Using Peroxymonosulfate-Ozone Advanced Oxidation For The Treated Wastewater Disinfection and Amoxicillin Micro-Pollutant Removal Simultaneously

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Abstract

Due to the recent efforts to improve the conventional disinfection methods efficiency of wastewater treatment plants effluent, in this study, the efficiency of the peroxymonosulfate-ozone (PMS+O_3) advanced oxidation process in lab scale by the aim of disinfection and simultaneous removal of existing amoxicillin micro-pollutant under optimum operational condition was investigated for the first time. Furthermore, the results were compared with those obtained from the experiments conducted employing persulfate-ozone (PS+O_3), hydrogen peroxide-ozone (H_2O_2+O_3), and ozonation (O_3) processes. For this purpose, the main parameters including the total coliforms, amoxicillin concentration, turbidity, chemical oxygen demand (COD), biochemical oxygen demand (BOD_5), total nitrogen (TN), electrical conductivity (EC), total dissolved solids (TDS), and total suspended solids (TSS) were considered. The test results show that under optimized operational conditions (retention time of 20 minutes, ozone dosage rate of 0.83 mmol/L, and peroxymonosulfate concentration of 0.06 mmol , 99.99% total coliforms (e.g., the number of total coliforms reached consistently less than 400 MPN in 100 ml) removal was reached by peroxymonosulfate-ozone advanced oxidation process. Also, amoxicillin concentration removal efficiency reached 90±2%. In comparison, although the total coliforms reduction of PS+O_3 and H_2O_2+O_3 methods in 30 min are approximately the same, the amoxicillin concentration removal efficiency is about 60-70%. Due to the importance of ensuring effluent quality, the related removal efficiency of other considered parameters is also evaluated and presented. Eventually, the peroxymonosulfate-ozone method can be considered as a novel efficient approach for wastewater plants effluent disinfection and amoxicillin micro-pollutant removal simultaneously which is a novel approach.

1. Introduction

Although reusing the treated wastewater for urban and agricultural purposes has been considered as a part of integrated management of extractable water resources (US Environmental Protection Agency 2012; Ofori et al. 2021), the risk of pathogenic microorganisms discharges in the environment caused by inappropriate disinfected wastewater is one of the important concerns (Nasuhoglu et al. 2018). Therefore, the necessity of an efficient and ensured disinfection method for effluent of urban wastewater treatment plants is obvious (Malato et al. 2009).

In recent decades, pharmaceutical compounds residuals have been considered as the most important water contaminant due to their wide variety, high consumption, and stability in the environment (Homem and Santos 2011; Zaied et al. 2020). Among various pharmaceutical compounds, special attention has been given to the antibiotics due to their capability of producing antibiotical resistance in pathogen bacteria (Dimitrakopoulou et al. 2012; Zhou et al. 2021). More than 65% of consumed antibiotics in the world belong to the β-lactam group (Githinji et al. 2011). Amoxicillin is a broad-spectrum β-lactam antibiotic (with a chemical formula of $C_{16}H_{19}N_3O_5S$ and a molecular weight of 365.4 g/mol) which belongs to penicillin group and is used systematically for the treatment of gastrointestinal bacterial infections in medicine and veterinary medicine (1998; Putra et al. 2009; Gao et al. 2020). In the previous studies, some methods were used to remove amoxicillin from water sources including: biological adsorption, advanced oxidation processes (AOPs), ion-exchange, coagulation/flocculation and combination of these methods (Kanakaraju et al. 2018; García-Menéndez et al. 2020; Rekhate and Srivastava 2020; Jalali et al. 2021). In the present study, removal of amoxicillin and disinfection of treated wastewater was conducted simultaneously using peroxymonosulfate-ozone advanced oxidation process.

Ozone is a powerful disinfectant and oxidant that is traditionally applied for water and wastewater treatment and higher disinfection efficiency compared to chlorination and ultraviolet (UV) radiation processes (Verma et al. 2015). In real experience, ozone is quite selective in the oxidation of organic compounds, and it has a very low reactivity with aromatics compounds (such as amoxicillin) (Oh et al. 2003). So, the advanced oxidation processes (AOPs) were used to dominate the ozone limitation.

Using advanced oxidation methods result in the production of hydroxyl radical (OH°) (E°=2.8), (Rodríguez-Chueca et al. 2017; Badalians Gholikandi et al. 2018) which has high reactivity and acts in a non-selective way (Gholikandi et al. 2017b). The results of studies of recent years have always indicated the capability of advanced oxidation methods in significant removal of the microbial community in the tested specimens (Badalians Gholikandi et al. 2014, 2018; Gholikandi et al. 2017a, b; Gholikandi and Kazemirad 2018; Masihi and Badalians Gholikandi 2018; Rasouli Sadabad and Badalians Gholikandi 2018). In the last decade, studies on advanced oxidation processes (AOPs) based on sulfate have increased (Guerra-Rodríguez et al. 2018). Sulfate radicals have high oxidation reactivity (E = 2.5-3.1V) (Cong et al. 2015; Wu et al. 2019) and acceptable performance at a wide range of pH values of 4–9 (Ren et al. 2015). They are often obtained by activating peroxymonosulfate (PMS: HSO_5^-) and persulfate (PS: $S_2O_8^{2-}$) using ozone, heat, UV, ultrasound, or heterogeneous and homogenous catalysts (Alkhuraiji et al. 2017; Rodríguez-Chueca et al. 2017; Wacławek et al. 2017; Wang and Wang 2018a, b; Latif et al. 2019). Studies have been conducted on sulfate-based AOP methods for deactivation of pathogenic Escherichia coli (Wordofa et al. 2017; Xia et al. 2018). Ozone/hydrogen peroxide (O3/H2O2) is also used in water treatment facilities to remove many organic micropollutants. The O3/H2O2 process, also known as peroxone AOP, uses a radical chain system to decompose ozone, which is activated by the hydroperoxide anion HO_2^-(Rekhate and Srivastava 2020). Badalians Gholikandi et al. (2018) conducted a comparative study on sludge stabilization using H_2O_2 + O_3, PMS + O_3, PS + O_3, and O_3 methods and found that PMS + O_3 had a better performance than the other methods (Badalians Gholikandi et al. 2018).

In this study, the PMS + O_3 advanced oxidation process which is able to produce sulfate and hydroxyl radicals simultaneously was employed to remove total coliforms and amoxicillin micropollutant from the urban wastewater treatment plant effluent. Also, the obtained results were compared with ozonation, hydrogen peroxide-ozone, and persulfate-ozone methods capability. The comparison is made in the first instance based on the two main parameters, e.g., total coliforms and amoxicillin removal. Further, the main parameters for removal efficiency under optimized operational conditions as the main considered parameters relating to wastewater treatment plants effluent quality were analyzed including the total coliforms, amoxicillin concentration, turbidity, chemical oxygen demand (COD), biochemical oxygen demand (BOD_5), total nitrogen (TN), EC, total dissolved solids (TDS), and total suspended solids (TSS).

2. Materials And Methods

2.1. Materials

2.1.1. Treated wastewater Sample (before disinfection)

The treated wastewater samples (wastewater treatment plant effluent before chlorination) used in this experiment were taken daily from the activated sludge urban wastewater treatment plant which is located in the north east Tehran. Characteristics of the treated wastewater samples are listed in Table 1.

Table 1 The characteristics of the treated wastewater samples.

Parameter	Unit	Value
pH	-	7.1 ± 0.2
Total coliform	MPN in 100 milliliter	$(2.1 \pm 0.3)\times10^6$
Turbidity	NTU	10 ± 3
Chemical oxygen demand (COD)	mg/L	34 ± 5
Biological oxygen demand (BOD_5)	mg/L	20 ± 5
Total Nitrogen (TN)	mg/L	40 ± 5
Total phosphorus (TP)	mg/L	10 ± 2
Temperature	˚C	18 ± 1
Electrical conductivity(EC)	µS⁄cm	715 ± 20
Total dissolved solid (TDS)	mg/L	453 ± 50
Total suspended solids (TSS)	mg/L	18 ± 5

2.1.2 Test Setup

A cylindrical reactor in a laboratory scale with a diameter of 3 cm and a height of 40 cm was used to conduct the studies (Fig. 1). The ozone generator (Arda Company, Ozoneplus series-COG high voltage) with a capacity of 500 mg/h was employed to produce ozone. In addition, two gas washing bottles were attached in series to test the reactor's output ozone.

2.1.3. Used materials

Potassium peroxymonosulfate, potassium persulfate, and hydrogen peroxide (35%) purchased from Merck Company and amoxicillin made by Sigma-Aldrich Company were utilized in this experiment.

2.2. Experimental procedure

In order to measure the Total Suspended Solids (TSS) concentration, Total Dissolved Solids (TDS) concentration, Chemical Oxygen Demand (COD), Biological Oxygen Demand (BOD_5), Total Nitrogen (TN) concentration, Total Phosphorus (TP) concentration, ozone concentration, and total coliform, the following methods were used respectively: 2540D, 2540C, 5220D, 5210B, 4500N-C, 4500B-C, 2350E, and 9221B standard methods (APHA 1992). Also, HANNA pH meter-211, OSK 14821 conductivity meter, and Lovibond turbicheck devices were used to measure pH, electrical conductivity, and turbidity, respectively. In order to measure the concentration of amoxicillin, the Jenway 6315 UV-spectrophotometer at a wavelength of 228.3 nm was used (Weng et al. 2013). The experiment was conducted in the reactor shown in Fig. 1. The initial concentration of amoxicillin was considered to be 36.5 mg/L because the concentration of more than 36.5 mg/L leads to minimal inhibitory for the test organisms (Moreira et al. 2015). The concentration of amoxicillin was zero at effluent (treated wastewater sample), and amoxicillin was added synthetically to the samples.

3. Results

3.1. Reduction of total coliform and amoxicillin

In the present study, the effect of using $H_2O_2 + O_3$, PMS + O_3, PS + O_3, and O_3 on the removal of total coliform has been studied, and the optimum operational conditions have been determined. In these processes, radicals of hydroxyl or sulfate (or both of them) act as oxidation agents. Therefore, increasing the production of these agents leads to an increase in the removal of total coliform. An allowable limit for the total coliforms, e.g., 400 MPN in 100 ml, was considered as the criterion for successful disinfection of wastewater (in this way, the allowable limit criterion for the presence of fecal coliform according to the wastewater reuse standards for agricultural purposes is met, too). Regarding the importance of the removal of specific contaminants with a pharmaceutical origin, the amoxicillin removal as one of the commonly residual drugs in wastewater was also investigated. Also, the theoretically required ozone dosage to disinfect secondary clarifier output is 0.083 to 0.2 mmol/L (Tchobanoglous et al. 1990) that in this study the dosage of ozone was considered to be 0.083 mmol/L which is the least dosage needed for ozonation according to Metcalf and Eddy (Metcalf and Eddy 2003).

3.1.1. Ozone and hydrogen peroxide ($H_2O_2 + O_3$)

As shown in Fig. 2, in the absence of H_2O_2, when only the ozone participates in the reaction, the amount of the log (MPN) decreases after a reaction time of 30 min from 6.3 to 3.7, e.g., the number of coliforms becomes from 2100000 to 5300 MPN/100 ml. In this method, the production of hydroxyl radical is occurred based on Reaction 1 (Munter 2001). After starting the hydrogen peroxide addition, when its dosage reaches 0.35 mmol/L, the amount of log (MPN) decreases after a reaction time of 30 min from 6.3 (2100000) to 2.8 (624). In the same reaction time, by increasing the dosage of hydrogen peroxide to 0.7 mmol/L and then 1.05 mmol/L, the amount of log (MPN) reduces from 6.3 (2100000) to 2.27 (185) and 1.81 (65), respectively. Based on the obtained results, by increasing the dose of H_2O_2, the amount of total coliform removal, increases, too. The highest amount of removal is for H_2O_2 dosage of 1.05 mmol/L in a reaction time of 30 minutes. However, the optimum operating conditions of the reactor to achieve the allowable maximum coliforms number of 400 in 100 ml can be obtained by H_2O_2 dosage of 0.7 mmol/L and a reaction time of 20 min, resulting in log (MPN) = 2.57 (e.g., 371 MPN/100 ml < 400).

$H_2O_2 + 2O_3 \rightarrow 2OH^{\circ} + 3O_2$ (1)

3.1.2. Ozone and peroxymonosulfate (PMS + O_3)

As shown in Fig. 3, in the absence of PMS, when only ozone participates in the reaction, the amount of the log (MPN) after a reaction time of 30 minutes, reduces from 6.3 (2100000) to 3.7 (5300). In this method, the production of hydroxyl and sulfate radicals is carried out based on Reactions 2–9 (Yang et al. 2015, 2016; Badalians Gholikandi et al. 2018). After starting the dosing of peroxymonosulfate, while its concentration reaches 0.03 mmol/L, the amount of log (MPN) after a reaction time of 30 min reduces from 6.3 (2100000) to 3.12 (1324). In the same reaction time, by increasing the dosage of peroxymonosulfate to 0.06 mmol/L and then 0.09 mmol/L, the amount of log (MPN) reduces from 6.3 (2100000) to 2.16 (145) and 1.98 (95), respectively. Based on the obtained results, it can be said that by increasing the dose of PMS, the amount of total coliform removal, increases, too. The highest removal efficiency is by a PMS dosage of 0.09 mmol/L in a reaction time of 30 min. It should be noted, however, that the optimum operational conditions for the reactor to achieve the allowable limit of maximum 400 coliforms in 100 ml in a PMS dosage of 0.06 mmol/L, can be obtained by achieving log (MPN) = 2.16 (e.g.145 MPN/100ml) in a reaction time of 20 minutes.

$O_3 + OH^- \rightarrow HO_2^- + O_2$ (2)

$O_3 + HO_2^- \rightarrow OH_2^{\circ} + O_3^{-\circ}$ (3)

$O_3^{-\circ} + H_2O \rightarrow OH^{\circ} + O_2 + OH^-$ (4)

$KSO_5 \leftrightarrow K^+ + HSO_5^-$ (5)

$HSO_5^- \rightarrow H^+ + SO_5^{2-}$ (6)

$SO_5^{2-} + O_3 \rightarrow SO_5^{-\circ} + O_3^{-\circ}$ (7)

$SO_5^{-\circ} + O_3 \rightarrow SO_4^{-\circ} + 2O_2$ (8)

$2SO_5^{-\circ} \rightarrow 2SO_4^{-\circ} + O_2$ (9)

3.1.3. Ozone and persulfate (PS + O_3)

As shown in Fig. 4, when the dosage of PS is zero, the amount of the log (MPN) reduces after a reaction time of 30 min from 6.3 (2100000) to 3.7 (5300). In this method, the production of sulfate radical is carried out according to the reaction 10 (Reisner, 2016). When the injection of persulfate is started, and its dosage reaches 1 mmol/L, the amount of log (MPN) decreases after 30 min from 6.3 (2100000) to 3.36 (2298). In the same reaction time, by increasing the dosage of persulfate to 2 mmol/L and then 3 mmol/L, the amount of log (MPN) reduces from 6.3 (2100000) to 3.04 (1105) and 2.64 (435), respectively. Based on the obtained results, it can be said that by increasing the dose of PS, the amount of total coliform removal, increases, too. The highest amount of removal is for a PS dosage of 3 mmol/L in a reaction time of 30 minutes.

$S_2O_8^{2-} + OH^{\circ} \rightarrow SO_4^{-\circ} + SO_4^{-\circ} + 1/2O_2 + H^+$ (10)

3.1.4. Amoxicillin removal

Amoxicillin was not present in treated wastewater. The concentration of 36.5 mg / L of amoxicillin was added synthetically to the samples. Under optimum conditions of methods, their ability to remove amoxicillin was studied. PMS + O3, H2O2 + O3, and PS + O3 methods reduced amoxicillin levels by 90, 62.5 and 67.5%, respectively, in addition, to reduce total coliforms to the standard limit. Also, O_3 alone reduced the amount of amoxicillin by 53.6%. As the results showed, the PMS + O_3 method had the best performance and had the ability to disinfect and reduce the micropollutant simultaneously. As it can be seen from the results, high concentrations of H_2O_2, PS and PMS did not further increase the efficiency of the process which can be due to the fact that the excessive dose of oxidants could not act as scavenger of $^{\cdot}OH$ and $SO_4^{\cdot-}$ and also it can facilitate the abundant $SO_4^{\cdot-}$ to transform into the useless ions which can cause a decrease in removal of amoxicillin.

3.2. Other Parameters analysis

The effect of using $H_2O_2 + O_3$, PMS + O_3, and PS + O_3 on the qualitative parameters of wastewater discharge including turbidity, COD, BOD_5, TN, TP, EC, TSS, and TDS has been investigated under optimum conditions of disinfection, the results of which are shown in Tables 2 and 3.

Table 2 The effect of methods on characteristics of the treated wastewater sample

Parameter		Unit	Effluent	Methods			
				PMS + O_3	PS + O_3	H_2O_2 + O_3	O_3
optimal Condition	Time	min	-	20	30	20	30
	Dosage of the PMS,PS, H_2O_2 and O_3	mmol/L	-	0.06	3	0.7	0.083
Turbidity		NTU	7.39	2.78	2.89	3.43	4.21
Chemical oxygen demand (COD)		mg/L	36	23	34	16	35
Biological oxygen demand (BOD_5)		mg/L	21	16	19	13	19
Total nitrogen(TN)		mg/L	38.67	38.67	38.67	38.67	38.67
Total phosphorus(TP)		mg/L	10.5	10.5	10.5	10.5	10.5
Temperature		ºC	17.8	17.6	17.6	17.6	17.8
Electrical conductivity(EC)		µS/cm	709	878	1695	692	712
Total dissolved solids (TDS)		mg/L	471	569	1154	470	472
Total suspended solids (TSS)		mg/L	15.6	3.8	4.2	5.1	10.2

Table 3 The removal efficiency of methods on turbidity, COD, BOD, and TSS of the treated wastewater sample

Parameter		Unit	Methods			
			PMS + O_3	PS + O_3	H_2O_2 + O_3	O_3
optimal Condition	Time	min	20	30	20	30
	Dosage of the PMS,PS and H_2O_2	mmol/L	0.06	3	0.7	0.083
Turbidity removal		%	62.4	60.9	53.6	43
COD removal		%	36	5.56	55.56	2.7
BOD removal		%	23.8	9.5	38.1	9.5
TSS removal		%	75.6	73.1	67.3	34.6

Based on the results, a comparison between investigated methods under optimum operational conditions at a temperature of 17.6°C and constant ozone dosage rate of 0.083 mmol/L shows the following removal efficiencies: turbidity removal of PMS + O_3 (= 62%), PS + O_3 (= 61%), and H_2O_2 + O_3 (= 54%); COD removal of H_2O_2 + O_3 (= 56%), PMS + O_3 (= 36%), and PS + O_3 (= 6%); BOD_5 removal of H_2O_2 + O_3 (= 38%), PMS + O_3 (= 24%), and PS + O_3 (= 10%); TSS removal of PMS + O_3 (= 76%), PS + O_3 (= 74%), and H_2O_2 + O_3 (= 67%), and amoxicillin removal of PMS + O_3 (= 90 ± 5%), PS + O_3 (= 67 ± 5%), and H_2O_2 + O_3 (= 62 ± 5%). The EC value at the temperature of 17.6°C was H2O2 + O3, PMS + O3, and PS + O3, respectively as the lowest values. TDS for the raw sample was measured 471 mg/L, whereas it reached 569 mg/L, 1154 mg/L, and 470 mg/L using the PMS + O_3, PS + O_3, and H_2O_2 + O_3 methods, respectively. None of the methods had an effect on the TN and TP concentrations.

4. Overall Discussion

In this study, treated wastewater effluent disinfection and residual amoxicillin removal employing PMS + O_3, PS + O_3, H_2O_2 + $O_{3,}$ and O_3 alone with the aim of secure availability for reuse purposes were investigated. Under the same operation conditions, O_3 alone is incapable of achieving coliforms reduction requirement in this regard. The PMS + O_3 process is an appropriate method to reach efficient disinfection of wastewater treatment plants effluent and to achieve an effective residual amoxicillin removal simultaneously. In comparison to other investigated methods, higher efficiency in turbidity and TSS removal is achievable. Regarding other parameters, e.g., COD and BOD_5, the removal efficiency is still considerable. Summing up the results, it can be concluded that this method is very useful for effluent of a pharmaceutical sewage treatment plant. Although this method is incapable of removing existing TN and TP concentrations, adverse byproducts generation in reaction with these parameters is not feasible.

5. Conclusion

Special attention has been given to the reliable usage of urban sewage treatment plants' wastewater in agriculture and greenspace irrigation in recent decades. Especially optimum disinfection of wastewater discharge is one of the main prerequisites of reuse. In this study, a new approach for improving the performance of ozone disinfection method by simultaneous usage of ozone compared to ozone/persulfate and ozone/hydrogen peroxide has been studied. The focus of recent researches has been on the presence of micropollutants in sewage treatment plants' wastewater, including the residue of pharmaceutical materials, therefore in this research the possible amount of amoxicillin removal under optimum conditions of the mentioned methods was evaluated. Although the obtained results indicate an almost identical performance of disinfection for the studied methods, the amount of amoxicillin removal in ozone/peroxymonosulfate method was higher than the other ones. It was also found that this method can remove amoxicillin and similar pharmaceutical materials in addition to wastewater disinfection. Therefore, the ozone/peroxymonosulfate method is proposed as a new novel approach for amoxicillin removal. Complementary study on the possible byproducts of the process on a semi-industrial scale is essential in follow-up to this research.

Abbreviations

˚C	Centigrade
g	gram
h	hour
L	Liter
μS	microsiemens
mg	milligram
ml	milliliter
mm	millimeter
mmol	millimole
min	minute
MPN	Most Probable Number
nm	nanometer
NTU	Nephelometric Turbidity Unit
V	Volt

Declarations

Ethics approval and consent to participate:

Not applicable

Consent for publication:

Not applicable

Availability of data and materials:

The datasets used and/or analysed during the current study are available from the corresponding author on reasonable request.

Competing interests:

The authors declare that they have no competing interests

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Authors' contributions:

All authors contributed to the study conception and design. Material preparation, data collection and analysis were performed by Atefeh Mollazadeh, Hamidreza Masihi and Gagik Badalians Gholikandi. The first draft of the manuscript was written by Hamidreza Farimaniraad and Atefeh Mollazadeh. All authors commented on previous versions of the manuscript. All authors read and approved the final manuscript.

Conflicts of interests:

The authors have no conflicts of interest to declare that are relevant to the content of this article.

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Figures

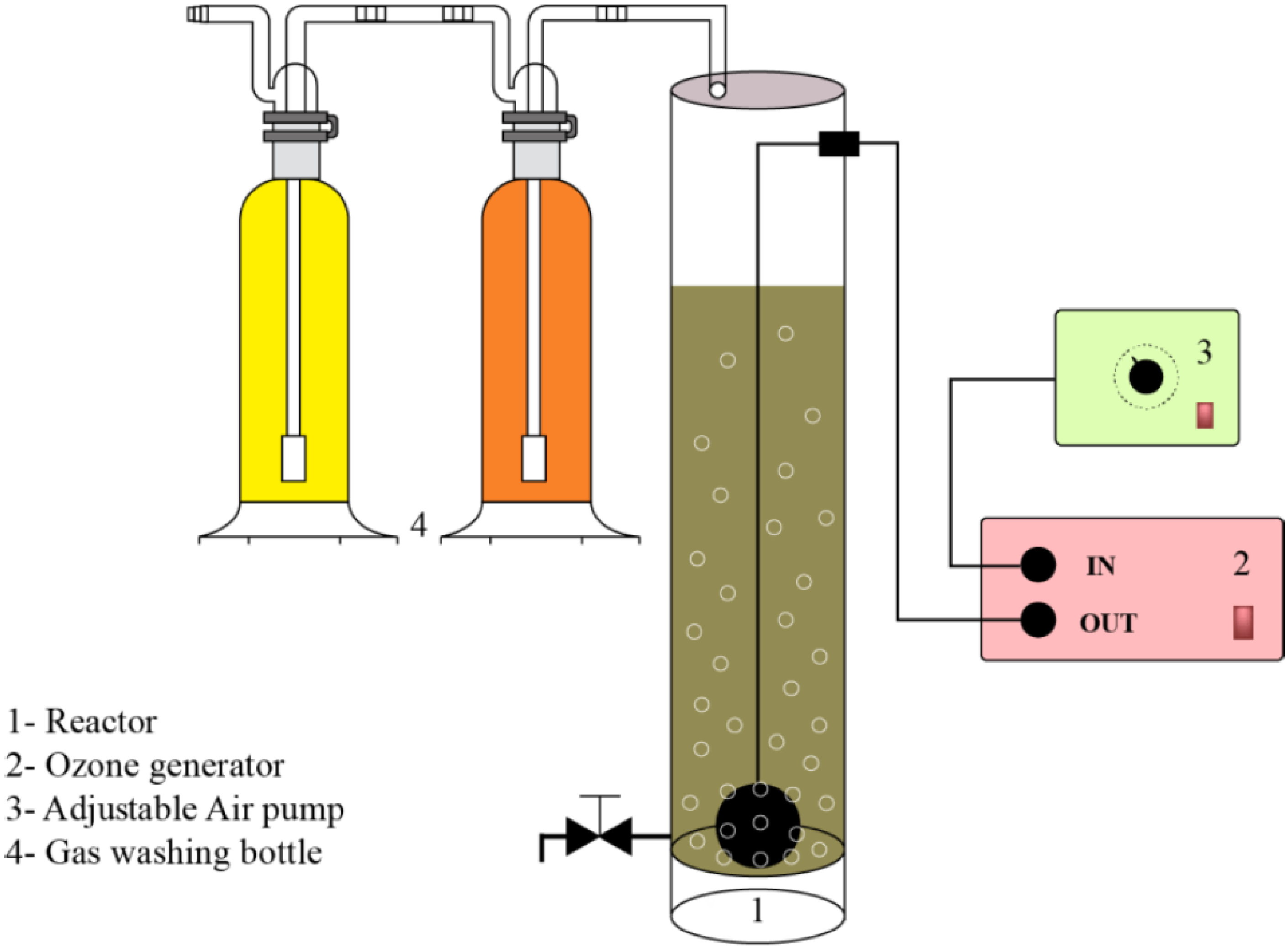

Figure 1

Schematics of the test setup

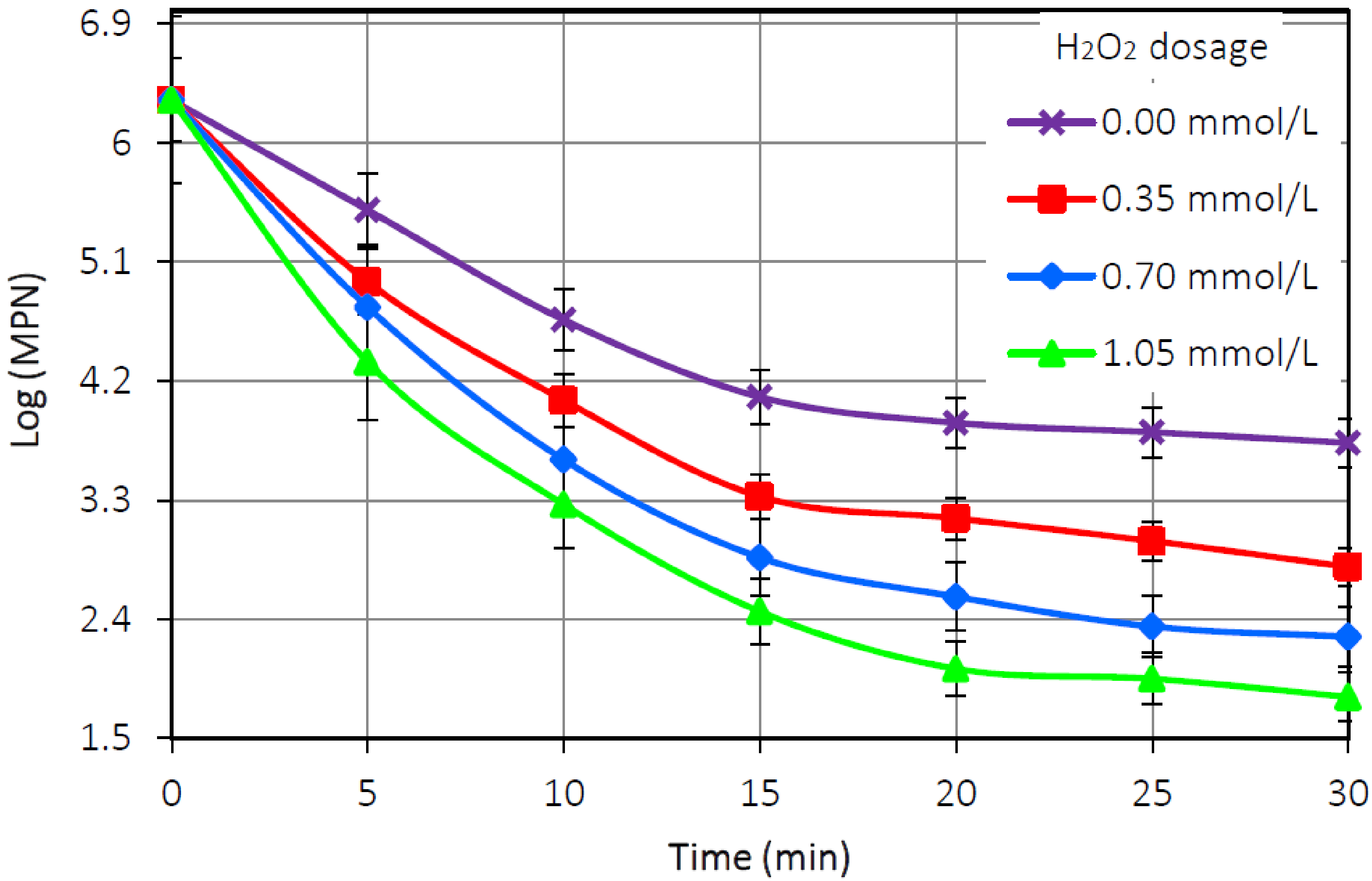

Figure 2

The effect of H2O2+O3 method on the removal of total coliform in constant conditions of pH = 6.9 ± 0.1 and ozone doze = 0.083 mmol/L.

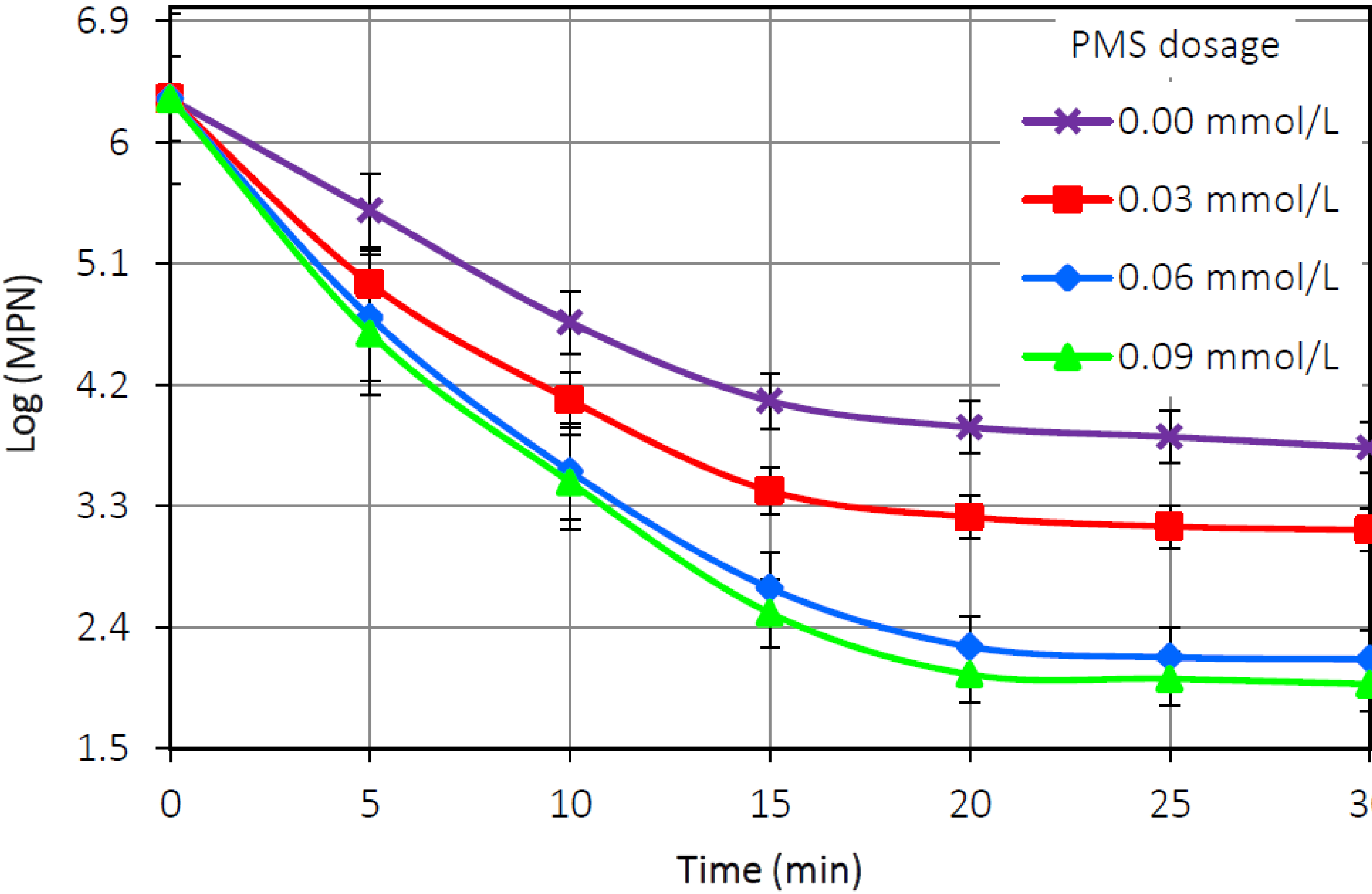

Figure 3

The effect of PMS+O3 process on the removal of total coliform in constant conditions of pH = 6.9 ± 0.1 and ozone doze = 0.083 mmol/L.

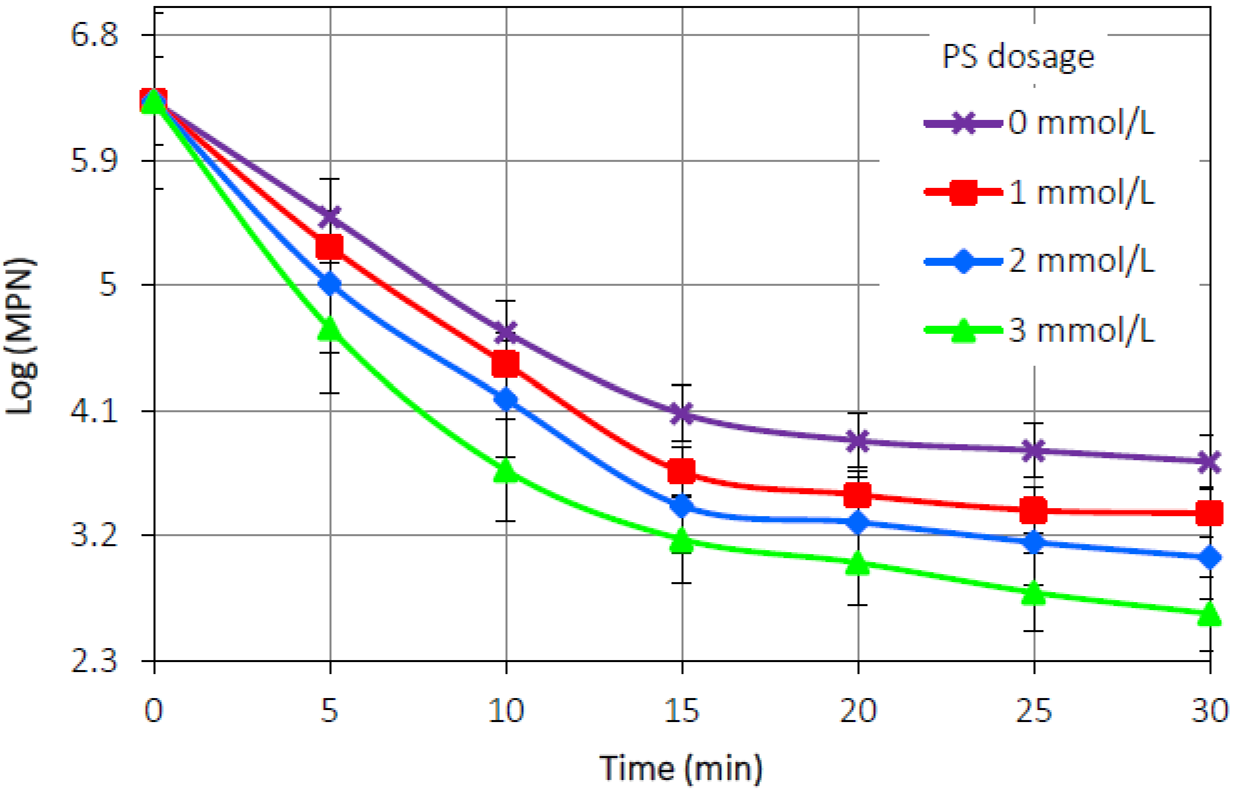

Figure 4

The effect of PS+O3 method on the removal of total coliform in constant conditions of pH = 6.9 ± 0.1 and ozone doze = 0.083 mmol/L.