

Nitrogen removal from reject water with primary sludge as denitrification carbon source

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ABSTRACT

A novel system was used for nitrogen removal from reject water. This system includes one anoxic/oxic reactor for nitrification and a special reactor for denitrification in which primary sludge was added intermittently as electron donor. In denitrification reactor, sludge fermentation and denitrification reaction took place simultaneously and promoted each other. It was found that effluent recycle could improve nitrogen removal efficiency due to reclaiming of alkalinity. Under steady state conditions, the average solid retention time (SRT) in denitrification reactor was 12–15 d, a total nitrogen loading rate was 0.2 kg N/(m³ day) and TN removal efficiency was more than 90% without extra carbon source addition. Primary sludge was degraded so that volatile suspended solid (VSS) decreased by 50%. Further investigation showed that ORP could be taken as a control parameter for sludge addition.

Key words | denitrification, fermentation, nitrogen removal, primary sludge, reject water, VFA

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INTRODUCTION

Water deposited during sludge treatment process, called reject water, is often recycled to the headwork of the wastewater treatment plant (WWTP). Because organic nitrogen partly reduces to ammonia during digestion, reject water usually contains high strength ammonia accounting for 15%–25% of the total nitrogen load of WWTP (Janus & van der Roest 1997). Thus reject water is suggested to be treated separately to relieve the ammonia loading on WWTP. In particular, reject water has an unfavourable COD:N ratio, so that convenient external carbon source for denitrification should be employed.

The number of WWTP is increasing and large amounts of waste sludge are produced. Sludge treatment and disposal has become a major problem due to shortage of land for sludge filling and more stringent standards for farming use. However, waste sludge is an abundant carbon source, which can be used to improve biological nutrient removal (Pitman *et al.* 1992; Thomas *et al.* 2003).

In addition, naturally volatile fatty acid (VFA) produced from fermentation has been demonstrated to be as good as synthetic VFA for denitrification (Elefsiniotis & Wareham 2007). Several methods have been reported to exploit soluble carbon source from primary sludge fermentation (Teichgräber 2000), especially the sludge fermentation–elutriation processes which include both in-line and side stream configurations. In a fermentation–elutriation system, settled sludge is fermented at the bottom of primary settler tanks and recycled partially to elutriate VFA; or a separate SBR or UASB unit is designed to enhance fermentation, and then fermented sludge returned to primary settler can elutriate VFA with influent wastewater (Bouzas *et al.* 2007). Both schemes have been successfully applied in real-scale WWTP, but some disadvantages of fermentation–elutriation system are also noticed:

- (1) sludge retention time (SRT) is designed short enough to avoid methanogenic activity in order to improve

the efficiency; however, a comparatively long SRT is more beneficial for fermentation process (Chanon *et al.* 2006).

- (2) The elutriation efficiency is not satisfied since VFA increase in effluent is limited. So alkali or surfactant is usually added to enhance fermentation process (Chen *et al.* 2007; Jiang *et al.* 2007).

Primary sludge could be also directly mixed with wastewater containing nitrite and nitrate in one reactor to enhance denitrification performance (Wett & Rauch 2003). In this case, methanogenesis may cease due to the presence of NO_x^- ; even accumulation of VFA was observed at the end of the incubation period (Tugtas & Pavlostathis 2007a). Consequently, NO_x^- injection to sludge fermentation system could decrease methane product and make more VFA used for denitrification. But from the literature, little research has been carried on the integration of denitrification and fermentation (Vigneron *et al.* 2007).

In this research primary sludge was used to enhance nitrogen removal from reject water. And a special reactor is designed to make full use of the carbon source in primary sludge. Reject water was nitrified to nitrite in an anoxic/oxic (A/O) reactor, then its effluent containing high strength nitrite was pumped into a special reactor SFDR (sludge fermentation and denitrification reactor) where primary sludge was added intermittently. Nitrate and nitrite presence could inhibit methanogenesis to benefit primary sludge fermentation process. Simultaneously, fermentation products in the reactor such as VFA and hydrogen could also be used for denitrification.

The specific objectives of this study were to: (1) establish an approach to remove nitrogen from reject water in this novel reactor and evaluate feasibility of the system, (2) investigate the connection of two different processes: fermentation and denitrification, and better understand the effect of NO_x^- on fermentation process.

METHODS AND MATERIALS

Experiment system description

A schematic diagram of the pilot-scale plant consisting of A/O reactor, sludge fermentation and denitrification

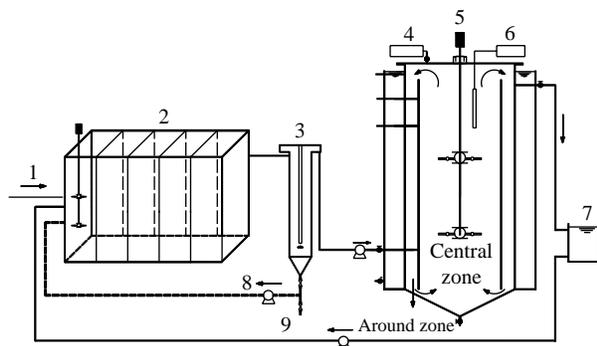


Figure 1 | Scheme of treatment system used (1-influent of reject water, 2-A/O reactor, 3-sludge settler, 4-gas meter, 5-sludge fermentation and denitrification reactor, 6-ORP meter, 7-effluent, 8-return sludge, 9-effluent recirculation).

reactor, clarifier is presented in Figure 1. The ratio of anoxic zone to aerobic zone of A/O reactor was 1:8, which are named as A and O1–O8 respectively. Sludge recycle ratio was maintained at 100%, and the reaction was carried out at room temperature ($T = 24.5\text{--}29.6^\circ\text{C}$). SFDR was divided into two compartments: “central zone” for sludge fermentation and nitrite/nitrate reduction; “around zone” for mixture sedimentation. The reactor was sealed with plastic covers and rubber stoppers for gas and ORP value measurement, and it was operated at 35°C with water jacket around. Reject water was pumped into A/O reactor 60 L/d to achieve nitrification and partial nitrogen removal. After sludge had settled in clarifier, effluent was pumped into the continued stirred tank reactor, which was fed with primary sludge. After sedimentation in around zone of SFDR, supernatant was decanted and part of it could be recycled to the inlet of A/O reactor in order to supply alkalinity and further oxidise ammonia released in SFDR. Other characteristics of the reactor are described in Table 1.

Source of reject water and primary sludge

Both the reject water and primary sludge used in this research were collected from GaoBeiDian municipal wastewater treatment plant in Beijing, China. The reject

Table 1 | Design of characteristics of pilot plant elements

Parameter	Volume (L)	Parameter	Volume (L)
A-O reactor	36	SFDR-central	150
Settler	18	SFDR-settle	100

Table 2 | Characteristics of the primary sludge

Parameter (mg/L)	Mean value
TSS (total suspended solids)	22,600
VSS (volatile suspended solids)	17,200
SCOD (soluble chemical oxygen demand)	780
TCOD (total chemical oxygen demand)	22,500
VFA (amount of acetic)	440
NH ₄ ⁺ -N	75

water was from dewatering of sludge from a mesophilic anaerobic digester, and stored in a 400 L tank. Ammonium concentration of the reject water was 350–400 mg/L, chemical oxygen demand (COD) was 300–450 mg/L, alkalinity concentration was about 2,000 mg CaCO₃/L, pH value range 7.8–8.4.

The primary sludge was obtained from the sludge transport pipes out of the primary sedimentation tank, and then stored at 4°C for later use. Before being added into the SFDR primary sludge was filtered in order to remove large blocks of substances such as grits; the other detailed characteristics are shown in Table 2.

Experimental process

Reject water after nitrification was pumped into SFDR. As carbon source of sludge ran out, NO_x⁻ concentration in the effluent became higher than given level. Then 20 L or 50 L mixture was disposed from SFDR and displaced with the same volume fresh sludge. Two test runs (Run A, Run B) were carried out over the course of this study. In Run A, nitrogen removal efficiency was investigated with recycle ratio being at 0, 100%, and 200%, respectively. The effect of alkalinity produced by denitrification process was also investigated. After that, Run B was conducted to investigate microorganism activity in SFDR and the effect of

Table 3 | Operation characteristics of A/O-SFDR process

Phase	Operation day	Effluent recycle ratio	Sludge addition volume	Sludge addition time*
A-I	1–13	0	20 L	Day 1,10,13
A-II	14–27	100%	50 L	Day 19,24
A-II	28–34	200%	50 L	Day 28,34
B	35–57	0	50 L	Day 41,48,55

*Each time equal mixture liquid in SFDR was excluded before primary sludge addition.

NO_x⁻ on denitrification. The effluents did not recycle in this period. Other details were presented in Table 3.

Analytical methods

In order to analyse nitrogen variation in A/O reactor and the fermentation products in SFDR during the experimental operation, the sludge was centrifuged and then the supernatant was measured in 12 h. NH₄⁺-N, NO₃⁻-N, NO₂⁻-N were measured according to the standard method (APHA 1995). VFA was measured by gas chromatography (14B, Shimadzu, Japan). TOC was measured by a multiN/C 3,000 analyser (Analytik Jena AG). OPR and pH value were real time monitored by using WTW pH/oxi340i and WTW pH730 and corresponding OPR and pH probes (WTW Company, Germany).

RESULTS AND DISCUSSION

The recycle ratio effect on nitrogen removal

The A/O-SFDR system showed a good performance on nitrogen removal from reject water. Figure 2 shows typical removal nitrogen variations of the system with effluent recycle ratio being 200%. In anoxic zone of A/O reactor the initial ammonia concentration was about 100 mg/L; due to free ammonium and free nitric acid inhibition, there was nearly no nitrate produced along the reactor, and the nitrate concentration was always less than 10 mg/L at the end. A/O reactor kept stable short-cut nitrification and nitrite accumulation (NO₂⁻/NO_x⁻) was more than 90%. After a short start-up, SFDR achieved good and stable performance for denitrification. In SFDR nitrate and nitrite were almost reduced when carbon source was sufficient. Although the ammonia concentration increased slightly in SFDR due

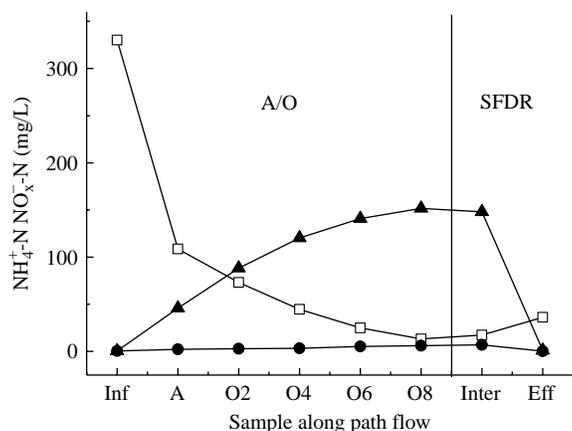


Figure 2 | Nitrogen variation in the flow path with effluent recycle ratio being 200%: $\text{NH}_4^+\text{-N}$ (—□—), $\text{NO}_2^-\text{-N}$ (—▲—), $\text{NO}_3^-\text{-N}$ (—●—).

to organic nitrogen decomposed or dissimilatory nitrate reduction to ammonia, average total nitrogen concentration in effluent was less than 60 mg/L. The effluent was always feasible to be recycled to the main treatment plant.

In order to get high total nitrogen (TN) removal efficiency, different processes should be coordinated together. Firstly, reject water was short of alkalinity, nitrification ceased at low pH due to alkalinity consumption, and furthermore residual ammonia cannot be removed in anaerobic conditions. So extra alkalinity was required for effective nitrification. Secondly, an optimum condition for SFDR was important in order to provide sufficient electron donor and decrease released ammonia amount.

In this investigation, influent alkalinity was 2,000 mg CaCO_3/L ; total oxidation of influent ammonia needed at least 2,800 mg CaCO_3/L . Extra alkalinity could reclaim from the effluent of SFDR because denitrification process produced certain amount of alkalinity. The theoretical alkalinity value of SFDR was calculated according to Formula (1):

$$\text{alk}_{\text{theory}} = \text{alk}_{\text{inf}} + 3.57 \times (\text{NO}_{\text{xinf}}^- - \text{NO}_{\text{xeff}}^-) \quad (1)$$

Figures 3 and 4 show the alkalinity and nitrification efficiency were related to effluent recycle ratio. Figure 3 indicates that measured alkalinity value matched theory value well. At the initial days predicted alkalinity was higher than measured value. Extra alkalinity was produced because CO_2 was generated by anaerobic metabolism and dissolved in liquid. Figure 4 illustrated that ammonia

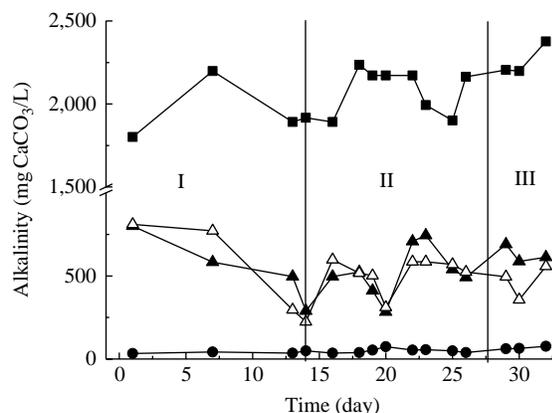


Figure 3 | Variation and simulation of alkalinity in the reactor: influent (—■—), AO effluent (—●—), In SFDR (—▲—), simulation in SFDR (—△—).

concentration increase was limited in SFDR. And it is obvious that the nitrogen removal efficiency was improved as the effluent recycle ratio increased from 0 to 200%. So in SFDR denitrification, rather than assimilation reaction or dissimilatory nitrate reduction to ammonia, was considered to be the main pathway of nitrate and nitrite reduction. In phase I, A/O effluent alkalinity basically ran out, low pH ($\text{pH} < 6$) strongly inhibited nitrification, so NH_4^+ kept at about 100 mg/L. As effluent recycled, it afforded alkalinity for nitrification; and nitrification produced more nitrate and nitrite to be reduced in SFDR. So alkalinity in effluent kept at 500 mg/L. Effluent ammonia removal efficiency increased with the effluent recycle ratio evaluation. As the recycle ratio was 100%, average effluent ammonia declined to 51 mg/L. As recycle ratio kept at 200%, almost all ammonia was oxidised and TN removal

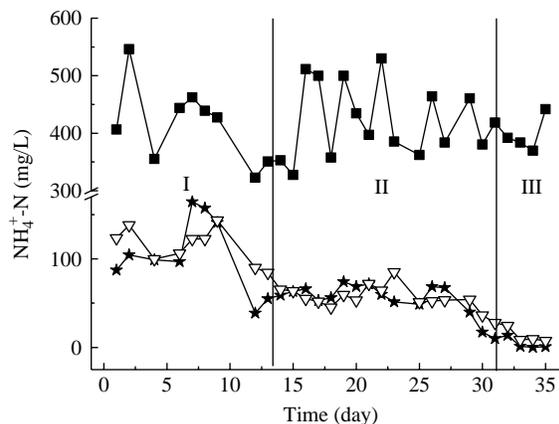


Figure 4 | $\text{NH}_4^+\text{-N}$ variation of system: A/O Influent (—■—), A/O effluent (—★—), SFDR effluent (—▽—).

efficiency was more than 90%. Higher recycle ratio was not investigated, because it would decrease hydraulic residence time and affect the sludge settle characteristic. Besides, the soluble microbial products in effluent recycle might inhibit nitrification process (Ichihashi *et al.* 2006).

Microorganism activity in SFDR

In Run B, NO_2^- , ORP value, VFA and TOC were measured continually in order to investigate the bio-reactions in SFDR. Figure 5 demonstrates a typical variation of different parameters in one period (between day 34 and day 41). Due to fresh primary sludge containing an abundant carbon source, both TOC and VFA concentration increased rapidly after sludge addition (day 35 and day 41). Soluble carbon substrate was converted to VFA, and VFA was consumed further by denitrifiers. TOC decreased gradually and descent rate became lower. In contrast VFA depleted sharply at the beginning, and then the value kept low until sludge was added again.

Although acidification rate usually is slower than denitrification rate, it could occur strongly in certain periods (Mosey 1983). After primary sludge addition, fermentation substrate was sufficient in the beginning and a certain quantity of VFA was injected simultaneously; acidogenesis bacteria were capable of producing VFA in a short time interval. As a result, electron donor was sufficient for nitrite and nitrate reduction.

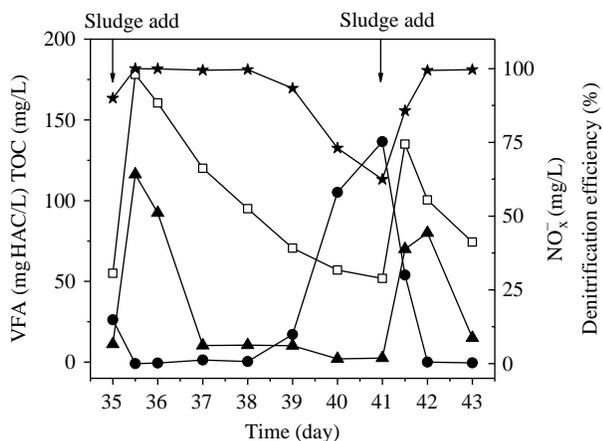


Figure 5 | Variation of different parameters in a typical SRT: NO_2^- (—●—), TOC (—□—), VFA (—▲—), and denitrification efficiency (—★—).

At a comparatively long HRT condition, fermentation and denitrification kept in balance, VFA was produced by acidification bacteria and oxidised simultaneously by denitrifiers. Consequently, nitrite was not accumulated even though VFA concentration was low. Afterwards, fermentation substrate decreased and the limited step hydrolysis became obvious. At the end of a period, hydrolysis became slow, resulting in denitrifiers lacking sufficient electron donor. Although TOC was more than 100 mg/L, most of them were not suitable for fermentation and denitrification. Therefore nitrite appeared in reactor and increased rapidly to more than 70 mg/L in 2 d; correspondingly the nitrite and nitrate removal efficiency declined to 60%. Accordingly fresh primary sludge was again added into SFDR.

ORP relationship with NO_2^- concentration

High concentration of nitrite is harmful to aquatic life; even if it is recycled to main treatment zone, it still requires soluble carbon source for denitrification, which is negative for municipal wastewater nitrogen removal, especially when the influent has a low C/N ratio. So nitrite concentration of separate reject water treatment should be below the given level. In this study, primary sludge as carbon source could be overloaded in case denitrification was incomplete. However, it meant a comparatively short solid retention time in SFDR, and primary sludge could not be treated well. Besides, in that situation more carbon source was carried away with effluent. Residual SCOD oxidised in A/O reactor was negative for nitrification due to competition between heterotrophic bacteria and nitrifiers. Therefore a simple and effective indicative parameter was required to control proper sludge addition.

It is reported that oxidation-reduction potential (ORP) can be used as process control parameter for denitrification process (Peng *et al.* 2007). Appearances of “nitrate knee” and “nitrite knee” are taken as the end of nitrate and nitrite reduction. Correspondingly, in this experiment ORP curve could be used as a parameter to indicate the appearance of nitrite in SFDR and the time for primary sludge addition. It should be noted that many complex biochemical reactions exist in SFDR; hence the ORP value was affected by many different materials. However, regularly ORP value could be calculated based on

the Nernst Equation (expressed in Equation (2)):

$$E = E^{\circ} - \frac{2.303RT}{nF} \log \frac{[\text{Red}]}{[\text{Ox}]} \quad (2)$$

E° is the standard oxidised reductive value, R is molar gas constant, T is temperature, n is numbers of transferred electrons, F is Faraday constant, $[\text{Red}]$ is reduced state materials concentration, and $[\text{Ox}]$ is oxidised state materials concentration. From Formula (2), ORP variation in SFDR is dependent on the concentration of oxidised and reduced material, especially the concentration of nitrite. At the beginning, injected nitrite was reduced rapidly and no accumulation was observed. So SFDR stayed under an anaerobic condition and the OPR value was low. As reaction continued, reductive materials such as TOC were gradually consumed and it would slow down the denitrification rate. ORP would rise significantly as a result of nitrite accumulation in SFDR.

ORP value and nitrite concentration in SFDR were continually measured. The result is presented in Figure 6. From the paragraph ORP was obviously relative to nitrite concentration. And variation trend of ORP was consistent in different periods. Typical ORP value in one period comprised four parts. Firstly, nitrite was reduced rapidly after sludge addition, ORP value declined sharply from -200 mV to -550 mV in a few hours, and conditions in SFDR transited from anoxic to anaerobic. Secondly, nitrite concentration was undetected and ORP value was level at about -500 mV for 2–3 d. Thirdly, nitrite was accumulated in SFDR, ORP value rose quickly from -555 mV to -250 mV. Finally, ORP value rose slowly to higher

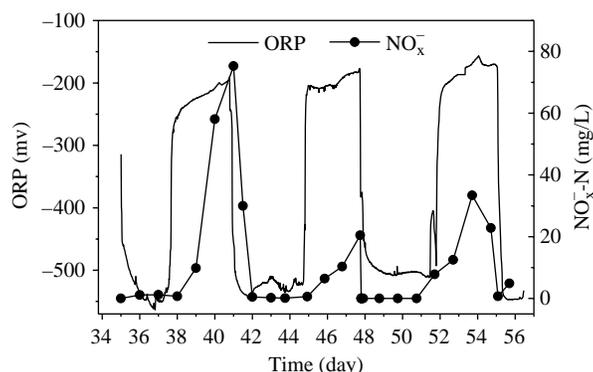


Figure 6 | ORP and nitrite variations in different periods.

than -200 mV in 2–3 d. Based on the semi-redox reaction NO_2^-/N_2 , $E = 956$ mV is obtained (Madigan *et al.* 1997). So the accumulation or disappearance of nitrite induced a sharp rise or decline of ORP value of SFDR, respectively. Nitrite concentration significantly affected the ORP value curve.

By the above analysis, during the denitrification, the variation of ORP profile could display the changes of nitrite concentration, and furthermore it could indicate whether the carbon source of sludge had been used up. Fresh primary sludge addition was optional at the turn point of ORP value curve. In addition, on the basis of the ORP profile, real time control strategy can be built: sludge addition is started when $\text{ORP} < -550$ mV and stopped when $\text{ORP} > -150$ mV. It was beneficial for making full use of sludge on the basis of good nitrogen removal efficiency.

Relationship between sludge fermentation and denitrification

Experimental results indicated that fermentation and denitrification could be integrated successfully in SFDR. If there was no fermentation process, SCOD in injected sludge would be used up in 6 h and nitrite and nitrate would appear correspondingly. As a matter of fact, complete denitrification can proceed over 2–3 d. Average VSS/SS ratio of primary sludge descended from 68.6% to 38.7% when disposed from the SFDR. It indicated that partial granular substrate was converted into SCOD and consequently used as electron donor.

Average VSS/COD ratio of primary sludge used in the experiment was 1.30 (shown in Table 2). According to the VSS of primary sludge variation results, combined gas output and nitrogen elimination results, 1 g NO_2^- -N reduction corresponded to 5.23 g VSS consumption in SFDR, which was close to theoretical value: 5.14 g VSS/g NO_2^- -N. Further calculation indicated 1 g VSS could produce 0.43 g VFA for denitrification (result was not shown). The conversion ratio was higher than common traditional fermentation–elutriation system. The reasons may lie in three aspects:

- (1) Methanogens converting acetic acid into methane may occur in 5 d in anaerobic conditions. However, in an

optimum situation VFA should be used by denitrifiers other than methanogens. Generally, methanogens grow slowly so that a comparatively short SRT is used in operation in order to decrease the methane formation. But in contrast a long SRT is beneficial to fermentative bacteria accumulation. In this experiment, the existence of nitrite and nitrate had a negative effect on methanogen bacteria, especially nitrite and other intermediate products of denitrification (Kluber & Conrad 1998). Due to these inhibitions, SRT in SFDR was prolonged to more than 12 d, which was longer than ordinary elutriation system.

- (2) Fermentation product used for denitrification process could benefit the whole process. It is reported that the application of micro-aerophilic conditions was likely to increase hydrolysis rate (Nguyen *et al.* 2007). In our investigation, nitrate and nitrite may enhance hydrolysis with the same mechanism. Hydrolysis/acidification was improved because final product was consumed continually; therefore more VFA was produced, which significantly improved the sludge use efficiency.

In ordinary anaerobic digesters, hydrogen partial pressure keeps low due to methanogenic bacteria converting hydrogen to methane. Low hydrogen partial pressure is important for the acetogenesis step where long VFAs are converted into acetic acid and hydrogen by acidogenic bacteria (Leslie Grady *et al.* 1999). Therefore, hydrogen partial pressure could reach high values in traditional fermentation–elutriation systems. In that case, the conversion will not proceed consecutively, causing long chain VFAs accumulation. H_2/CO_2 can be also used as feasible electron donor for nitrate and nitrite reduction, and the main pathway is denitrification (Tugtas & Pavlostathis 2007b). Although methanogens were inhibited in SFDR reactor, hydrogen partial pressure can be kept low by the action of denitrifiers. Therefore acetogenesis step can always proceed well.

- (3) Denitrification reclaimed alkalinity to evaluate pH value in SFDR. Figure 7 showed pH variation in Run A, though A/O effluent pH was low at 6.0–6.5 with different effluent recycle ratio. pH in SFDR ascended gradually, which was higher than 9.2 at the end of

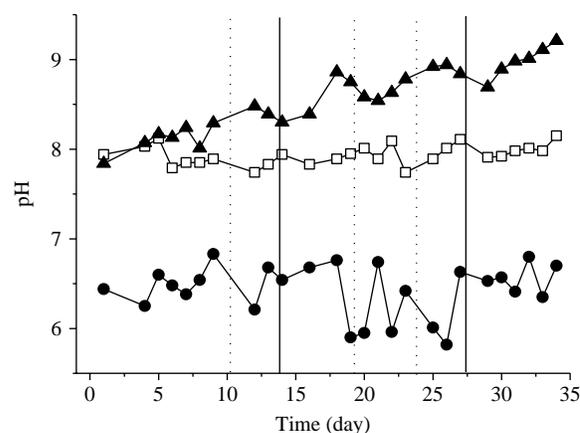


Figure 7 | pH variations of the system: influent (□), A/O effluent (●) and SFDR (▲).

phase 3. The fermentation product was consumed rapidly and alkalinity was produced continually in SFDR. As a result, SFDR could avoid pH being too low to inhibit fermentation process. In addition, it has been reported that the fermentation efficiency was improved at a neutral pH (Dinopoulou *et al.* 1988). And between pH 6.0 and 11.0 sludge hydrolysis rate increased with pH value (Chen *et al.* 2007). Ahn *et al.* (2004) also found optimal pH value was 9 for hydrolysis/acidogenesis of piggery waste.

Denitrification also benefited from the integration. In traditional fermentation–elutriation system, about 30% SCOD remained in sludge and concentration was diluted by elutriating water. In SFDR, a high C/N ratio improves the denitrification procedure; the acetic acid portion of total organic acids increases with increasing pH (Ahn & Speece 2006). Besides, in SFDR reactor influent oxygen was depleted rapidly. Thus a completely anaerobic condition could be kept to avoid the negative effect of dissolved oxygen, which improved the denitrification efficiency significantly.

CONCLUSIONS

Nitrogen removal from reject water was investigated in a novel A/O-SFDR system. And it was concluded with following results:

- (1) AO-SFDR system showed a stable and efficient nitrogen removal from reject water using primary

sludge as denitrification carbon source. TN removal efficiency was more than 90% and the effluent was suitable to be recycled to main plant directly.

- (2) ORP value could be used as a control parameter for nitrite concentration to determine primary sludge addition. And primary sludge was degraded obviously.
- (3) Fermentation and denitrification can be integrated in one reactor and benefit each other under optimum conditions.

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