

Chapter 2

Nutrient removal performance of the anaerobic/oxic/anoxic process combined with sludge ozonation and phosphorus adsorption

SUMMARY

A continuous A/O/A (anaerobic/oxic/anoxic) process with sludge ozonation and phosphorus adsorption was operated under various ozonation conditions to meet increasingly stringent requirements for environmental protection and phosphorus resource recovery. In the process operation, adequate sludge reduction was achieved when the sludge amount for ozonation was set at 16 % of total MLSS per day. However, nitrogen removal efficiency was deteriorated because the population density of nitrifying bacteria decreased. The decrease in the sludge amount for ozonation (9.4% of total MLSS per day) resulted in efficient nitrogen removal while MLSS concentration was gradually increased. No excess sludge was withdrawn during at least 2 months operation under this ozonation condition. Phosphorus was removed not only by normal PAOs (polyphosphate-accumulating organisms) but also by DNPAOs (denitrifying PAOs). On the other hand, not only DNPAOs but also endogenous denitrifying bacteria contributed to nitrogen removal. The accumulated phosphorus in sludge was solubilized by ozonation and a large part of the solubilized phosphorus consisted of PO_4 -P. Over 90% of PO_4 -P was adsorbed by zirconium-ferrite adsorbent in the phosphorus adsorption column.

2.1 INTRODUCTION

Activated sludge processes such as the University of Cape Town (UCT) process and processes employing an anaerobic-anoxic-oxic (A/A/O) system have been widely used to effectively remove nutrients from municipal wastewater. In wastewater treatment plants (WWTPs), treatment and disposal of excess sludge have been serious problems, and the treatment of excess sludge may account for 25% to 65% of total plant operating costs (Liu, 2003; Zhao and Kugel, 1997). Furthermore, the regulations regarding disposal have become increasingly strict in most countries as the amount of excess sludge increased with the expansion of populations and industries (Liu, 2003; Ødegaard, 2004).

Many efforts have been made to reduce excess sludge production, and this has been reviewed by Liu (2003) and Ødegaard (2004). One method is the solubilization of excess sludge by using ozone as a strong oxidant, and recirculation of the solubilized excess sludge, containing readily biodegradable carbon, into a biological treatment system (Ahn *et al.*, 2002c; Cesbron *et al.*, 2003; Cui and Jahng, 2004; Déléris *et al.*, 2001, 2003,2004; Kamiya and Hirotsuji, 1998; Nishimura *et al.*, 1999; Sakai *et al.*, 1997; Saktaywin *et al.*, 2005; Salhi *et al.*, 2003, 2004; Yasui *et al.*, 1996; Yasui and Shibata, 1994).

On the other hand, it is also effective for the reduction of excess sludge to control the sludge production potential in biological processes. In enhanced biological phosphorus removal (EBPR), normally polyphosphate-accumulating organisms (PAOs) release intercellular polyphosphate as orthophosphate with the assimilation of short-chain fatty acids under an aerobic condition, and then a large amount of phosphate is accumulated by PAOs under the subsequent oxic condition. Unlike PAOs, less sludge production was confirmed by using denitrifying polyphosphate-accumulating organisms (DNPAOs), which are capable of utilizing nitrate/nitrite as electron acceptors, to simultaneously remove phosphorus and nitrogen from wastewater (Ahn *et al.*, 2002a, 2002b; Kishida *et al.*, 2006; Kuba *et al.*, 1996, 1997; Soejima *et al.*, 2006; Tsuneda

et al., 2006). Employing DNPAOs has another advantage in limiting the energy source for denitrification. Previously, Tsuneda *et al.* (2006) described an anaerobic/oxic/anoxic (A/O/A) process and succeeded in causing DNPAOs to take an active part in simultaneous nitrogen and phosphorus removal in an acetate-fed sequencing batch reactor (SBR) while the additional acetate was required for the inhibition of phosphorus uptake under an oxic period. Thus, the A/O/A process requires an additional organic carbon loading line to the oxic tank; however, it has still an advantage over a UCT process, which also attains phosphorus removal by DNPAOs but requires two recirculation lines, an oxic tank to an anoxic tank and an anoxic tank to an aerobic tank.

We previously proposed an advanced system, the continuous A/O/A process combined with sludge reduction process by ozonation and phosphorus adsorption process by a zirconium-ferrite ($ZrFe_2(OH)_8$) adsorbent (Suzuki *et al.*, 2006). In this system, excess sludge is ozonated and the supernatant of the ozonated sludge passes through the phosphorus adsorption column. Then, both the supernatant after phosphorus adsorption and the residual solid after ozonation were transferred to the oxic tank to inhibit the oxic phosphorus accumulation by PAOs. During the bench-scale reactor operation receiving raw municipal wastewater, this process achieved effective nitrogen and phosphorus removal without sludge production while the effluent TOC concentration was deteriorated by the slowly biodegradable carbon derived from the solubilized sludge. The inhibition of oxic phosphorus uptake was also failed, and thus most phosphorus was accumulated by PAOs in the oxic tank.

In this study, to achieve the efficient nutrients removal and to induce DNPAOs to take a large part in phosphorus accumulation, the previous bench-scale continuous A/O/A process combined with ozonation and phosphorus adsorption was improved by changing the ozonation system and the recirculation lines of the ozonated sludge. During 152 days operation, operational conditions were optimized by changing the applied ozone concentration, the applied ozone dosage, and the return ratio of the supernatant of the physicochemical process (ozonation and phosphorus adsorption) to the anaerobic and oxic tanks.

2.2 MATERIALS AND METHODS

2.2.1 Long-term process operation

A bench-scale continuous A/O/A process with working volume of 36 L (an anaerobic tank, 10.3 L; an oxic tank, 10.3 L; an anoxic tank, 15.4 L) was operated for 152 days. The A/O/A process employed both the ozonation system and the phosphorus adsorption column (Fig. 1). The ozonation system was a cylindrical reactor with an internal diameter of 0.2 m and a height of 0.8 m (effective volume of 20 L). The ozonation system received excess sludge containing high phosphorus (about 5% mg-P/mg-MLSS) continuously collected from the end of an anoxic tank by a pump. When the amount of excess sludge reached approximately 12 L, sludge ozonation was conducted under specified ozonation conditions as shown in Table 1. Ozone gas was generated by an ozone generator (PO-10; Fuji Electric, Japan) and the applied ozone dose was monitored by a UV Ozone Monitor (PG-620HA; Ebara Jitsugyo, Japan). Then, ozone gas and excess sludge from the anoxic tank were mixed in turbulent flow by a current pump under high pressure (Nikuni swirling current pump M15NPD02S; Nikuni Co., Japan), and then the mixture was pumped out to the reactor. After both the ozonation and subsequent settling for one hour, most of the residual particulate materials settled down to the bottom of the tank and they were transferred to the anaerobic tank. The supernatant was flowed into the phosphorus adsorption column. The phosphorus adsorption column (internal diameter 75 mm; height 0.7 m; effective volume 2 L) was filled with 1.5 L of spherical zirconium-ferrite ($ZrFe_2(OH)_8$) adsorbent with an effective diameter of 0.7 mm (Japan Enviro Chemicals, Japan). Flow rate of the supernatant was set at 25 mL/min. After phosphorus adsorption, the adsorbent was backwashed with 15 L of tap water to remove residual SS. Both the solution after phosphorus adsorption (about 10 L) and the tap water containing the removed residual SS (about 15 L) were recirculated to the anaerobic tank and the oxic tank at an appropriate rate for around 17 h (Table 1).

The hydraulic retention time (HRT) was adjusted to 10 h. When the solution after the

ozonation and phosphorus adsorption was recirculated to the anaerobic and oxic tanks, HRT was decreased. Sludge return ratio from the settling tank to the anaerobic tank was controlled at 80%. The reactor was inoculated with activated sludge (initial MLSS was adjusted to 4,000 mg/L), which was collected from a WWTP performing efficient biological phosphorus removal (A/A/O process). Raw rural wastewater was collected from a rural sewage treatment plant (Ibaraki Prefecture, Japan) daily and flowed into the system. The characteristics of the rural wastewater are listed in Table 2.

In this study, 4 different operational conditions were conducted to investigate the appropriate operational conditions (Table 1). In each phase, nutrient removal efficiency was evaluated. Furthermore, to confirm the contribution of DNPAOs to phosphorus removal, phosphorus uptake rate (PUR) was estimated in Phase 4.

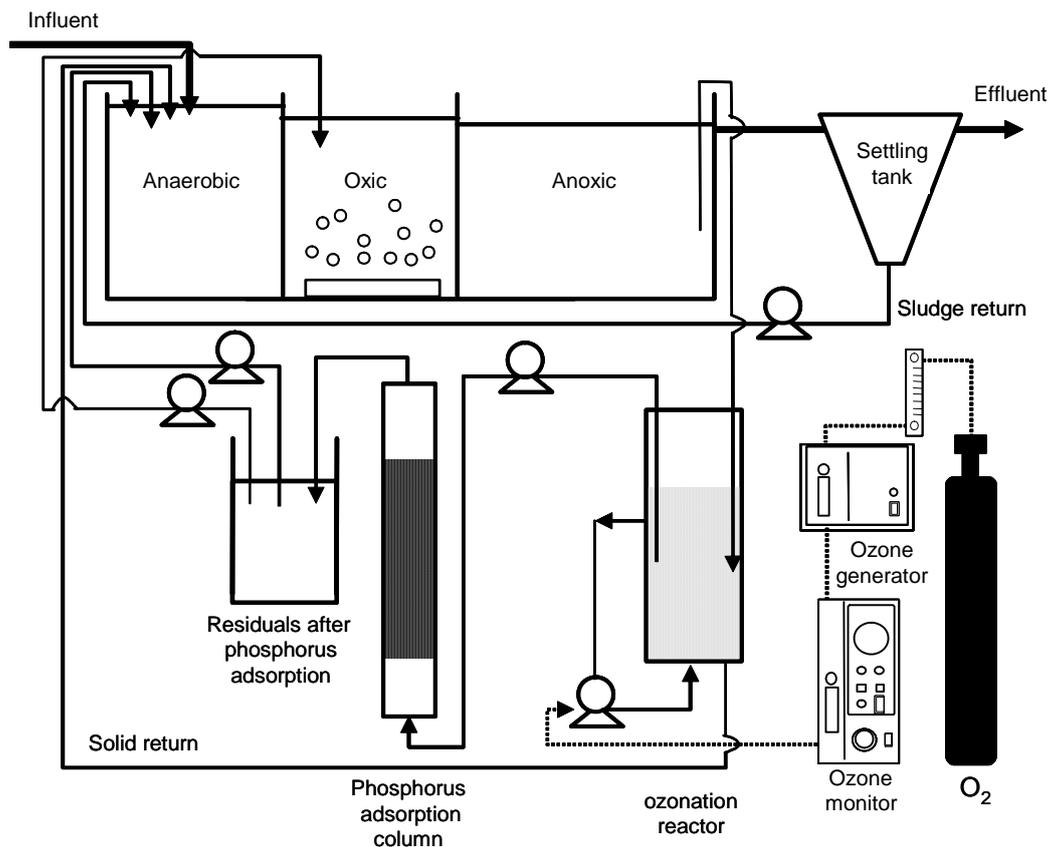


Fig. 1 Schematic diagram of the A/O/A process with ozonation and phosphorus adsorption.

Table 1 Operational conditions of the A/O/A process.

	Phase			
	1	2	3	4
Operating time (day)	0-22	23-54	55-83	84-152
Sludge amount for ozonation (L)	8.4	5.6	5.6	3.4
Ozone concentration (mg/L)	40	40	40	40
Ozone dose (mg-O ₃ /g-MLSS)	20	20	20	30
Flow rates of the supernatant derived from the physicochemical process				
Anaerobic tank (mL/min)	12.5	12.5	15	15
Oxic tank (mL/min)	12.5	12.5	10	10
Flow rate of influent wastewater (mL/min)	60	60	60	60
Flow rate of sludge return from the settling tank to the anaerobic tank (mL/min)	48	48	48	48

Table 2 General characteristics of raw wastewater.

Item	Conc. (mg/L)
T-N	45 - 55
NH ₄ -N	30 - 40
T-P	4.0 - 5.5
PO ₄ -P	2.5 - 3.0
TOC	55 - 80
BOD	160 - 220
SS	160 - 200

2.2.2 Phosphorus uptake rate (PUR) analysis

Phosphorus uptake rate (PUR) in Phase 4 was estimated according to the previous studies with some modification (Kuba *et al.*, 1997; Tsuneda *et al.*, 2006; Wachtmeister *et al.*, 1997). A 200 mL of activated sludge was collected from the settling tank at Phase 4 and transferred to a 500-mL polystyrene cup. The same volume of influent raw rural wastewater was soaked into the cup and incubated 90 min anaerobically. After anaerobic incubation, the sludge sample was divided into two cups. One was exposed to oxic conditions and the other was exposed to anoxic conditions (use of nitrogen gas and addition of 20 g-N/m³ NaNO₃ in the cup). Phosphate uptake

rate (PUR) was estimated from the slope of the line describing the linear decrease in phosphate concentration. The ratio of anoxic PUR to aerobic PUR (anoxic/oxic PUR ratio) was used as an index reflecting the fraction of DNPAOs (Kuba *et al.*, 1997; Tsuneda *et al.*, 2006; Wachtmeister *et al.*, 1997). The contribution of DNPAOs to denitrification was also estimated from the ratio of anoxic PUR to denitrification rate in anoxic condition (Kuba *et al.*, 1997).

2.2.3 Phosphorus recovery efficiency

Phosphorus recovery efficiency was determined in Phase 4. The total phosphorus in the influent wastewater was finally divided into three fractions: the discharged phosphorus from the system as effluent from the A/O/A process, the adsorbed phosphorus in the phosphorus adsorption column, and the accumulated phosphorus in the sludge. In this study, the actual recovered phosphorus as sodium phosphate in each phase could not be determined because the phosphorus recovery was conducted only once at day 120.

2.2.4 Analytical procedures

MLSS was measured following the standard methods (APHA, 1998). To determine soluble TOC (S-TOC), $\text{NH}_4\text{-N}$, $\text{NO}_x\text{-N}$, $\text{NO}_3\text{-N}$, and $\text{PO}_4\text{-P}$, water samples were filtered using a glass-fiber filter (GF/C, Whatman Japan KK, Japan). TOC and S-TOC were measured using a SHIMADZU TOC-VSCH (Shimadzu, Japan). Total nitrogen (T-N), total phosphorus (T-P), soluble T-N (ST-N), soluble T-P (ST-P), $\text{NH}_4\text{-N}$, $\text{NO}_x\text{-N}$, $\text{NO}_3\text{-N}$, and $\text{PO}_4\text{-P}$ were measured using a TRAACS 2000 (Bran+Luebbe, Japan).

2.3 RESULTS AND DISCUSSION

2.3.1 Long-term process operation

The A/O/A (anaerobic/oxic/anoxic) process with ozonation and phosphorus adsorption was operated. Figs. 2 and 3 show effluent water quality and MLSS concentration in the A/O/A process, respectively. Effluent water quality and sludge production were affected by the operational conditions of ozonation (Figs. 2 and 3). In Phase 1, MLSS in the A/O/A process dramatically decreased from the initial stage. On day 23, the sludge amount for ozonation was decreased from 8.4 L/day (23 % of total MLSS in the A/O/A process) to 5.6 L/day (16 % of total MLSS) (Phase 2), and as a result, MLSS concentration was maintained around 3,000 mg/L (Fig. 2). Although adequate control for sludge reduction was achieved, efficient nitrogen removal was not achieved and effluent T-N concentration increased to approximately 20 mg/L. Effluent T-N was mostly composed of $\text{NH}_4\text{-N}$, indicating that nitrification performance was deteriorated in the system. It is well known that organic carbon induces the dissolved oxygen competition between nitrifying bacteria and heterotrophic bacteria in an oxic condition, and thus it results in the deterioration of nitrification performance. Then, to reduce the organic carbon in the oxic tank, the return ratio of the supernatant of the ozonated sludge to the anoxic tank and the oxic tank was changed from 5:5 (anaerobic tank, 12.5 mL/min; oxic tank, 12.5 mL/min) to 6:4 (anaerobic tank, 15 mL/min; oxic tank, 10 mL/min) whereas the ozonation conditions were not changed in Phase 3 (Table 1). However, nitrification performance was not improved. In Phase 4, to maintain the population density of nitrifying bacteria (slow-growing autotrophic bacteria), the sludge amount for ozonation was decreased from 5.6 L/day to 3.4 L/day (9.4 % of total MLSS), and the ozone dose was increased to 30 mg- O_3 /g-MLSS (Table 1). As a result, effective nitrification performance and efficient nitrogen removal were achieved over 2 months with the slight increase of MLSS concentration (2,910 mg/L on day 84 to 4,030 mg/L on day 147 shown in Fig.3). In Phase 4, sludge reduction by ozonation was performed once in three days. Then, HRT was

decreased when the residuals from the physicochemical process recirculated to the A/O/A process. Although the nutrient dosage was also changed by this recirculation, stable nutrient removal was achieved. Consequently, 87%, 76%, and 81% of TOC, T-N, and T-P in the influent wastewater were removed in Phase 4, respectively.

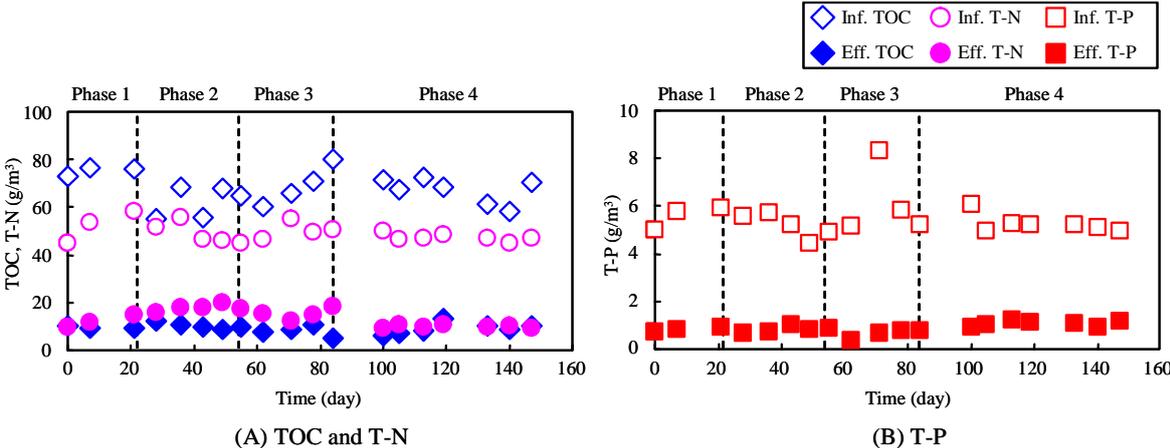


Fig. 2 Time course of water quality profile of influent and effluent.

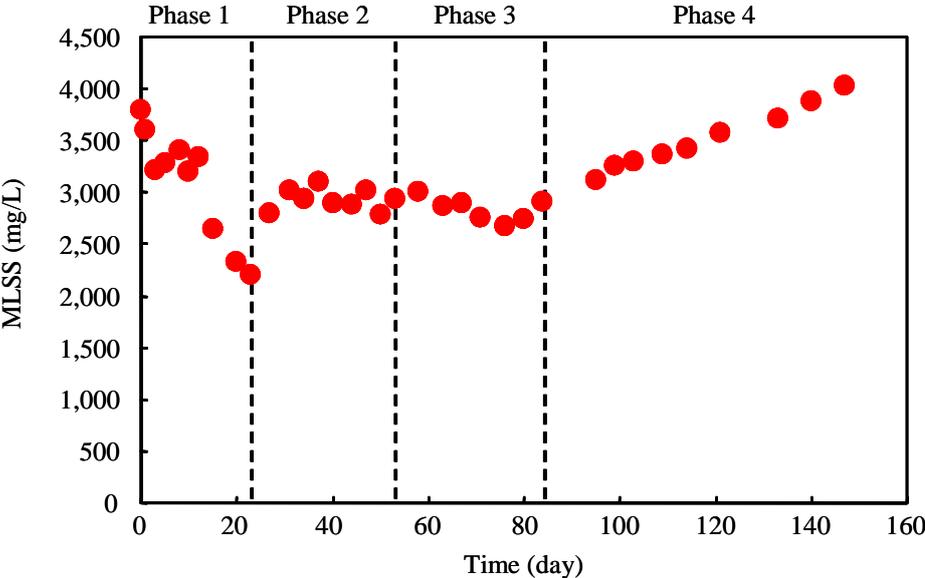


Fig. 3 MLSS concentration in the A/O/A process.

In our previous study (Suzuki *et al.*, 2006), the A/O/A process system equipped with a conventional ozonation system and phosphorus adsorption column was operated, and the residues of both the phosphorus adsorption column and the ozonation system were recirculated to the oxic tank. As a result, effluent S-TOC was deteriorated by slowly biodegradable materials which were derived from ozonated sludge (Suzuki *et al.*, 2006). In this study, this deterioration was not observed. These results indicated that some of the biorefractory and/or slowly biodegradable materials were oxidized to easily biodegradable materials by changing ozonation system and/or these slowly biodegradable materials were degraded in the anaerobic tank.

2.3.2 Phosphorus uptake by DNPAOs

Fig. 4 shows the water quality profile in Phase 4 (day 147). In the anaerobic tank, phosphorus release by PAOs and DNPAOs with assimilation of organic carbon was confirmed. Phosphorus was then accumulated in sludge both in the oxic tank by PAOs and in the anoxic tank by DNPAOs, suggesting that oxic phosphorus uptake was inhibited and the remaining phosphorus was accumulated by DNPAOs in the anoxic tank. To confirm the fraction of DNPAOs in total PAOs, phosphorus uptake rate (PUR) was estimated in Phase 4 and was compared with that reported in previous studies (Table 3). In this study, both the oxic- and anoxic PUR were lower than those in other studies. This was due to the low abundance of PAOs and DNPAOs in the process. On the other hand, the ratio of anoxic PUR to oxic PUR (anoxic/oxic PUR ratio) was almost the same as University of Cape Town (UCT) processes, in which the contribution of DNPAOs to phosphorus removal was confirmed (Kuba *et al.*, 1997). It demonstrated that DNPAOs took part in phosphorus removal. On the other hand, the anoxic/oxic PUR ratio was lower than that in the anaerobic/anoxic (A/A) SBR and A/O/A SBRs (Kuba *et al.*, 1997; Soejima *et al.*, 2006; Tsuneda *et al.*, 2006). These high anoxic/oxic PUR ratios in the A/A SBR and A/O/A SBRs were due to not the differences in the process types but the differences in influent wastewater: the A/A SBR and A/O/A SBRs received synthetic wastewater containing acetate as main organic carbon to enrich DNPAOs.

In the A/O/A process, nitrite/nitrate was reduced to nitrogen without S-TOC in the anoxic tank. The ratio of anoxic PUR to denitrification rate (P/N ratio) in this study and previous studies are shown in Table 3. The P/N ratio in this study was quite lower than that in previous studies, suggesting that other endogenous denitrifying bacteria, which had no ability to accumulate phosphorus, contributed to denitrification in the A/O/A process. The N/P ratio of only DNPAOs has not been determined because major DNPAOs have not been isolated, however, the endogenous denitrifying bacteria should contribute to denitrification in this reactor more than previous studies.

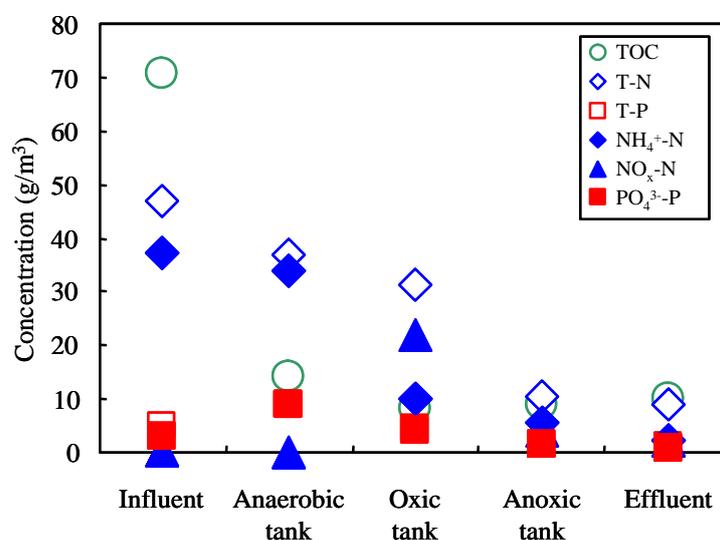


Fig. 4 Water quality profile at day 147 (Phase 4). Soluble TOC, T-N and T-P in A/O/A process were determined as TOC, T-N and T-P, respectively.

Table 3 Summary of kinetics in the PUR analysis of this study and previous studies.

	This study	UCT ^a	UCT ^a	A/A SBR ^a	A/O/A SBR ^b	A/O/A SBR ^c
Oxic PUR (mg-P/g-MLSS·h)	2.4	3.8	13	45	21 ^d	37 ^d
Anoxic PUR (mg-P/g-MLSS·h)	0.96	1.6	6	40	9.4 ^d	22 ^d
PUR ratio (Anoxic/Oxic)	40	40	50	90	44	60
Anoxic PUR/denitrification rate (mol-P/mol-N)	0.16	0.50 ^e	0.90 ^e	0.95 ^e	n.d ^f	n.d ^f

a: Kuba *et al.*, 1997. b: Tsuneda *et al.*, 2006. c: Soejima *et al.*, 2006 d: The value was obtained by using not MLSS but MLVSS. e: The value was obtained by multiplying the data as the unit "mol-P/mol-e" reported by Kuba *et al.* (1997) by 5 according to their assumption (5 electrons per mol nitrate denitrified). f: not determined.

2.3.3 Phosphorus recovery from excess sludge

Phosphorus concentrations in the influent and effluent of the phosphorus adsorption column are shown in Fig. 4. During the operation, about 70% of the phosphorus in the ozonated sludge was solubilized by ozonation, and a large part of the solubilized phosphorus consisted of $\text{PO}_4\text{-P}$. Over 90% of the solubilized phosphorus was adsorbed by zirconium-ferrite adsorbent in the adsorption column and effluent $\text{PO}_4\text{-P}$ concentration was maintained at less than 1 mg/L until day 119 (Fig.5). The phosphorus adsorption was continued until the breakthrough point of the adsorbent ($\text{PO}_4\text{-P}$ concentration: 1 mg/L). After 119 days operation, the adsorbent reached the breakthrough point and increase in effluent $\text{PO}_4\text{-P}$ concentration in the adsorption column was observed (Fig.5).

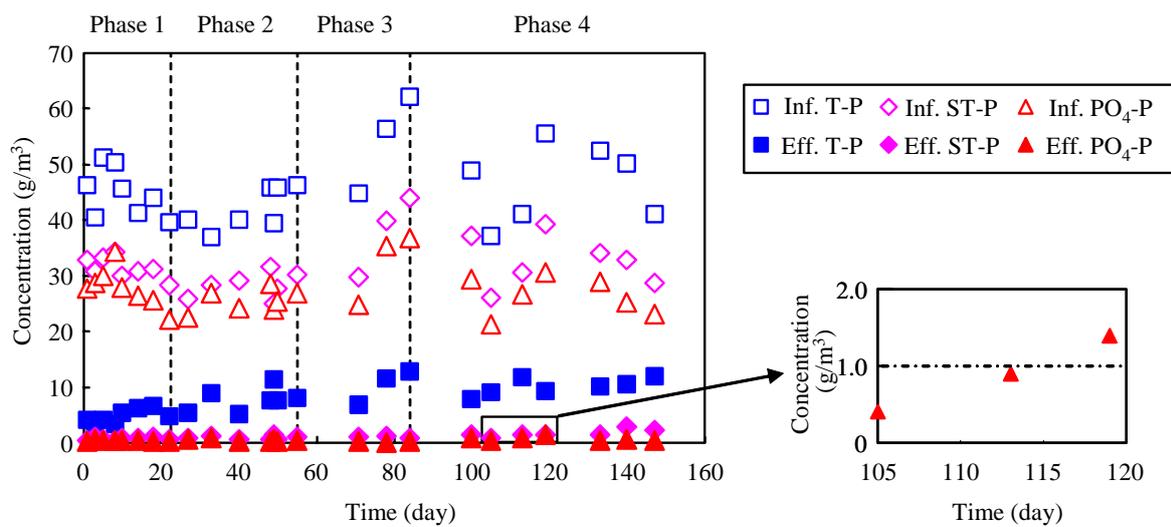


Fig. 5 Concentration of each type of phosphorus in influent and effluent of phosphorus adsorption column. Right small figure shows the breakthrough point ($\text{PO}_4\text{-P}$ concentration: 1 mg/L) of the adsorbent on day 119.

2.3.4 Feasibility of the ozonation system

As the amount of excess sludge increases, regulations for disposal have become increasingly stringent (Liu, 2003; Ødegaard, 2004). Many attempts have been made to reduce excess sludge production. In this study, an ozonation system was added to the A/O/A process and no excess sludge was withdrawn during 152 days operation. Adequate sludge reduction was achieved in Phases 2 and 3 (5.6L /day: 16% of total MLSS); however, nitrification efficiency was deteriorated. Consequently, the reduction in the amount of ozonated sludge improved the nitrification efficiency in Phase 4 (3.4L /day: 9.4% of total MLSS). Ozonated sludge would also act as a nutrient load in the A/O/A process. Cui *et al.* (2004) reported that ammonia derived from solubilized sludge was not completely reduced to nitrogen gas by energy sources originating from solubilized sludge, and that an additional carbon source was necessary for denitrification. Moreover, in the A/O/A process, HRT was decreased by the recirculation of the residuals which derived from the physicochemical process. Although nitrogen compounds in the residuals were not analyzed in this study, the deterioration of nutrient removal efficiency by this recirculation was not confirmed (Fig. 2).

Reduction of sludge production potential is also an important factor contributing to excess sludge reduction. Kuba *et al.* (1996) reported that sludge production efficiency could be decreased by using DNPAOs. In this study, the population density of PAOs and DNPAOs was slightly lower than the previous studies; however, DNPAOs played an important role for both the nutrient removal and the sludge reduction in the same manner as the previous studies (Table 3). Endogenous denitrifying bacteria contributed to nitrogen removal more than previous studies. These bacteria might also contribute to the reduction of sludge production potential. Meanwhile, sludge production potential should increase in the oxic tank by heterotrophic bacterial growth using the residuals of the physicochemical processes. To find the optimal condition, which achieve both no sludge production and efficient nutrient removal, would difficult in this process and changes in the operational conditions must be required for each type of wastewater. In this study, although slight increase in MLSS concentration was confirmed, both efficient nutrient

removal and no excess sludge withdrawing was achieved during at least 2 months operation (Phase 4). Further studies are necessary to determine the sludge production potential, and we believe that changing ozonation conditions may be one of the most important parameters affecting nutrient removal because the solubilized sludge contains both materials which are easy to biodegrade and materials which are difficult to biodegrade and nitrogenous compounds.

2.4 CONCLUSIONS

In order to meet increasingly stringent requirements for environmental protection and phosphorus resource recovery, an A/O/A process equipped with ozonation and phosphorus adsorption was tested under various ozonation conditions. Our main conclusions are as follows.

(1) Under the optimum ozonation conditions (3.4 L/day: 9.4% of total MLSS), 87%, 76%, and 81% of TOC, T-N, and T-P were removed, respectively. No excess sludge was withdrawn at least 2 months operation.

(2) In the A/O/A process, phosphorus was accumulated not only by PAOs in the oxic tank but also by DNPAOs in the anoxic tank. This existence of DNPAOs also contributed to the reduction of sludge production potential.

(3) Most of the phosphorus was solubilized from excess sludge by ozonation and a large part of the solubilized phosphorus consisted of $\text{PO}_4\text{-P}$. Over 90% of $\text{PO}_4\text{-P}$ was adsorbed by zirconium-ferrite adsorbent in the phosphorus adsorption column.

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