Desalination and Water Treatment
www.deswater.com

doi: 10.5004/dwt.2017.20526

70 (2017) 150–156
April

Effects of mixed liquor ozonation in an activated sludge system for sewage treatment

S. Pesante, G. Morales, C. Baeza, G. Gómez, G. Vidal*

Engineering and Environmental Biotechnology Group, Faculty of Environmental Science and EULA-Chile Centre,
University of Concepción, P.O. Box 160-C, Concepción, Chile, Tel. +56 41 2204067; Fax: +56 41 2207076;
email: glvidal@udec.cl (G. Vidal), Tel. +56 41 2661033; emails: spesante@udec.cl (S. Pesante), gabmorales@udec.cl (G. Morales),
gloriagomez@udec.cl (G. Gómez), Tel. +56 41 2204068; email: anabaeza@udec.cl (C. Baeza)

Received 24 September 2016; Accepted 20 January 2017

ABSTRACT

Sludge generation is a drawback of the application of activated sludge (AS) technology in sewage treatment. The reduction of sludge production by the ozonation of an AS system's mixed liquor is an approach to reduce excess sludge in the process. The aim of this study is to evaluate the application of four ozone doses to mixed liquor fractions from a laboratory-scale AS sewage treatment system. The effect of ozonation in the mixed liquor supernatant was also evaluated. The doses ranged from 0.35 to 0.80 $gO_3/gVSS$. The results show that the applied ozone doses reduced the volatile suspended solids (VSS) concentrations by 25.5%–70.5%, which demonstrates the effectiveness of ozone in reducing the sludge concentration. In the supernatant of the ozonized mixed liquor, the chemical oxygen demand (COD) concentrations increased by 237%, 327%, 597% and 701% after the four doses. Moreover, the total nitrogen and total phosphorus concentrations in the supernatant increased by 9.4%–47% and 9.7%–8.2%, respectively, with respect to a mixed liquor without ozone application. The COD concentration in the effluent is affected by the ozonation. Thus, the COD removal efficiency of the AS system decreased by 6%–63%, whereas the nutrient removal efficiency was constant. In contrast, the specific oxygen uptake rate index was greatly inhibited (maximum inhibition of 31%) after the application of 0.50 $gO_3/gVSS$.

Keywords: Activated sludge; Effluent quality; Mixed liquor; Ozone; Sewage

1. Introduction

Activated sludge (AS) wastewater treatment systems produce large amounts of sludge. The mass ratios indicate sludge production of 30–45 g total suspended solids (TSS)/inhabitant·d [1], and the treatment and final disposal costs of this sludge can reach 25%–60% of the total operational cost of a wastewater treatment plant (WWTP) [2–4]. In response, technologies have been developed to reduce sludge production in the water line (influent treatment) or in the sludge line (post-sludge production). Mechanical, chemical, thermal and biological disintegration technologies can be applied in the water line [5–7], and ozonation is one of the most effective chemical technologies due to the oxidation potential of ozone (2.07 V) [7]. This technology can be coupled to the biological system, in which a fraction of the mixed liquor of the AS system is oxidized to solubilize the cellular content of the biomass [3,6,8]. Results of previous studies have demonstrated that ozonation decreases the generation of sludge by 17%–100%, and the applied doses have varied over a wide range [8,9]. Doses have been reported as functions of the total solids (TS) in the mixed liquor between 0.0008 and 0.08 gO_3/gTS [3,4,9], TSS between 0.05 and 0.27 gO_3/gTS [8,10,11] or a net ozonation rate of 0.012–0.13 $gO_3/gTS·h$ [7].

The effects of ozonation can be observed in several phases of the treatment operation. The sludge's TSS concentration has been observed to decrease by a maximum of 77% with 0.05 gO_3/gTS [4,12] and by 88% after the application of 0.13 $gO_3/gTSS·h$ [7]. Moreover, effects have been identified in the supernatant of the ozonated sludge, such as increase of

---

* Corresponding author.

the chemical oxygen demand (COD), total nitrogen (TN) and total phosphorus (TP) of 699%, 169%, and 2,379%, respectively, when 0.05 gO_3/gTS was applied for 105 min [3].

Yan et al. [10] determined that the COD removal efficiency remained constant at approximately 91% when 0.15 gO_3/gTSS was applied. Similarly, Gardoni et al. [9] determined that the efficiency remained between 84% and 82% when between 0.8 and 2.5 gO_3/kgTS of ozone was applied to sludge recirculation. Takdastan [13] observed a decrease from 92% to 42% after 0.025 gO_3/gTS was applied to the sludge recirculation of a sequencing batch reactor (SBR).

Ozonation may also alter the AS system activity, which is evaluated by the specific oxygen uptake rate (SOUR). The SOUR has been reported to decrease by 87% after the application of 0.02 gO_3/gTSS [13]. Moreover, the sludge's settling properties, which are measured by the sludge volume index (SVI), were enhanced by ozonation because it decreased from 120 to 75 mL/gTS [9]. Similarly, Takdastan [13] found that the addition of ozone was associated with decreased SVI; the SVI decreased by approximately 20 mL/gTS with 20 mgO_3/gTSS.

Based on these results, this study evaluates the effects of the application of ozone doses on the fraction of mixed liquor and the effluent characteristics during biological treatment.

2. Materials and methods

2.1. Raw sewage

Raw sewage (that was later used as the influent for an AS system) samples were obtained from a WWTP in southern Chile. The WWTP treats sewage from a community of 20,000 inhabitants. The samples were obtained after pretreatment that consisted of solids removal by thin grids, and they were stored in the dark at 4°C ± 1°C [14].

2.2. Inoculum

The AS system was inoculated with 2.5 gVSS/L of return activated sludge from the same WWTP. The volatile suspended solids (VSS) to TSS ratio (VSS/TSS) of the inoculum was 0.75.

2.3. Experimental setup

2.3.1. AS system

A continuous laboratory-scale AS system was operated. Fig. 1 shows the configuration of the AS system and the ozonation system. Fig. 1(a) shows a schematic of the experimental setup. The AS system consists of an aeration tank and a settler, which are both made of glass and have volumes of 0.81 and 0.45 L, respectively. The system was operated for 84 d, including 36 d to reach steady-state conditions (reference operation), and four ozone doses were applied from days 37 to 84.

The operational temperature was 14°C ± 2.8°C. The dissolved oxygen (DO) concentration was maintained above 2 mgO_2/L. The sludge was periodically recycled from the settler between the aerobic reactors to maintain approximately 2.5 gVSS/L. There was no excess sludge wastage during the operational period.

During the operational period, the hydraulic retention time (HRT) ranged between 9 and 10.3 h, and the organic loading rate (OLR) ranged between 0.5 and 0.9 kgCOD/m^3·d. Table 1 shows the operational parameters, where D0 corresponds to the reference operation (i.e., no ozone application).

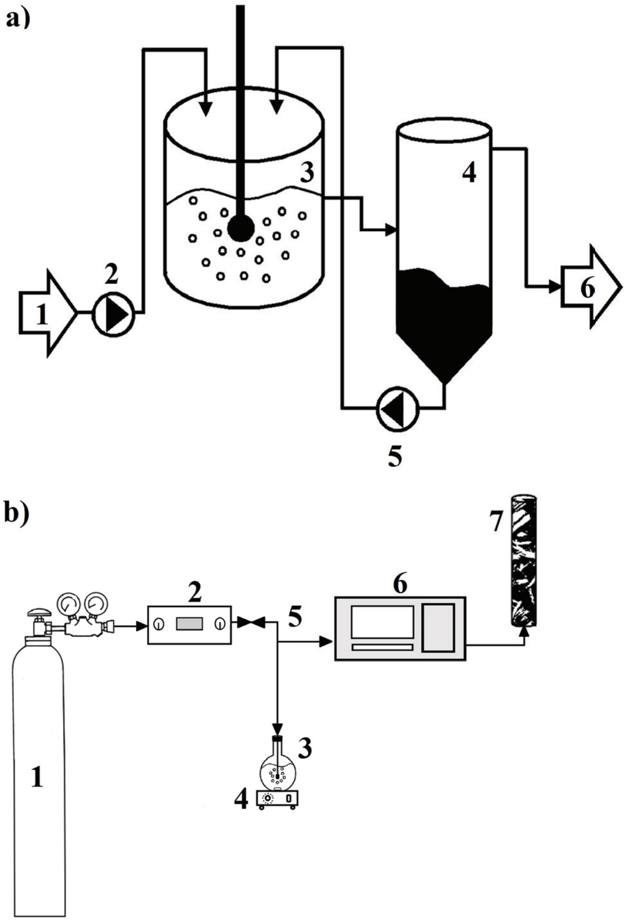

Fig. 1. Schematic diagram of the (a) experimental setup of the activated sludge system: (1) feed sewage influent, (2) influent pump, (3) aeration tank, (4) settler, (5) recirculation pump and (6) effluent; (b) experimental setup of the ozonation system: (1) oxygen cylinder, (2) ozone generator, (3) ozone contactor, (4) magnetic stirring, (5) off-gas line, (6) gas analyzer (spectrophotometer) and (7) ozone destructor (cellulose trap).

The monitored control parameters for the mixed liquor in the aeration tank were the temperature, pH, DO and electrical conductivity (EC), which were measured daily. TN and TP were estimated to evaluate the AS system's efficiency and the removal of COD.

The TSS and VSS concentrations were used to evaluate the mixed liquor, and the SOUR was evaluated by respirometry.

2.3.2. Ozonation

Between 16% and 30% of the mixed liquor in the aeration tank was ozonated in four trials from 15 to 45 min and subsequently returned to the aeration tank. The ozone doses were 0.35, 0.45, 0.50 and 0.80 gO_3/gVSS, which corresponded to D1, D2, D3 and D4, respectively. Table 1 shows the corresponding ozone doses and operational parameters for the sludge ozonation.

Table 1
Operational parameters of the activated sludge system during the reference operation and ozonation periods

Operational parameters	D0[a]	D1	D2	D3	D4
Operation day (d)	1–36	37	62	68	84
OLR (kgCOD/m^3 d)	0.9 ± 0.4	0.7 ± 0.1	0.8 ± 0.1	0.7 ± 0.1	0.5 ± 0.1
HRT (h)	9 ± 1.7	10.2 ± 2.0	10.3 ± 2.0	8.1 ±0.3	9 ± 0.5
pH	8.3 ± 0.3	7.7 ± 0.1	7.7 ± 0.1	7.1 ± 0.1	7.7 ± 0.1
MLVSS (g/L)	2.1 ± 0.5	1.8 ± 0.1	1.9 ± 0.2	2.5 ± 0.2	2.1 ± 0.3
Ozonation parameters					
Ozone concentration (mgO$_3$/L)	0	20.9	22.4	22.4	23.3
Ozonation time (min)	0	15	30	45	30
Ozonated fraction (%)	0	16	25	30	25
Doses (gO$_3$/gVSS)	0	0.35	0.45	0.50	0.80

OLR: organic loading rate, HRT: hydraulic retention time and MLVSS: mixed liquor volatile suspended solids.
[a] D0 corresponds to the reference operation and D1–D4 corresponds to ozone doses.

Fig. 1(b) describes the ozonation system. Ozone was generated by corona discharge in an OZOCAV ozone generator with a maximum capacity of 0.5 gO$_3$/h using pure oxygen as the feed gas. Ozone was bubbled through a porous diffuser in a glass ozone contactor with an effective volume of 1 L and mixed with a magnetic stirrer. The ozone concentrations at the inlet and outlet of the ozone contactor were measured with a UV spectrophotometer (Spectronic Genesys 5). The applied dose of ozone was determined as a function of the ozonation effect on the VSS of the sludge following He et al. [15].

After ozonation, the TSS and VSS concentrations of the mixed liquor were measured to evaluate the solid reduction promoted by ozone. A sample of the ozonated mixed liquor was centrifuged by a Megafuge Heraeus Model 1 at 2,000 rpm for 5 min to separate the particulates from the soluble fraction (supernatant) and evaluate the contributions of COD, TN and TP in the mixed liquor supernatant. The effect of the mixed liquor on the solubilization is represented by the disintegration degree (DD$_{COD}$) as defined by Zhang et al. [3] according to Eq. (1):

$$DD_{COD} = \frac{COD_{ozone} - COD_0}{COD_T - COD_0} \quad (1)$$

where DD$_{COD}$ is the disintegration degree, COD$_{ozone}$ is the supernatant COD of the ozonated mixed liquor (mg/L), COD$_0$ is the supernatant COD of the untreated mixed liquor (mg/L) and COD$_T$ is the total COD of the untreated mixed liquor.

The COD removal kinetics after ozonation was also evaluated. The ratio between COD removal and time was calculated to identify the decline and recovery periods with respect to COD removal.

2.4. Analytical methods

The COD, NH$_4^+$–N, TSS and VSS analyses were performed according to the Standard Methods [16]. TN was measured by the Koroleff method using Merck kit number 1.14763.0001, and TP was determined by the phosphorus–molybdenum blue method using Merck kit number 1.14729.0001. Both analyses were performed using the same Merck Spectroquant NOVA 60 photometer.

The samples of COD, NH$_4^+$–N, TN and TP were filtered through Whatman membranes with a pore size of 0.45 μm. The mixed liquor SOUR was determined using a biological oxygen monitor (YSI 5300 system). The system was operated with an air-tight respiration vessel fitted with a DO probe YSI 5231; the vessel was continuously stirred and thermally controlled, and the biomass was washed twice previously with a phosphate buffer according to Mosquera et al. [17] modified at 25°C. Measurements were taken 1 hour after ozonation.

The pH, temperature and EC were measured by an OAKTON multiparameter portable meter (PC650–480485), and DO was measured with an Oxical Oxi330 WTW oxygen meter.

3. Results and discussion

3.1. Influent sewage characterization

Table 2 shows the characterization of the influent sewage. The pH remained near neutral in the range of 7.1–8.8. Organic

Table 2
Physicochemical characterization of the influent sewage

Parameter	Average value	Range	*n*
COD (mg/L)	310.3 ± 95	206.5–504.7	12
TSS (g/L)	0.7 ± 0.3	0.4–1.2	8
VSS (g/L)	0.6 ± 0.3	0.3–1.1	8
TN (mg/L)	111 ± 13	95–139	10
TP (mg/L)	15.4 ± 1.4	12.8–16.7	10
NH$_4^+$–N (mg/L)	85 ± 3.3	82.6–87.2	5
pH	7.7 ± 0.7	6.5–8.9	24
Temperature (°C)	13.7 ± 2.6	9.7–17.7	24
EC (μS/cm)	1.6 ± 0.2	1.1–1.9	24

COD: chemical oxygen demand, TSS: total suspended solids, VSS: volatile suspended solids, TN: total nitrogen, TP: total phosphorus, NH$_4^+$–N: ammonium, EC: electrical conductivity and *n*: number of samples.

matter, which was measured as COD, had a mean value of 310.3 ± 95 mg/L. Similar COD values of 279.5–398.1 mg/L were obtained by Vera et al. [14] on the primary influents of the same WWTP.

VSS represented 80%–90% of the TSS, the TP concentrations ranged from 12.8 to 16.7 mg/L (variation <10%), and NH_4^+–N corresponded to 77% of the TN.

3.2. Effects of ozone on the reduction of solids concentration

Fig. 2 shows the solid concentrations in the mixed liquor in the AS system. Fig. 2(a) shows the solid concentrations of the mixed liquor before and after the application of ozone. Before ozonation, the TSS and VSS concentrations ranged from 2.0 to 2.9 gTSS/L and 1.8 to 2.4 gVSS/L, respectively. Demir and Filibeli [4] reported similar values of 2.1 ± 0.35 gTSS/L and 1.8 ± 0.04 gVSS/L, respectively. After ozone application, the VSS concentrations decreased by 25.5%–70.5% (Fig. 2(b)). The maximum reduction of the VSS concentration occurred with the highest dose (0.8 gO_3/gVSS). A similar study reported that the TS and volatile solids of an SBR sludge decreased by 49.1% and 45.7%, respectively [3]. Similarly, Demir and Filibeli [4] determined that the TSS and VSS of mixed liquor decreased by 77.8% and 71.6%, respectively, whereas Richardson et al. [7] found a maximum reduction of 88% of the TSS of waste secondary sludge.

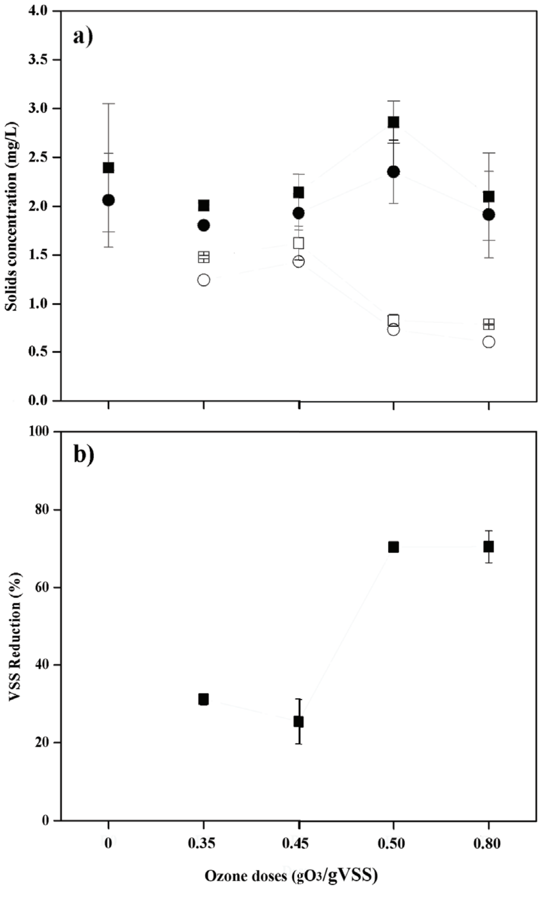

Fig. 2. (a) Evolutions of the TSS (■) and VSS (●) mixed liquor concentrations before ozonation and the TSS (□) and VSS (○) mixed liquor concentrations after ozonation; (b) VSS reduction efficiency (■) after ozonation.

3.3. Effects on the mixed liquor supernatant

Fig. 3 shows the COD, TN and TP concentrations of the mixed liquor supernatant before and after ozonation. Fig. 3(a) shows that the COD concentration increased by 237%, 327%, 597% and 701% after D1, D2, D3 and D4, respectively. A direct relationship is observed between the increases in the ozone dose with the COD concentrations after ozonation. Demir and Filibeli [4] and Isazadeh et al. [18] attributed this behavior to the increased amount of intracellular organic substances released as a result of ozonation. Figs. 3(b) and (c) show the concentrations and the increases of the TN and TP in the ozonized mixed liquor supernatant, respectively. The TN increased by 9.4%–47%, and the TP increased by 9.7%–68.2%. Similarly, Demir and Filibeli [4] found that the TN and TP concentrations increased by 2,057% and 375%, respectively. The greatest contributors to the nutrient concentration in the supernatant were nitrogen and organic phosphorus [6].

The increase in the COD concentration is also confirmed by the COD/TN ratio of the ozonated mixed liquor supernatant. The ratio in the mixed liquor remained in the range of 1–2 before being ozonized, whereas it ranged between 3.7 and 5.7 after ozonation. In addition, the COD/TP ratio increased

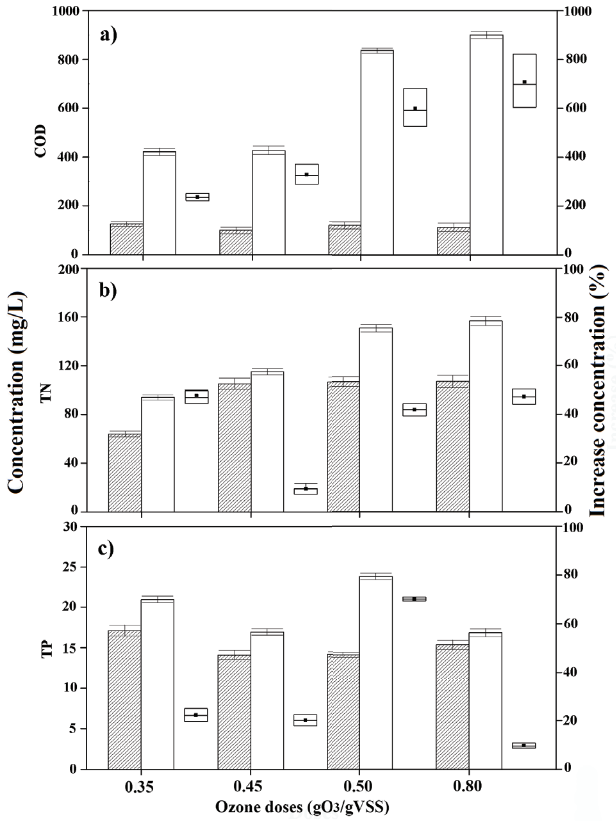

Fig. 3. (a) COD, (b) TN and (c) TP concentrations in the mixed liquor supernatant before (▨) and after ozonation (□, bars) and percentage increases in concentration (□, box-plots).

from 7–8 to 20–53. These results indicate that the COD concentration is the most variable parameter. The COD/TN ratio that was obtained in this study is similar to that obtained by Zhang et al. [3], which had an initial value of 4.6 and increased to 11.5. In the same study, the initial COD/TP ratio was 75, which was10 times higher than the value obtained by this study. In this sense, the TP in the supernatant increased by 2,379% after ozonation, which reduced the COD/TP ratio from 75 to 22 [3]. This may be attributed to different sludge compositions.

The solubilization of the mixed liquor after being ozonized was evaluated by the degree of disintegration (DD_{COD}). The DD_{COD} values obtained in this study were 22%, 17%, 42% and 40% for D1, D2, D3 and D4, respectively. The highest DD_{COD} values were obtained for the highest doses of 0.50 and 0.80 gO_3/gVSS. However, DD_{COD} decreased after D3, which can be explained by the increased bubbling during ozonation with increasing ozone dose, which decreased the ozone mass transfer [19]. Zhang et al. [3] observed similar behavior when sludge was ozonized with lower ozone doses than in this study. Their results showed that when 0.025 and 0.05 gO_3/gTS were applied, the DD_{COD} values were 10.3% and 40.3%, respectively. When the dose increased to 0.08 gO_3/gTS, DD_{COD} remained at 40%. Thus, nonlinear behavior is observed between the increase in DD_{COD} and the applied dose, as was confirmed by recent studies [4,12].

3.4. System removal efficiencies

Fig. 4 shows the COD removal efficiency during the reference operation (D0) and the applications of D1–D4. During D0, the average COD effluent concentration was 96 mg/L. The average COD removal efficiency was 68.2% at an OLR of 0.9 ± 0.4 kgCOD/m^3·d and an HRT of 9 ± 1.7 h and reached a maximum of 82%. These results are similar to those obtained by Colmenarejo et al. [20], who achieved a COD removal efficiency of 80.3% ± 7.3% and an HRT of 6–8 h.

The removal efficiency decreased by 10%, 15%, 6% and 63% after applications D1, D2, D3 and D4, respectively, and the COD concentrations in the effluent reached 110–131 mg/L. This is because, in these cases, the different ozone doses application is just for the cell hydrolysis, where COD is not removed, but carbon changes the oxidation state, depending on the cases.

This result is attributed to the release of dissolved, colloidal and inert COD after ozonation, which negatively impacts the effluent characteristics [8,18,21]. Demir and Filibeli [12] also observed a decrease of approximately 7% in COD removal efficiency, which is similar to the results of this study for D1, D2 and D3. Gardoni et al. [9] indicated that the removal efficiency decreased by 2%, and Takdastan [13] determined that the COD removal efficiency decreased by 54%. This behavior is consistent with the values that were observed after the application of the highest dose (0.80 gO_3/gVSS), where the removal efficiency decreased by 63%. This reduction in removal efficiency was produced by the retention of organic material released by D4 ozonation, which contributed to the effluent COD concentration.

Fig. 5 shows the COD removal, in which two periods are identified; the first 4 h correspond to a period of decline, which is followed by a recovery period. During the decline period, the maximum decrease (25%/h) occurred in the second hour after D2 and is possibly attributed to the lower VSS mixed liquor concentration (1.9 ± 0.2 g/L) than in D3 and D4 (2.5 ± 0.2 and 2.1 ± 0.3 g/L, respectively). In the case of D4, the rate of decrease in the third hour (24%/h) was similar to that of D2, but it occurred 1 h later.

The maximum recovery rate was observed for D2 from 4 to 6 h (27%/h). The removal efficiency recovered 20 h after the application of the ozone doses.

Fig. 6 shows the TN and TP removal efficiencies, which remained constant at 15% ± 9% and 4% ± 2%, respectively, during the entire period of operation.

Yan et al. [10] obtained similar results; the TN concentration in the effluent of an AS system with ozonated excess sludge was 19.8 mg/L, while the concentration in a control was 20.2 mg/L, which indicates that there were no significant negative effects. The authors observed an increase in TP of 30% in the effluent concentration with respect to the control. However, those results were obtained after 122 days of ozonation. Furthermore, Gardoni et al. [9] found that phosphorus

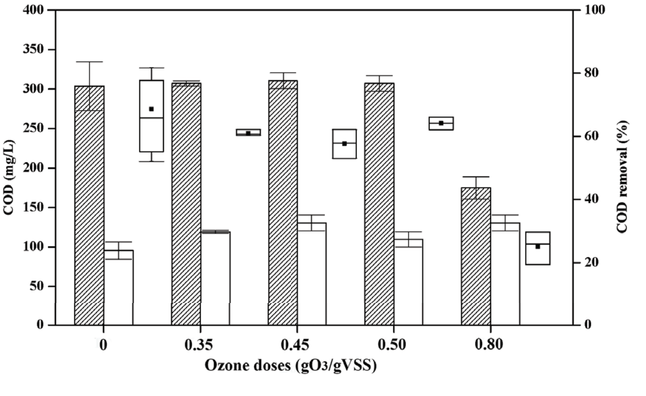

Fig. 4. Evolutions of the COD influent concentration (▨, bars), COD effluent concentration (□, bars) and COD removal (□, box-plots) during the entire operation of the activated sludge system.

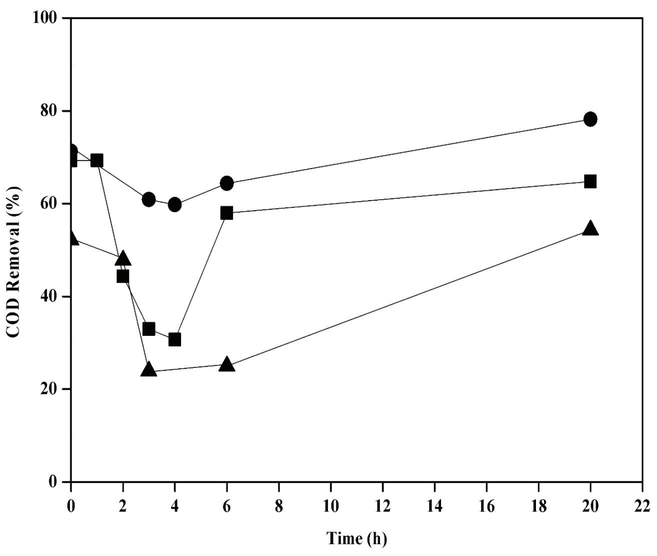

Fig. 5. COD removal after ozonation with D2 (●), D3 (■) and D4 (▲).

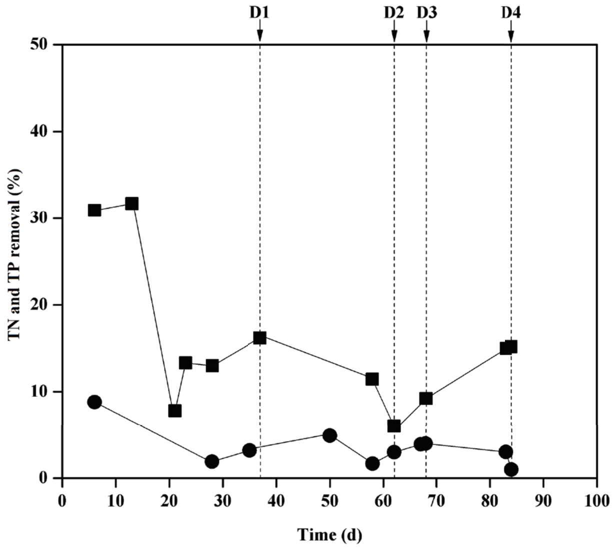

Fig. 6. Evolutions of TN (●) and TP (▲) removal during the entire operation of the activated sludge system.

removal decreased from 70% to 28% due to the reduction in biomass production, which is the main mechanism of phosphorus removal.

The increases in the TN and TP concentrations in the mixed liquor supernatant after ozonation (Figs. 3(a) and (c)) did not impact the nutrient removal efficiency, which indicates that the system was able to resist the load.

Decrease in pH of 0.57–1.23 was observed after application of the ozone doses (ozone doses: 0.35, 0.45, 0.50 and 0.80 $gO_3/gVSS$). Chu et al. [22] and Zhang et al. [3] found similar values in the range of 0.5–0.7 after applying doses of 0.025–0.050 mgO_3/gTS, and they were principally attributed to cell lysis. Zhang et al. [3] suggested that the decrease in pH is caused by lysis and the release of intracellular substances that contain deoxyribonucleic acids or possibly by the oxidization of some organics into low-molecular-weight acids by ozone.

3.5. Effects on mixed liquor SOUR

Fig. 7 shows the evolution of the SOUR in relation to the OLR for the AS system during the 84 d of operation. During the control operation, the SOUR was 0.45 ± 0.01 $gO_2/gVSS \cdot d$ and decreased by 29%, 31% and 13% with D2, D3 and D4, respectively (the SOUR results after D1 are not presented). The minimum observed value was 0.31 ± 0.02 $gO_2/gVSS \cdot d$ after D3. The reduction of the SOUR by applying increasing ozone doses has been attributed to the death of some of the microorganisms in the mixed liquor [13,23]. Microorganism death also contributes to the organic content of the medium, which increases the OLR of the AS system to 1.7–2.9 $kgCOD/m^3 \cdot d$.

Takdastan [13] found that the SOUR decreased from 0.43 to 0.07 $gO_2/gVSS \cdot d$ (83% decrease) after applying incremental doses of 0–0.025 $gO_3/gTSS$. Likewise, Torregrossa et al. [24] indicated that the SOUR was almost 40% lower after the application of 0.015 $gO_3/gTSS$ to the sludge recirculation. The variability of the results shows that the behavior of SOUR depends on complex reactions of ozone with the mixed liquor because it is a mixture of mineral and organic material as well as various microorganisms [25], and the level of the toxic compounds generates ammonia and other substances [26]. However, adaptation is a key factor to increasing the removal efficiency [27,28].

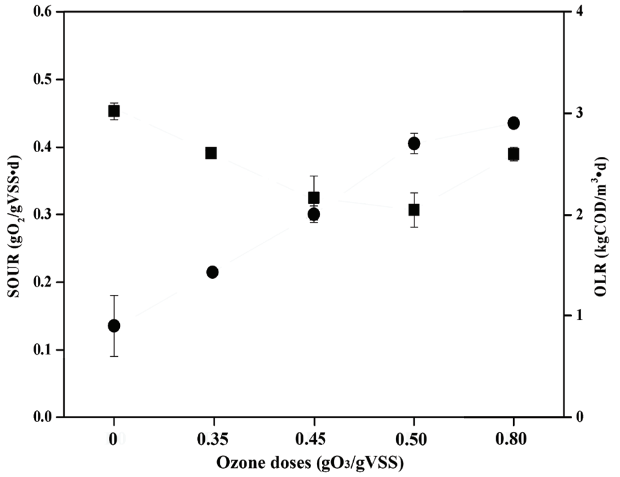

Fig. 7. SOUR of the mixed liquor (■) vs. OLR in the activated sludge system (●) after ozonation.

In contrast, the SOUR increased from 0.31 ± 0.03 $gO_2/gVSS \cdot d$ after D3 to 0.39 ± 0.01 $gO_2/gVSS \cdot d$ after D4. This result can be attributed to a possible adaptation of microorganisms to the imposed conditions. In this case, the COD concentration of the supernatant indicates that the OLR increased in D4 to 2.9 $kgCOD/m^3 \cdot d$, which could be sufficient to enhance the use of oxygen by microorganisms. Isazadeh et al. [18] determined that the SOUR increased when the ozone application was equivalent or less than 0.006 $gO_3/gTSS$, which indicates that floc disruption allowed better substrate uptake by the biomass. Finally, the observed trend in the SOUR after D0–D3 is directly related to the decrease in COD removal efficiency from 68.2% (in the reference operation) to 58% and 64% after D2 and D3, respectively.

4. Conclusions

The application of ozone to mixed liquor during biological treatment decreased the VSS concentrations by 25.5%–70.5% and had a maximum reduction with 0.8 $gO_3/gVSS$.

The COD, TN and TP concentrations in the supernatant of the ozonized mixed liquor increased depending on the ozone doses applied, regarding the mixed liquor without ozone application.

The SOUR was 0.45 ± 0.01 $gO_2/gVSS \cdot d$ during the reference operation, and after the application of D3 the SOUR values was 0.31 ± 0.02 $gO_2/gVSS \cdot d$.

Acknowledgment

This work was supported by INNOVA BIOBIO Grant No. 13.211-EM.TES, CONICYT/FONDAP/15130015.

References

[1] I. Vera, K. Sáez, G. Vidal, Performance of 14 full-scale sewage treatment plants: comparison between four aerobic technologies regarding effluent quality, sludge production and energy consumption, Environ. Technol., 34 (2013) 2267–2275.

[2] J. Lee, H. Cha, K. Park, K. Song, K. Ahn, Operational strategies for an activated sludge process in conjunction with ozone oxidation for zero excess sludge production during winter season, Water Res., 39 (2005) 1199–1204.

[3] G. Zhang, J. Yang, H. Liu, J. Zhang, Sludge ozonation: disintegration, supernatant changes and mechanisms, Bioresour. Technol., 100 (2009) 1505–1509.

[4] O. Demir, A. Filibeli, Fate of return activated sludge after ozonation: an optimization study for sludge disintegration, Environ. Technol., 33 (2012) 1869–1878.

[5] P. Neumann, S. Pesante, M. Venegas, G. Vidal, Developments in pre-treatment methods to improve anaerobic digestion of sewage sludge, Rev. Environ. Sci. Biotechnol., 15 (2016) 173–211.

[6] L. Chu, S. Yan, X. Xing, X. Sun, B. Jurcik, Progress and perspectives of sludge ozonation as a powerful pretreatment method for minimization of excess sludge production, Water Res., 43 (2009) 1811–1822.

[7] E. Richardson, A. Hanson, J. Hernandez, Ozonation of continuous-flow activated sludge for reduction of waste solids, Ozone Sci. Eng., 31 (2009) 247–256.

[8] H. Yasui, M. Shibata, An innovative approach to reduce excess sludge production in the activated sludge process, Water Sci. Technol., 30 (1994) 11–20.

[9] D. Gardoni, E. Ficara, R. Fornarelli, M. Parolini, R. Canziani, Long-term effects of the ozonation of the sludge recycling stream on excess sludge reduction and biomass activity at full-scale, Water Sci. Technol., 63 (2011) 2032–2038.

[10] S. Yan, H. Zheng, A. Li, X. Zhang, X. Xing, L. Chu, B. Jurcik, Systematic analysis of biochemical performance and the microbial community of an activated sludge process using ozone-treated sludge for sludge reduction, Bioresour. Technol., 100 (2009) 5002–5009.

[11] S. Yan, L. Chu, X. Xing, A. Yu, X. Sun, B. Jurcik, Analysis of the mechanism of sludge ozonation by a combination of biological and chemical approaches, Water Res., 43 (2009) 195–203.

[12] O. Demir, A. Filibeli, Effects of partial ozonation on activated sludge process for the minimization of excess sludge production during biological treatment, Desal. Wat. Treat., 52 (2013) 3063–3075.

[13] A. Takdastan, Investigation of the excess sludge reduction in SBR by oxidizing some sludge by ozone, Iran J. Chem. Chem. Eng., 28 (2009) 95–104.

[14] I. Vera, F. Araya, E. Andrés, K. Sáez, G. Vidal, Enhanced phosphorus removal from sewage in mesocosm-scale constructed wetland using zeolite as medium and artificial aeration, Environ. Technol., 35 (2014) 1639–1649.

[15] S. He, G. Xue, B. Wang, Activated sludge ozonation to reduce sludge production in membrane bioreactor (MBR), J. Hazard. Mater., 135 (2006) 406–411.

[16] APHA, Standard Methods for the Examination of Water and Wastewater, 20th ed., American Public Health Association, Washington, D.C., USA, 1998.

[17] A. Mosquera, F. González, J. Campos, R. Méndez, Partial nitrification in a SHARON reactor in the presence of salts and organic carbon compounds, Process Biochem., 40 (2005) 3109–3118.

[18] S. Isazadeh, M. Feng, L. Urbina Rivas, D. Frigon, New mechanistically based model for predicting reduction of biosolids waste by ozonation of return activated sludge, J. Hazard. Mater., 270 (2014) 160–168.

[19] S. Yang, W. Guo, G. Cao, H. Zheng, N. Ren, Simultaneous waste activated sludge disintegration and biological hydrogen production using an ozone/ultrasound pretreatment, Bioresour. Technol., 124 (2012) 347–354.

[20] M.F. Colmenarejo, A. Rubio, E. Sánchez, J. Vicente, M.G. García, R. Borja, Evaluation of municipal wastewater treatment plants with different technologies at Las Rozas, Madrid (Spain), J. Environ. Manage., 81 (2006) 399–404.

[21] L. Chu, J. Wang, X. Xing, X.L. Sun, B. Jurcik, Evaluation of biodegradation of soluble and particulate matter produced during sewage sludge ozonation by a combination of chemical and microbial approaches, J. Environ. Sci. Health, Part A, 45 (2010) 1315–1321.

[22] L. Chu, J. Wang, B. Wang, X. Xing, S. Yan, X. Sun, B. Jurcik, Changes in biomass activity and characteristics of activated sludge exposed to low ozone dose, Chemosphere, 77 (2009) 269–272.

[23] J.S. Albuquerque, J.C. Domingos, G.L. Sant'Anna Jr., M. Dezotti, Application of ozonation to reduce biological sludge production in an industrial wastewater treatment plant, Water Sci. Technol., 58 (2008) 1971–1976.

[24] M. Torregrossa, G. Di Bella, D. Di Trapani, Comparison between ozonation and the OSA process: analysis of excess sludge reduction and biomass activity in two different pilot plants, Water Sci. Technol., 66 (2012) 185–192.

[25] M.A. Dziurla, M. Salhi, P. Leroy, E. Paul, P. Ginestet, J.C. Block, Variations of respiratory activity and glutathione in activated sludge exposed to low ozone doses, Water Res., 39 (2005) 2591–2598.

[26] G. Morales, S. Pesante, G. Vidal, Effects of black liquor shocks on activated sludge treatment of bleached kraft pulp mill wastewater, J. Environ. Sci. Health, Part A, 50 (2015) 627–633.

[27] M. Belmonte, C. Xavier, J. Decap, M. Martínez, R. Sierra, G. Vidal, Improvement of the abietic acid biodegradation contained in ECF effluent due to biomass adaptation, J. Hazard. Mater., 135 (2006) 256–263.

[28] J. Correa, V. Domínguez, M. Martínez, G. Vidal, Interaction between 2,4,6-TCP increasing concentration content in ECF bleached effluent and aerobic bacteria degradative activity, Environ. Int., 29 (2003) 459–465.