of the biodegrability induced by ozonation has been mentioned by many works in diverse wastewaters [33-34]. In the first stage of ozonation, the improvement in biodegradability has been attributed to formation of smaller, oxygenated species more suitable to microbial attack, and possibly to the reduction of compounds with bactericidal proprieties [35].

These results have allowed to show the increase of biodegrability of organic matter considering that the residual concentration of DBO_5 (65.7 mg/ l) stays higher than the Algerian rejection standards (35 mg/ l) [36]and WHS standards (30 mg/l) [37]as well as to the extreme standards limited to water of irrigation (30 mg/l) [30].The load of biodegradable organic matter after ozonation (47 mg/l) and biological treatment (65.7 mg/l) is due to the abundance of the bacterial population responsible for this elimination as indicated thereafter in one hand, and, to the decrease in the oxygen content due to its consumption by micro-organisms on the other hand [38-39].

The ozone acts on the longest carbonized chains with difficult biodegrability. It breaks and transforms them into acid organic chains which have difficult biodegrability by chemical oxidation but easily degradable by biological oxidation.

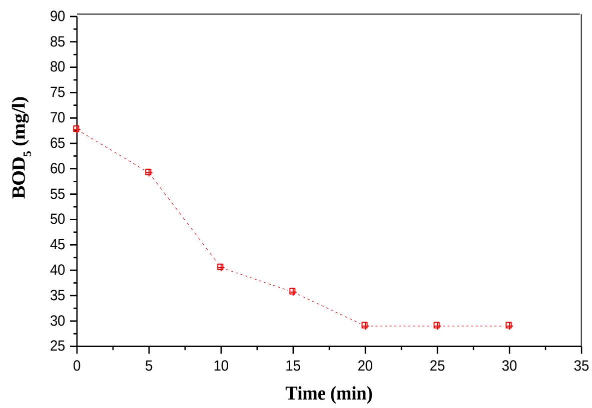

Figure 6: The effect of DBD treatment on BOD_5

4.2.2. The effect of treatment by ozone on COD

The value of the COD indicates the quantity of oxygen required for oxidation for all organic substances in the in the wastewater [40]. In this study the concentration of COD was an important parameter used to evaluate the pollutants of wastewater. Previous studies had shown the high reduction of COD during ozonation [41-42]. In this study the concentration of COD is an important parameter used to evaluate the pollutants in wastewater. Previous studies had shown the high COD reduction during ozonation. Figure 7. shows the reduction of the COD with respect to time during 30 min of treatment.

The decrease of COD is due to the degradation of cyclic compounds. Afterwards the reduction slows down due to the degradation of aliphatic compounds which require higher energy than the cyclic compounds.The COD reduction in the effluent finds its explanation in the good performance of the treatment technique. In addition, the value of COD after ozonation (85.2 mg/l) remains very low compared to that after biological treatment (210.29 mg/l). We also note that the COD obtained, after ozonation, remains lower than the Algerian standard of rejections (120 mg/l) [36], thus, to the World Health Organization standard (90 mg/l) [37].The previous application of ozone treatment allows a significant improvement of COD reduction following the biological oxidation [41-42].

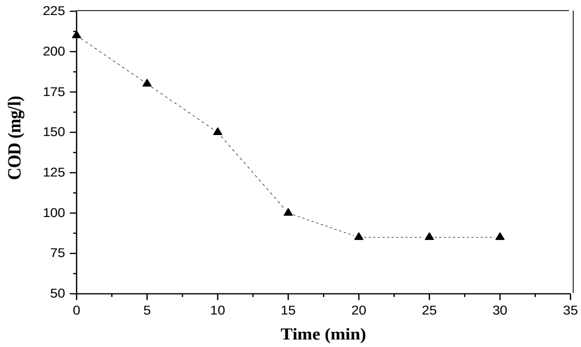

Figure 7: The effect of DBD treatment on COD

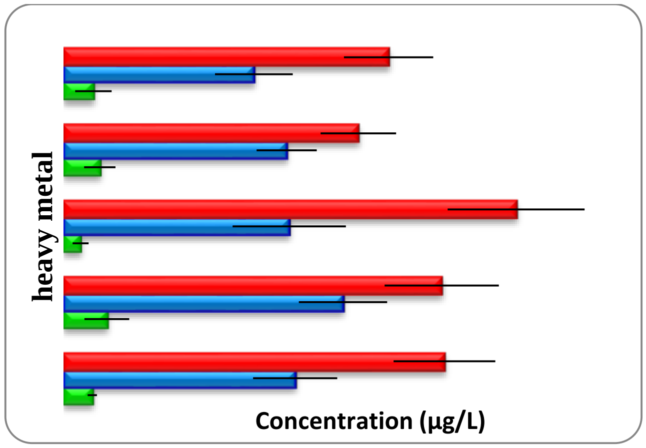

Figure 8: Elimination of inorganic micro pollutants in the WWTP after ozonation process: Input WWTP (■), Output WWTP (Before ozonation) (■), Output (After ozonation) (■)

4.3. Heavy metals removal

The analysed substances had been selected with respect to their occurrence in the wastewater [43-44], for their physicochemical properties (mainly non biodegrable substances and hydrophyls), for their toxicity and regulation. Also taking into consideration their limit of quantification as well as the availability of a reliable analytical method. All the studied technologies emerge on the considered application which is the treatment of micro pollutant in wastewater.In this work we have considered the determination of the following elements: Cd, Cr, Pb, Ni, Cu, Fe, Mn and Zn which are the most studied heavy metals in surface waters. These metals even with their low concentrations, their ecological and sanitary impacts can be important.The analysis of the wastewater treated at the WWTP exit reveals the absence of the following heavy metals: Pb, Cd and Ni in figure 8 and the presence of Fe, Zn, Cu, Cr and Mn.

The obtained data show that the ozonation offers good results. Certain inorganic substances are well oxidized by the ozone process. The figure 8 summarizes the main results obtained in term of efficiency in eliminating micro pollutants with respect to conventional (primary and secondary) treatment of domestic wastewater (>80%). The efficiency of these processes for the elimination of the different classes of micro pollutants in the discharged effluent by the municipal WWTP had been demonstrated [45-47]. From this study the ozone process seems to be a competitive solution.

5 Microbiological Analyses

The nature of microbial population is diverse in wastewater [28].The bacteria are usually searched in water as indicators of fecal contamination [48].

The WHO (1989) has selected many indicators meeting certain requirements. This concerns the coliforms, group D fecal streptocoques, E. coli, Salmonella and sometimes *Clostridium perfringens*. Water can play the role of a vector of potentially dangerous microbial agents such as Salmonella, choleric vibrions and parasites [49]FC and E. coli were chosen as standard fecal indicators in this study, because they are usually regulated for wastewater discharge or reuse [50].

According to figure 9 the results of the treated wastewater analysis reveal the presence of gems indicators of fecal contamination and certain pathogen germs. It appears the used biological treatment in the WWTP is not efficient for destroying the germs. The obtained results can be explained by the fact that the biological treatment used in the WWTP favors bacterial growth to degrade the carbonized pollution and by the lack of disinfection treatment necessary to destroy pathogen germs. The wastewater is also considered as an optimal environment for microbial proliferation.

The evolution and abundance of pathogen germs (fecal Coliform FC, Total Coliform TC, Fecal Streptocoques FS, Salmonella SM and E. coli EC) during the treatment by ozone is show in figure 9.

5.1. Treatment effect on the TC, FC, SF, SM and E. coli

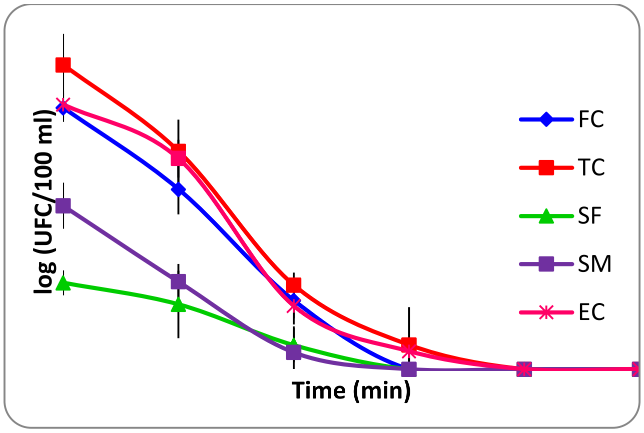

Figure 9: Kinetics of germs inactivation: fecal Coliform (■), total Coliformes (■), Fecal Streptocoques (■), Salmonella (■) and E. coli (■) during 30 min of DBD treatment. The values represent the average of 3 determinations ± SD (n=3).

After 5 min of treatment a loss of fecal coliform viability equivalent to -1.78 log UFC/ml has been recorded corresponding to an inhabitation speed of - 0.0595 log UFC/h. The inhibition accentuates after 10 min of cell exposure to DBD treatment showing an inhibition percentage of -73.70% equivalent to an inhibition speed of -0.448 log UFC/h. After 15 min of treatment it is observed the total inhibition of the FC. It is noticed that this treatment has exerted an effect of total inhibitor on Total Coliform only after the 20thminute.We notice the high reduction of E. Coli during the first 10 min which is equivalent to -4.41 log ufc/ml (-76.14% of cells).research works have shown damage in the cellular membrane at nucleic acids (ADN) during its activation, which can only be produced at higher concentrations of ozone [51].

The inhibition action of ozone continues with respect to Fecal streptocoques continues to drop after 10 minutes of treatment by -1.36 log UFC/ml (representing – 72.81%) of the recorded biomass with an inhibition speed of -0.1316 log UFC/h. This inhibition was very important with (-100% loss of viability) at the 15th minute reaching a total inhibition. The search for Salmonella revealed its presence (3.58 log UFC/ml) in the biologically treated water (at 0 min) while we observe its total inexistence in the water treated by ozone after 15 minutes of contact.

The efficiency of disinfection depends on the DBD, the turbidity, of the number and types of microorganisms present in the effluent [52- 53]. Our results show that this good inactivation of bacteria is due to a high ration of reduction of these different parameters particularly the turbidity which can rise the rate of disinfection. The process contributes to the total reduction of these bacteria and consequently became unable to develop their immunity with the presence of ozone [54]. The comparison was also made on the ratio of bacteria between the treated water by ozone and that of wastewater inactivation treatment station. The most important inactivation is obtained by the process of treatment by ozone as is shown in figure 9. The proportions of inactivation observed in this study show that the process of treatment used creates certain efficiency in the bacterial disinfection with respect to the process of purification treatment station in particular for E. coli [55].

The wastewater treatment by ozone besides on being a very performing process, does not generate any toxicity and hence improves the quality of the effluent (color, nitrites, COD, MES). A reduction of Total Coliforms, Fecal Coliforms and Fecal Streptocoques of more than 3 log UFC/ml was observed by [56]for a treatment by ozone (for 20 min contact time).

5.2. Model of Chick-Watson

The Chick-Watson’s classical first order model defines the inactivation rate of certain bacteria by ozone in particular E. coli. The Total coliform and Fecal coliform have revealed a regression of 5.73 log and 6.67 log in 10 min, with 73.70 and 72.36% reduction respectively in Figure 10.

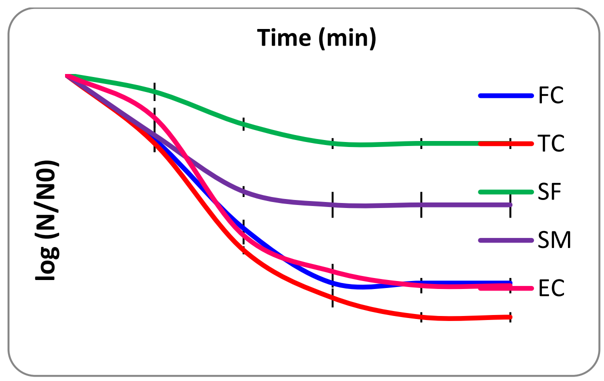

Figure 10: Degradation Kinetics of germs by ozone : fecal Coliform (■), total Coliformes (■), Fecal Streptocoques (■), Salmonella (■) and E. coli (■) during 30 min of DBD treatment. The values represent the average of 3 determinations ± SD (n=3)

[57] have shown reduction in FC in the order of 2.48 log to a concentration of ozone transferred from 15.2 mg/l and that can exceed 3 log even before reaching the demand of ozone [5-57].The three types of bacteria have a very important order of reduction and superior to that found by these studies which certainly shows the presence of ozone in high concentrations and their rapid bactericidal action. However, the fecal streptocoques show a resistance to the treatment by ozone where it is observed a reduction of 1.89log at the 15th minute.

6 Conclusions

The present research has tackled the problem of wastewater treatment in the zone of Sidi-Bel-Abbes city which has an important number of industrial plants requiring an higher need in water supply and facing a degradation of water resource linked to the environmental pollution. The results confirm that conventional treatments (decantation and biology) have a partial elimination of about an average less than 60 %. The treatment by ozone produced by DBD allows eliminating more than 80% of most of the analyzed chemical substances. The bacteriological analysis shows the quasi-complete disappearance of pathogen agents in the wastewater. The study shows the efficiency of the ozone treatment process which we may consider as an alternative or a complement to the one used in the wastewater treatment plant.

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Symbols

AC - Alternating current

BOD -Biochemical oxygen demand, mg/l

COD -Chemical oxygen demand, mg/l

CFU - Colony-forming unit

DBD - Dielectric barrier discharges

TS - Total suspended solids

E. coli - Escherichia coli, CFU/100 ml

FC - Fecal coliforms, CFU/100 ml

MPN -Most probable number

NTU -Nephelometric turbidity unit

pH - Potential hydrogen

TC - Total coliforms, CFU/100 ml

WWTP - Wastewater treatment plant

C - Concentration, mg/l

T - Time, min

WHO - World Health Organization.

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