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Nitrite Accumulation in Activated Sludge and Airlift Reactors: Process Performance Comparison

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ABSTRACT

Nitrogen removal via nitrite allows operational cost reduction in wastewater treatment of effluents with a low COD/N-NH_4^+ ratio, due to lower oxygen requirements for partial nitrification and a further diminishing in requirements of the external carbon source for the denitrification step. Two nitrifying systems—a suspended activated sludge (AS) reactor, and an airlift biofilm (AB) reactor—were compared to determine the best alternative for obtaining a maximum nitrite buildup. Both systems were operated under similar operational conditions; synthetic feed of ammonia nitrogen was applied to both reactors, at an ammonia loading rate (ALR) of 3.3 kg N-NH_4^+/m^3 and an hydraulic retention time of 4.3 h. Nitrite accumulation was studied by means of controlling dissolved oxygen (DO) concentration, keeping both pH and temperature constant. Similar results were obtained in both systems. In the activated sludge reactor maximum nitrite accumulation was achieved at 0.7 and at 3.5 mg DO/L in the airlift reactor. The obtained results showed that at a similar percentage of nitrite accumulation, the K_La value of the airlift reactor was always 3.6 times higher than the value obtained in the activated sludge reactor; however, the airflows required were similar in both reactors. The operation of nitrifying systems (biofilm and suspended biomass reactors) with low oxygen concentration to obtain nitrite accumulation make possible a decrease of required airflow, even at small nitrite accumulation. Airflow savings of 38 and 58% were obtained for the activated sludge reactor and the airlift reactor, respectively, at 15% of nitrite accumulation. When 40% of nitrite accumulation was detected in the activated sludge reactor and the airlift reactor, a savings of 80% and 65% were obtained, respectively.

Key words: nitrification; nitrite accumulation; K_La; activated sludge; airlift; airflow

INTRODUCTION

THE BIOLOGICAL NITROGEN REMOVAL PROCESS (nitrification/denitrification) is a technological, as well as economical, alternative for treating wastewater containing nitrogenous compounds. This process consists in two stages, nitrification and denitrification, which allow the transformation of ammonium into gaseous nitrogen.

An alternative to conventional sequences of predenitrification and postdenitrification is the denitrification

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*Corresponding author: Biochemical Engineering School, P. Universidad Católica de Valparaíso, General Cruz 34, Valparaíso, Chile. *Phone:* 56-32-273640; *Fax:* 56-32-273803; *E-mail:* gonzalo.ruiz@ucv.cl process via nitrite (Hellinga *et al.*, 1998; van Dogen *et al.*, 2001; Hao *et al.*, 2002; Ruiz *et al.*, 2003). This alternative is based on the fact that in the nitrification step, ammonia is first converted into nitrite, which is oxidized into nitrate at oxygen expenses. Subsequently, in the denitrifying step, nitrate is first reconverted into nitrite and finally into nitrogen gas, consuming organic carbon in each step. By a partial nitrification to nitrite and a post-denitrification from nitrite, about 25% oxygen and up to 40% organic carbon could be saved (Leal *et al.*, 2002; Ruiz *et al.*, 2003; Ciudad *et al.*, 2005). Other processes such as SHARON and OLAND carry out the denitrification step using the ANAMMOX process, where there was no need for organic matter addition. The SHARON process has been used at the industrial scale for the treatment of supernatant produced by sludge digestion, rather than the majority of wastewater due to its requirement of a high temperature. On the other hand, the OLAND system has not been tested on an industrial scale.

A lot of research has been focused on the study of the optimum condition for nitrite accumulation on nitrifying systems; most of the research is based on the effect of operational parameters (pH, temperature, free ammonia, and dissolve oxygen concentration). To obtain nitrite accumulation in a nitrifying process, nitrite oxidizing activity must be inhibited, maintaining high ammonium oxidizing activity. A stable operation under these conditions could be difficult due to the inhibition of nitrite over ammonium oxidizing micro-organisms, especially when the nitrifying reactors are operated at high ammonia loading rate (ALR) (Hunik *et al.*, 1992; Carrera *et al.*, 2004)

Free ammonia effect

According to Anthonissen *et al.* (1976), ammonia oxidizing and nitrite oxidizing bacteria are inhibited by their own substrates, and he proposed that nonionized forms of ammonium and nitrite, NH_3, and HNO_2, respectively, have an inhibitory effect on both ammonia oxidizing and nitrite oxidizing bacteria, but nitrite oxidizing bacteria are more sensitive to ammonia than ammonia oxidizing bacteria. Abeling and Seyfried (1992) reported that concentrations of 1–5 mg N-NH_3/L inhibited nitratation but not nitritation. Turk and Mavinic (1986, 1987) report nitrite accumulation with intermittent contact to high levels (5 mg N-NH_3/L) in the first chamber of a four-chamber system.

Temperature

Balmelle *et al.* (1992) showed that it is possible to accumulate nitrite by setting the temperature beyond 20–25°C. In the SHARON process (Hellinga *et al.*, 1998, 1999), the system is operated at temperatures above 25°C where ammonium oxidizers are selected.

Ratio DO/FA effect

Çeçen and Gonec (1994) reported that nitrite accumulation reached a considerable degree at a dissolved oxygen (DO) to free ammonia (FA) ratio lower than 5 during nitrification, and formation of nitrate was inhibited.

pH effect

Ruiz *et al.* (2003) and Jianlong and Ning (2004) did not find a pH effect over nitrite accumulation in a wide range of pH from 6.45 to 8.95. Nevertheless, their results showed that DO concentration is the key parameter to achieve a stable and high nitrite accumulation.

DO effect

The OLAND process controls DO between the above ranges to inhibit nitratification rate and the growth of nitrobacteria. Then the produced NO_2^- by NH_4^+ oxidation continue to oxidize the remaining NH_4^+ to produce N_2 by the ANAMMOX process, but such a process has only been tested at a lab scale.

Several works have studied nitrification systems with the aim of achieving partial nitrification with nitrite accumulation in biofilm systems (Garrido *et al.*, 1997; Lui and Tay, 2001), activated sludge (Hanaki *et al.*, 1990; Ruiz *et al.*, 2003), and sequencing batch reactor (Yoo *et al.*, 1999; Bae *et al.*, 2001), but these investigations only studied the effect of specific operational parameters over the different micro-organisms populations. Few works have been developed to give a full consideration of mass transfer aspects in the partial nitrification processes (Picioreanu *et al.*, 1997; Hellinga *et al.*, 1999; Hao *et al.*, 2002) considering parameters related with reactors design such as K_La or aeration flow, which of course, would have and important effects on the operational behavior and costs.

The object of this work was to compare nitrite accumulation in two reactor configurations: an activated sludge, and an airlift biofilm system. Both reactors were operated with a high load of synthetic wastewater without addition of organic matter. Nitrite accumulation is studied by means of controlling the DO on concentration. The mass transfer coefficient K_La was also evaluated for both reactors, and startup and stability of the operation were compared.

MATERIALS AND METHODS

Experimental setup

Experiments were carried out in two reactors—an activated sludge reactor, and an airlift reactor of 2.5 and 3.5 L of useful volume, respectively.

Table 1. Concentrated synthetic wastewater and trace solution composition.

Concentrated synthetic wastewater		*Trace solution*	
Compound	*Concentration (g/L)*	*Compound*	*Concentration (g/L)*
NH_4Cl	19.3	$EDTAH_2Na_2 \times 2H_2O$	50.0
$MgSO_4$	6.5	$ZnSO_4 \times 7$ H2O	2.2
KH_2PO_4	2.5	$CaCl_2$	5.54
$(NH_4)_2SO_4$	23.6	$MnCl_2 \times 4H_2O$	5.6
NaCl	20.0	$FeSO_4 \times 7H_2O$	5.0
Trace solution	5 (mL/L)	$(NH_4)_6Mo_7O_{24} \times 4H_2O$	1.1
		$CuSO_4 \times 5H_2O$	1.57
		$CoSO_4 \times 7H_2O$	1.9
		KOH	to pH 6

The activated sludge was operated with suspended nitrifying micro-organisms, while the airlift reactor has a nitrifying biofilm developed over sand-like support. Both reactors were inoculated with the same sludge, a nitrifying sludge enriched for several years (Campos, 2000).

Both reactors had an automatic pH control, to ensure stability of the process. Dissolved oxygen concentration was manually adjusted to the desire value, by means of controlling air flow rate twice a day.

Influent composition

Both reactors were fed with the same synthetic wastewater. Table 1 shows the concentrated stock solution of 10 gN-NH_4^+/L (Campos, 2000). This stock solution was diluted with tap water to obtain the desire concentrations used for the experiments (about 600–670 mg N-NH_4^+/L).

Analytical methods

Nitrogen compounds in the liquid phase were measured daily by spectrophotometric methods: 4500-NH_3-F, 4500-NO_2-B, and 4500-NO_3-B of Standard Methods (APHA, 1985) for ammonium, nitrite, and nitrate, respectively. DO was determined twice a day using an oxygen electrode (YSI-95, YSI Company, Yellow Springs, OH).

Experimental procedure

Operational conditions, except DO concentration, were maintained as constant during the whole experiment in both reactors: ALR of 3.3 kg N-$NH_4^+/m^3 \cdot$ day, hydraulic retention time (HRT) of 4.6 h, and temperature at 31°C. These conditions simulated the effluent of an anaerobic digester, where high ammonia and low COD concentrations are usually detected. pH was automatically controlled to 7.8 ± 0.05 with the addition of a sodium bicarbonate solution (60 g/L); this solution was also the carbon source for nitrifiers. Initial conditions for both reactors are presented in Table 2. The SHARON process was not considered, since it implies a change in the process configuration and it is expected to be efficient at higher temperatures (above 35°C) which will be useful only if the effluent of a thermophilic process is considered; otherwise, heating of the effluent must be needed, increasing operational cost. On the other hand, partial nitrification through DO control will reduce operational cost, and can be applied at 31°C, as in this study, or a lower temperature.

Once stable operation was achieved, step changes in DO concentration were applied to study its influence on nitrite accumulation. Once stable accumulation of nitrite had risen, a new decrease of DO was performed. This strategy was followed until a loss of efficiency, in nitrite accumulation terms, was observed or a high concentration of ammonia was detected. Table 3 shows a DO concentration and the superficial gas velocities for both reactors along the experiment.

Table 2. Reactor initial conditions.

Parameter	*Value*
N-NH_4^+ influent	600–670 mg N-NH_4^+/L
ALR	3.3 Kg N-$NH_4^+/m^3 \cdot$day
HRT	4.6 h
DO	5.7 mgDO/L
SRT	20 days[a]
Temperature	31°C
pH	7.75–7.85

[a] Only for activated sludge.

Table 3. Operational conditions applied to both reactors.

Activated sludge unit			*Airlift reactor*		
Days	*DO (mgDO/L)*	*Sup. gas velocity (m/s)*	*Days*	*DO (mgDO/L)*	*Sup. gas velocity (m/s)*
0–2	5.5	0.027	0–7	5.5	0.099
3–6	4.7	0.017	8–12	4.5	0.061
7–17	3.7	0.011	13–22	3.5	0.032
18–20	2.7	0.08	22–28	3.0	0.023
21–27	1.7	0.05	28–35	2.5	0.017
28–31	1.4	0.03			
32–44	0.7	0.01			
45–49	0.5	0.01			

K_La determination

The overall oxygen transfer coefficient (K_La) was determined in both reactors using the steady-state method proposed by Moo-Young (1985), based on the supposition of a liquid phase steady state, then oxygen balance can be written by the following equation:

$$K_La = \frac{N_A}{(C^* - C_L)} \qquad [1]$$

where K_La is the overall oxygen transfer coefficient (h^{-1}); C^* is the saturation oxygen concentration in the liquid (mg/L); C_L is the actual oxygen concentration in the liquid (mg/L); and N_A is the oxygen requirement for the biological ammonia oxidation (mg/L_h), expressed as:

$$N_A = \frac{F}{V} \times \left(C_{NO_3} \times Y\frac{O_2}{NO_3} + C_{NO_2} \times Y\frac{O_2}{NO_2} \right) \qquad [2]$$

where F is the feed flow (L/h); V is the reactor volume (L); $C_{NO_3^-}$ is the nitrate concentration produced by total ammonia oxidation (mg/L); $C_{NO_2^-}$ is the nitrite concentration produced by partial ammonia oxidation (mg/L); $Y_{\frac{O_2}{NO_3^-}}$ is the oxygen yield for total ammonia oxidation 4.57 mg DO/mg N; and $Y_{\frac{O_2}{NO_2^-}}$ is the oxygen yield for partial ammonia oxidation 3.43 mg DO/mg N.

Reducing the airflow in the reactors the DO concentration was modified. Once the new condition was applied, the K_La value was determined when the concentration of nitrogenous compound inside the reactors was stable, considering at that point steady-state conditions.

RESULTS AND DISCUSSION

Startup of the activated sludge reactor required 40 days to achieve removal of the ALR applied (3.3 kg

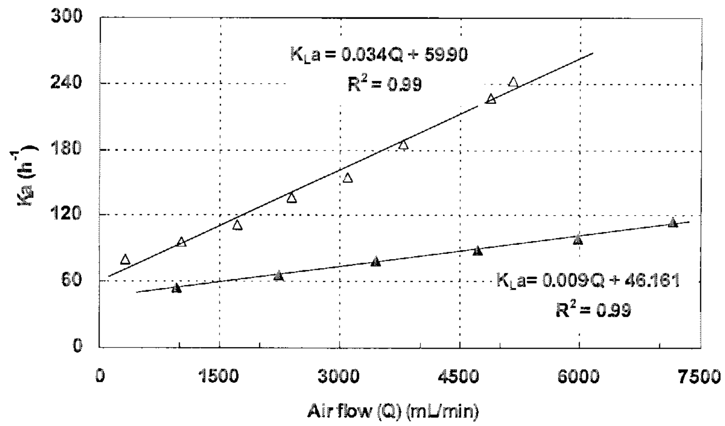

Figure 1. Overall mass transfer coefficient (K_La) determination for both reactors: (△) airlift reactor, (▲) activated sludge reactor.

N-$NH_4^+/m^3 \cdot$ day) while in the airlift reactor, the same results were achieved after 95 days of operation (data not shown), possibly due to biofilm formation. Several factors influence biofilm development such as support characteristics like roughness and porosity (Heijnen *et al.*, 1997). Diffusion restrictions are important factors in attached micro-organism systems, because the substratum must be transported from the bulk liquid to the inside of the biofilm, implying a lower growth rate than the suspended biomass, which has no (or less) diffusion problems.

Another parameter evaluated in both reactors was the K_La. Figure 1 presents the K_La as a function of air flow rate for both reactors. As can be seen, the K_La of the airlift reactor was 3.6 times higher (slope ratios) than the K_La of the activated sludge reactor. Higher values of K_La on the airlift reactor are possible due to the large rate of gas circulation, which results in high gas holdup.

After startup, once stable operation was achieved, the experiment of nitrite accumulation started. DO was decreased in small steps, giving enough time to achieve stable operation in each DO concentration. Figures 2A and 2B show the nitrogen compounds concentration during the experiment in the activated sludge and airlift reactors, respectively.

In the activated sludge reactor, total nitrification was achieved at DO concentrations between 5.7 to 2.7 mg DO/L. When the DO was reduced to 1.7 mg DO/L, an accumulation of 80 mg N-NO_2^-/L was detected. This

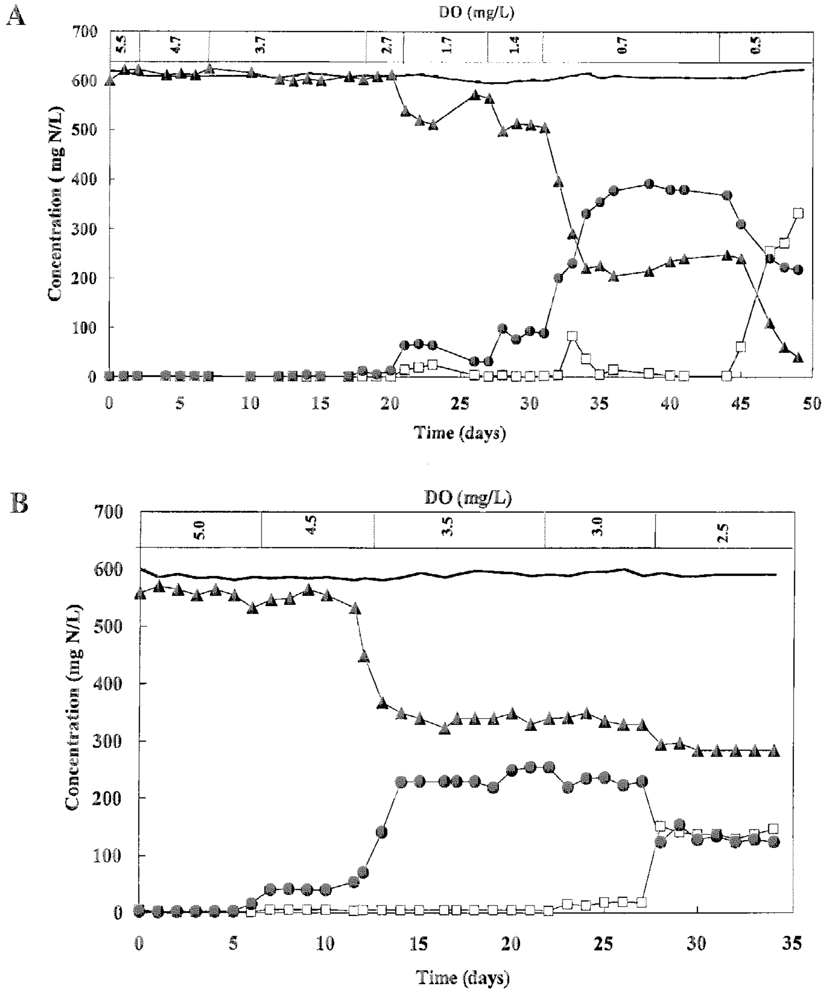

Figure 2. Nitrogen compounds evolution in the activated sludge (A) and airlft (B) reactors: (——) ammonia influent, (□) ammonia effluent, (●) nitrite effluent, (▲) nitrate effluent.

concentration was slightly increased (100 mg N-NO_2^-/L) by reducing DO to 1.4 mg O_2/L. Ammonia oxidizing bacteria were not affected by these oxygen reductions, as long as ammonia concentration in the effluent was kept below 3 mg N-NH_4^+/L.

At DO of 0.7 mg DO/L maximum nitrite accumulation was achieved with a concentration over 400 mg N-NO_2^-/L, a temporal increment of ammonium concentration was detected inside the reactor, but after a few days, ammonium concentration was negligible again.

A high accumulation of ammonia (over 350 mg N-NH_4^+/L) was observed at a DO lower than 0.7 mg DO/L, while nitrite concentration decreased to 250 mg N-NO_2^-/L, indicating a decrease in the activity of ammonia and nitrite oxidizing bacteria, due to the low DO concentration. These results show that the best DO concentration for maximum nitrite accumulation, for these experimental conditions, was around 0.7 mg DO/L. A complete ammonia conversion and a nitrite accumulation of more than 400 mg N-NO_2^-/L in the activated sludge reactor were obtained.

Airlift reactor has a similar behavior, but DO concentration at which nitrite accumulation occurred was higher. When DO concentration was 4.5 mg DO/L, a slight nitrite accumulation was observed in the reactor (less than 40 mg N-NO_2^-/L) while ammonium concentration inside the reactor was not detected. At 3.5 mg DO/L the maximum nitrite accumulation was achieved in this reactor, with a nitrite concentration over 270 mg N-NO_2^-/L (42% of total nitrogen fed to the reactor), and a complete ammonium removal. When DO concentration was decreased further, ammonium concentration was gradually increased up to 160 mg N-NH_4^+/L at a DO of 2.5 mg DO/L. These results showed that maximum nitrite accumulation in the airlift reactor occurs at a DO concentration around 3.5 mg DO/L, and under the conditions used in this study it was possible to accumulate over 270 mg N-NO_2^-/L by this system.

These results were quite different than the ones obtained by other authors in airlift reactors. Garrido *et al.* (1997) observed a maximum nitrite accumulation at 1.8 mg DO/L. This fact can be explained by the differences between the systems used such as geometrical relationships, ALR applied, biofilm thickness (internal diffusion), and hydraulic conditions (external diffusion).

With regard to the type of bubble regime (ven Benthum *et al.*, 1999) on the airlift reactor, when the DO concentrations were 5.5 and 4.5 mgDO/L the system show a *regime II*, which implies the stratification and stagnation of bubbles in the downcomer, while at 3.5, 3.0, and 2.5 mg DO/L the regime observed was *regime I*, in which gas was not present in the downcomer.

Figure 3 shows a comparison of ammonia removal and nitrite accumulation percentage in both studied systems.

The airlift reactor was sensitive at higher DO concentrations than the activated sludge reactor; at concentrations below 3.5 mg DO/L, ammonia oxidizing was affected, while in the activated sludge this fact happened at a concentration below 0.7 mg DO/L. On the other hand, the maximum percentage of nitrite accumulation was higher in activated sludge (62%) than in biofilm systems (43%).

Figure 4 shows the K_La and Q/V (air flow/volume of reactor) values obtained during the nitrite accumulation operation. These values were calculated using Equations (1) and (2), as mentioned before.

As was expected, the values of K_La obtained in both reactors (Fig. 4A) were lower than those obtained when the systems were operated for complete nitrification. This

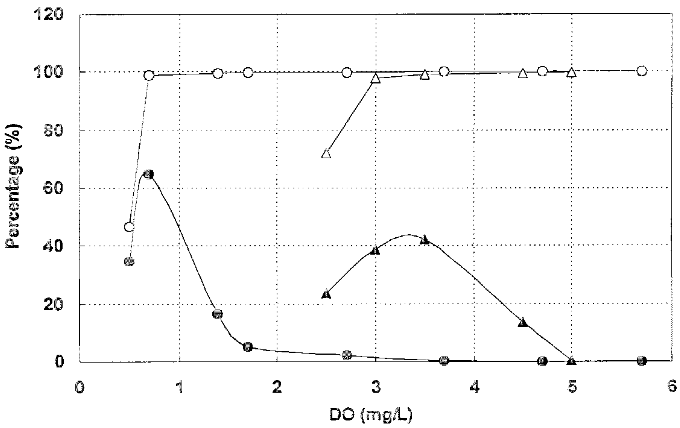

Figure 3. Ammonia removal percentage in the airlift reactor (△) and the activated sludge reactor (○). Nitrite accumulation percentage in airlift reactor (▲) and activated sludge reactor (●).

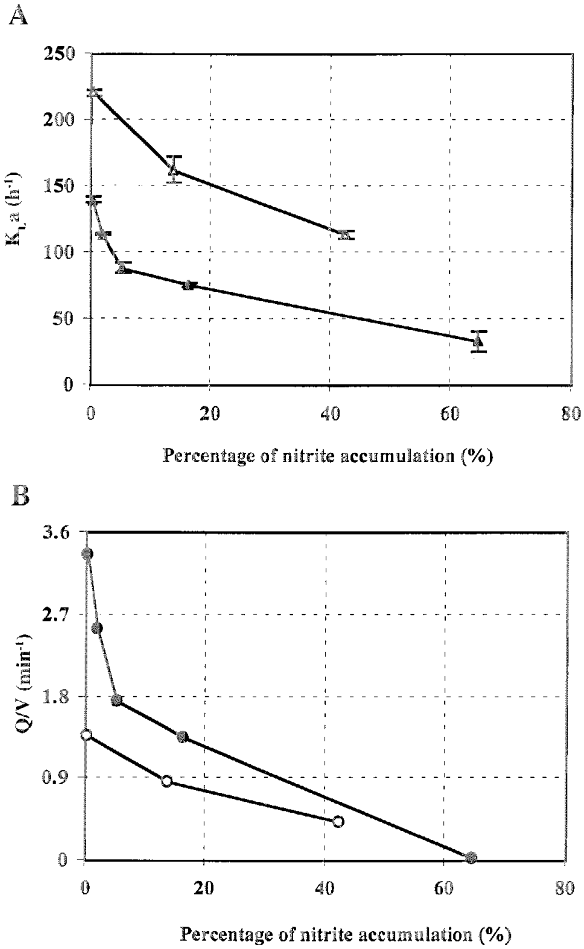

Figure 4. Nitrite accumulation operation: K_La (A) Activated sludge (▲) and airlift (△) bars indicate one standard deviation; Q/V ratio (B) Activated sludge (●) and airlift (○).

occurred due to the lower airflow requirement for the partial nitrification process.

With regard to the airflow required, Fig. 4B shows that for partial nitrification both reactors presented a large decrease in the airflow required. This figure shows that the activated sludge reactor can operate at lower airflow values, achieving higher percentages of nitrite buildup. The airlift reactor cannot work at the same operational conditions because when Q/V values decrease below 0.4 min^{-1}, an accumulation of ammonia inside the reactor was observed and, on the other hand, this reactor required higher values because the aeration makes possible the fluidization of the support, and also ensures well-mixed properties inside the reaction chamber.

Figure 5 shows the percentages of decrease of airflow in both reactors during nitrite accumulation operation in comparison with their operation at the lowest DO concentration values without nitrite accumulation, 3.7 mg DO/L and 5.0 mg DO/L, for activated sludge and airlift reactor, respectively. In the activated sludge reactor a high save of airflow was observed even when the percentage of nitrite accumulation was lower than 20%, as shown in Fig. 5. In this reactor it was possible to save up to 98% of aeration in the lab-scale reactor. For the airlift reactor a 70% decrease in aeration was achieved when the percentage of nitrite accumulation was 43%.

These results showed that both systems achieved similar results to ensure a stable operation when they were operated to attain nitrite accumulation. The activated sludge allows higher percentages of nitrite accumulation to be obtained than the airlift reactor. This occurred due to the better control of DO concentration on the activated sludge reactor in comparison with the airlift reactor. The fact that the biomass grows, developing a biofilm on the support surface, implied the existence of a profile of the

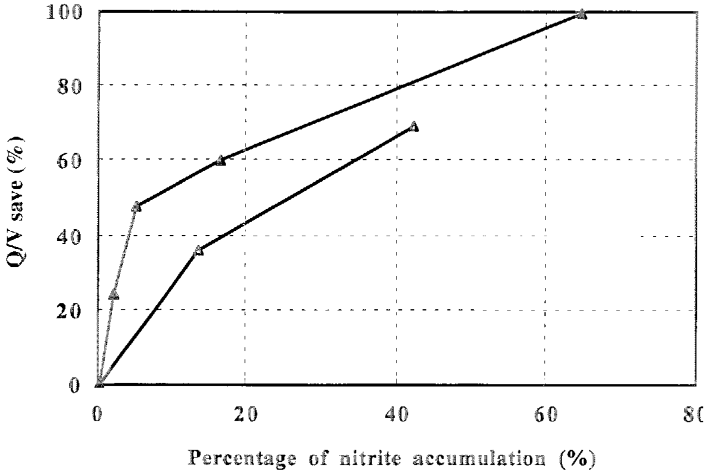

Figure 5. Percentage of Q/V save at different percentage of nitrite accumulation for (△) airlift and (▲) activated sludge reactors.

oxygen concentration inside the biofilm, implying that the micro-organisms in the deeper layer are exposed to a lower concentration of oxygen than the bacteria on the outer layers. So the activated sludge reactor seems to be the better alternative to develop a partial ammonia oxidation operation than the airlift reactors. However, the operation under nitrite accumulation condition could be a good alternative to upgrade and optimize biofilm systems as well as suspended biomass systems to save energy and the addition of a substrate in the next denitrification step.

To use the obtained results on the design of industrial-scale bioreactors the known scale up rules should be used for both reactors. To avoid the influence of geometry on the hydrodynamic parameters similar geometry should be used. If geometry similarities are followed and similar superficial gas velocities are achieved (main parameter to calculate oxygen mass transfer) then possible similarities between industrial scale systems and our results can be considered.

These results may not be generalized because nitrite accumulation will depend on other factors, such as FA, temperature, ALR applied, and biofilm thickness, among others. This may affect the DO concentration at which maximum nitrite accumulation is achieved. But the comparison between both reactors is acceptable because the same conditions were applied to both reactors.

CONCLUSIONS

It is possible to accumulate nitrite in a nitrifying reactor decreasing DO concentration. At experimental conditions used in this work, a maximum nitrite accumulation of around 0.7 and 3.5 mg DO/L was achieved in an activated sludge reactor and an airlift reactor, respectively.

As the K_La in both reactors were different—over 3.6 times higher in the airlift reactor—less air flow rate is needed in the airlift reactor for the same DO concentration in the activated sludge. But regardless the biofilm thickness, high DO concentrations are needed in the air lift reactor to obtain the same results than the activated sludge reactor. Both factors can be compared in terms of the airflow saving, showing a big save in the activated sludge reactor.

The operation of nitrifying systems with a low DO concentration to obtain nitrite accumulation makes possible a high reduction in aeration supply, even at small nitrite accumulation percentages. This reduction is higher in activated sludge reactors than the airlift reactor with the increase of the nitrite percentage buildup in the systems.

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