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Rate control-based strategy to enhance biological nitrogen removal during anoxic/oxic sludge digestion

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ABSTRACT

Anoxic/oxic (A/O) sludge digestion is a waste-activated sludge (WAS) treatment practice that simultaneously reduces volatile suspended solids reduction and removes nutrients from digested sludge. This study aims to optimize the performance of A/O sludge digestion by achieving the optimum alkalinity and nitrogen balance through rate control. Rate control involves oxic/anoxic cycling based on the activity of WAS (i.e. ammonification, nitrification, oxygen utilization, and denitrification rates) and controlling the processes of amonification, nitrification, oxygen ultilization, and denitrification in WAS. This strategy is different from pH, oxidation-reduction potential, dissolved oxygen controls, or fixed time control methods. Laboratory-scale batch aerobic digestion and A/O digestion tests were performed to verify the feasibility of the approach. The optimum operational cycles for the aeration and mixing were determined as 3.5 and 2.5 h, respectively (6 h full cycle). The sludge reduction in A/O digestion was greater than that in the aerobic digestion (51.83 vs. 48.13%) at a sludge retention time of 23 d. The total nitrogen reduction in A/O digestion was significantly higher than that in the aerobic digestion (65.20 vs. 12.27%). The performance of the proposed rate-control strategy for A/O digestion is superior to those of other experimental control schemes. In addition, this technique produces a sludge dewatering liquor with low nitrogen and phosphorus concentrations.

Keywords: Anoxic/oxic sludge digestion; Waste-activated sludge; Denitrification; Nitrification; Ammonification

1. Introduction

Aerobic digestion and anaerobic sludge digestion are two widely used methods for reducing wasteactivated sludge (WAS) reduction. However, when WAS is stabilized, nitrate or ammonia and phosphorous are produced from sludge mineralization [1]. Aside from sludge reduction, the nutrients produced by this treatment should also be determined. Nitrification, which occurs during aerobic digestion, disrupts the environmental alkalinity and reduces the pH. Furthermore, the relative post-process characteristics, such as instability, dewaterability, and filtrate quality, of the digested sludge deteriorate compared with those of raw waste sludge [2–4]. These properties can be

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improved by using combined anoxic/oxic (A/O) sludge digestion [3,5]. Alternating A/O operations can recover the sludge alkalinity and maintain the neutral pH condition over prolonged anoxic periods; in turn, these processes promote nitrification, improve the sludge reduction efficiency, and enhance the nitrogen removal [3,6]. Denitrification can be incorporated into aerobic sludge digestion either spatially [7] by introducing anoxic zones, or by providing periods of on and off cyclic aeration [8–11].

Numerous studies [6,12,13] have shown that during an A/O cycle, a balanced alkalinity is achieved when the nitric acid produced from the aerobic cycle is completely denitrified during the anoxic cycle. However, most of these studies focused only on sludge destruction in the aerobic phase as well as on the anoxic denitrification for nitrogen removal and alkalinitv neutralization. The overall balanced reactions during A/O digestion are shown in Eqs. (1)-(3). The destruction of volatile suspended solids (VSS) and the production of ammonia during the anoxic phase have not been discussed in previous studies.

A/O endogenous respiration:

 $C_5H_7NO_2 + 4NO_3^- \Rightarrow 5CO_2 + 2N_2 + NH_3 + 4OH^- \quad (1)$

$$C_5H_7NO_2 + 5O_2 \Rightarrow 5CO_2 + 2H_2O + NH_4^+ + OH^-$$
 (2)

$$NH_4^+ + 2O_2 \Rightarrow NO_3^- + H_2O + 2H^+$$
 (3)

According to Al-Ghusain stated [14], the major operational difficulties during A/O digestion include distinguishing the time needed to initiate the aerobic and anoxic periods and the duration of the two periods. Thus, the ratio of the anoxic time to the aerobic time is highly significant in A/O sludge digestion. The anoxic periods should be sufficiently long to consume the nitrate in the mix-liquor produced during the aerobic period, whereas the aerobic period should be adequately long to produce the appropriate amount of nitrate needed for the subsequent denitrification. A considerably long aerobic period would result in nitrate accumulation and decreased pH, whereas a significantly prolonged anoxic period would reduce the pH and result in anaerobic sludge digestion. Thus, it is vital for the development of a reliable control strategy for the A/O system.

Control strategies for determining the optimal cyclic operations for the aerobic/anoxic (A/A) digestion include real-time monitoring of relative parameters such as dissolved oxygen (DO) level [15],

nitrate content [16], oxidation-reduction potential (ORP) [17,18], and pH [14]. Controlling the DO does not reveal the sludge conditions in the reactor nor identify the optimum A/O cycle. Meanwhile, regulating the pH and ORP depends on the surface characteristics of the probe material; moreover, this type of control could not adequately differentiate the ends of the anoxic and aerobic phases [19]. However, the primary purpose of these control strategies is to maintain neutral pH conditions, rather than to reduce VSS, by alternating the aeration. Al-Ghusain et al. [20] compared various A/O lengths and concluded that a 12 h anoxic period and a 12 h aerobic period are optimal for both VSS reduction and nitrogen removal. Al-Ghusain et al. [3] also demonstrated that nitrogen and alkalinity equilibriums occur during A/O digestion. However, the researchers determined the A/O cycle solely from fuzzy cyclic aeration and did not consider the activity of process microbes. Moreover, the group failed to explain the principle of the control process. Thus, the A/O cycle conditions they proposed may not be optimal for achieving efficient removal of VSS and total nitrogen (TN), particularly when used for other WAS sources.

The lengths of the anoxic and aerobic periods are vital in controlling the A/O digestion process. Rate control involves regulating the A/O cycle by monitoring the ammonification, nitrification, endogenous denitrification, or anoxic ammonification rates, as well as the oxygen utilization rate (OUR) during cyclic A/O sludge digestion. The alkalinity balance, nitrogen balance, and maximum sludge removal can be achieved when 75% of the sludge is aerobically digested and the remaining portion is anoxically digested. The ratio is not equal to the ratio of aeration time and anoxic time. All these factors are related to sludge activity: the ammonification and endogenous denitrification are associated with VSS reduction; nitrification and denitrification are related to pH control and alkalinity equilibrium; and aerobic ammonification and nitrification are associated with OUR, which in turn correlates with the DO level during the aerobic period and with the difference between the aeration time and the aerobic time.

The time course of the DO concentration varies in a cyclic fashion as shown in Fig. 1.

If the time of the anoxic phase and that of the oxic phase are assumed to be t_{anoxic} and t_{oxic} , then based on the nitrogen equilibrium, the amount of ammonium nitrogen from aerobic ($dN_{\text{amm}}/dt \times t_{\text{oxic}}$) and anoxic ($1/4 \times dN_{\text{nitrate}}/dt \times t_{\text{anoxic}}$) respiration is equivalent to the amount of nitrate-nitrogen that participates in denitrification ($dN_{\text{nitrate}}/dt \times t_{\text{anixc}}$).



Fig. 1. Variation in the time course of the DO concentration in a cycle.

$$\frac{1/4 \times dN_{\text{nitrate}}/dt \times t_{\text{anoxic}} + dN_{\text{amm}}/dt \times t_{\text{oxic}}}{= dN_{\text{nitrate}}/dt \times t_{\text{anoxic}}}$$
(4)

This cyclic operation time is the sum of the durations of the anoxic and oxic processes:

$$t_{\rm anoxic} + t_{\rm oxic} = t_{\rm cycle} \tag{5}$$

The aeration time is equal to the aerobic digestion time minus the time required for the DO concentration to decrease from its initial point to zero. That is,

$$t_{\text{aeration}} = t_{\text{oxic}} - C_{\text{DO}} / (dC_{\text{DO}} / dt \times \text{VSS})$$
(6)

where $dN_{nitrate}/dt$ is the specific denitrification rate, dN_{amm}/dt is the specific ammonification rate, C_{DO} is the DO concentration in the sludge, dC_{DO}/dt is the specific oxygen utilizing rate, VSS is the VSS concentration in the reactor, and t_{cycle} is the length of an operation cycle.

Then, the A/O time can be calculated by these equations.

In the present research, the principle as well as the details for optimizing the A/O process parameters for nitrogen and sludge removal was determined by controlling the nitrate and alkalinity equilibrium and by determining the biomass activity. This study also aims to compare the performance of the proposed strategy with those of other control schemes by employing batch A/O and aerobic sludge digestion.

2. Material and methods

2.1. Source of experimental sludge

The experimental sludge for the activity analysis and the feed for the A/O digestion and aerobic

digestion were obtained from a full-scale, continuous wastewater treatment plant that uses an anoxic/anaerobic/oxic system to remove organic, nitrogen, and phosphate simultaneously from municipal wastewater. The system, which serves approximately 600,000 population equivalents, includes a grid chamber, primary clarifier, biological treatment tank, and secondary clarifier. The system is operated with a relatively high sludge age (20 d). The secondary sludge sample was harvested from the recycling stream. After passing through a 1.00 mm screen, the sludge was washed twice with pure water and further concentrated prior to use.

2.2. Analysis of WAS activity

The cyclic operation of A/O digestion was determined by the WAS activity, which was monitored using batch measurements of the specific ammonification, nitrification, and denitrification rates and OUR at 25°C according to Standard Methods [21].

The specific ammonification, nitrification, and endogenous denitrification rates and the OUR were determined to be 0.19 mg N/g VSS h, 1.15 mg N/g VSS h, 0.75 mg N/g VSS h, and 4.75 mg DO/g VSS h, respectively. The specific rate of nitrification is considerably higher than that of ammonification. Therefore, the nitrification time was ignored, and nitrification was considered to occur simultaneously with ammonification. Aerobic ammonification and anoxic ammonification were the rate-determining steps.

2.3. A/O and aerobic digestion tests

Two types of digester systems in two 5 L cylindrical stalinite vessels were used. A schematic diagram of the reactors is shown in Fig. 2. The sludge was



Fig. 2. Schematic illustration of the batch digester testers.

continuously mixed by paddles driven by an electric motor. The vented air was introduced to diffusers located at the bottom of the reactor. During the aerobic phase, the DO concentration was automatically maintained at approximately 4 mg/L by a DO controller. The operation cycle of the A/O sludge batch digestion was set to 6 h, and t_{anoxic} and t_{oxic} were set as 1.51 and 4.49 h, respectively, as calculated according to Eqs. (4)-(6). The aeration cycle consisted of 3.5 h of aeration on and 2.5 h of aeration off. The A/O digestion unit was operated in a 6 h cycle that consisted of 2.5 h of mixing and 3.5 h of aeration, whereas the aerobic digestion unit was aerated in full cycle. Both tests' devices were incubated at 25°C and the DO was controlled at approximately 4 mg/L during the aeration phase. The experiments were conducted over a period of 30 d. The aerobic digestion unit was aerated in full cycle and was used as the control set. Evaporation loss was compensated by adding distilled water to the required reactor volume for that experimental stage.

2.4. Analytical methods

For both anoxic periods for A/O sludge digestion and aerobic digestion, subsequent daily samples were simultaneously collected at the end of the anoxic periods to evaluate the digester responses. The total chemical oxygen demand (TCOD), suspended solids (SS), VSS, phosphate, and different forms of nitrogen were determined. The TCOD, SS, VSS, and TN parameters of WAS as well as the soluble chemical oxygen demand (SCOD), ammonia, nitrate, and nitrite parameters of the digested sludge were measured according to the Standard Methods for the Examination of Water and Wastewater [21]. The alkalinity was indirectly analyzed by determining variations of pH value in the reactor. The DO (METTLER, Inpro 6050, Germany) and pH (METTLER, Inpro 4010, Germany) were measured using an online monitoring system.

3. Results

3.1. VSS reduction

In this study, the reductions in SS and VSS were used as critical parameters of sludge digestion. The A/O and aerobic sludge digestion systems yield similar VSS destructions, as demonstrated by the 15 and 16% SS reduction after 5 d (Fig. 3). Afterward, the SS reduction for both systems began to level off. The changes in the VSS and TCOD in both systems were similar to those in the SS reduction. As shown in Fig. 3(a), the TCOD removal for the A/O digestion exhibited a zero-order relationship over time from 0 to 23 d. After 23 d, the VSS and TCOD for both systems exhibited significant reductions as the digestion progressed.

During the A/O digestion, the removal rates of SS, VSS, and TCOD were 34.76, 51.83, and 47.66%, respectively, whereas those during aerobic digestion were 34.03, 48.13, and 40.67%, respectively. The VSS reduction via A/O digestion was more efficient than that via aerobic digestion. This result is consistent with those of previous studies [12,20,22] (32–48 vs. 33%). Novak et al. [23] suggested that certain fractions of biological sludge can only be degraded aerobically, whereas others can only be degraded anaerobically. Therefore, sludge recycling via A/O digestion destroys a larger amount of solids compared with that via aerobic digestion.

3.2. Nutrients released during sludge digestion

The effect of A/O sludge digestion on nitrogen removal was particularly significant in this study. Therefore, the transformation of sludge nitrogen into its form in the liquid (e.g. ammonia, nitrate, nitrite, etc.) requires further investigation. The various forms of nitrogen analyzed during digestion in the two sludge digestion reactors are presented in Fig. 4(a)-(c). At the outset of the test, both reactors had a TN concentration of approximately 1,077 mg/L. The diagrams show the TN of the wet sludge, including solid N, aqueous total Kjeldahl nitrogen, and NO. After 5d of A/O sludge digestion, the TN removal was approximately 27%. Afterward, the TN decreased at the steady rate of 1.92 mg N/g VSS d until the end of the experiments. However, in the aerobic digester, the TN decreased at the lower rate of 0.38 mg N/g VSS d. At the conclusion of the experiment, the TN concentrations in the A/O and aerobic digesters were 448.36 and 965.53 mg/L, respectively. A/O digestion achieved a higher TN efficiency (65.20%) compared with that of aerobic digestion (12.27%). The TN removal efficiency during A/O digestion was significantly higher than those in semicontinuous and continuous A/O digestion processes [22].

In the A/O digester, the organic nitrogen in the sludge was transformed into ammonia via ammonification and then to nitrite and nitrate via nitrification. When the oxic phase changed into the anoxic phase, the nitrogen was removed via denitrification. During the A/O digestion, the highest amount of ammonium that accumulated in the reactor was 5.37 mg/L. Moreover, the nitrite and nitrate concentrations in the supernate were also considerably low prior to 17 d of



Fig. 3. Time course of the variations in the SS, VSS, and TCOD concentrations in the reactors: (a) A/O digestion and (b) aerobic digestion.



Fig. 4. Time course of the variations in the nutrient concentrations in the reactors: (a) nitrate, nitrite, and ammonia during A/O digestion and (b) aerobic digestion (c) TN concentration and (d) soluble phosphate.

operation. These results suggest that the proposed strategy can efficiently remove nitrogen during A/O sludge digestion. However, the sludge activity in terms of the denitrifying performance decreased because of the prolonged starvation time. This prolonged starvation led to nitrate accumulation and reduced pH, which further inhibited nitrification. For the aerobic digester, the initial nitrate concentration rapidly increased with the digestion time because of the continuous aeration and the complete nitrification. At the end of the aerobic digestion, the ammonium and nitrate concentrations reached 212.25 and 385.40 mg/L, respectively.

Even though phosphate could not be removed from the aerobic sludge digestion system (which changes the phosphorous from in the sludge into another form in the liquid), a large amount of phosphorous was released from the sludge into the liquid and caused liquor dewatering with high phosphate concentration. Fig. 4(d) shows the variation in the amount of soluble phosphate released from the sludge over time in the A/O digester. In the presence of bacterial storage substances, such as polyhydroxyalkanoates or glycogen, or polyphosphate particles [24], the cells decayed slowly and phosphate was released gradually during the first 5 d [25,26]. In this study, when the available intracellular substrate was digested in the A/O digester, the phosphate release increased at a rate of 2.50 g P/d from days 5 to 23. Afterward, the rate decreased until the end of the experiment. A similar phase was obtained in the aerobic digester, and the phosphate was released at a rate of 1.80 mg P/d from day 1 to day 17. When the experiment ceased, the phosphate concentration in the A/O digester was higher than that in the aerobic digester (49.84 vs. 30 mg/L). Several factors, such as biomass decay and lysis, pH value, and precipitation [24,27], may have affected the amount of phosphate released during endogenous respiration. The exact reason for this phenomenon will be the subject of further investigation.

Sludge digestion results in the rapid deterioration of cell viability, which is accompanied by rapid increase in the amounts of soluble organic substances. The variation in SCOD in the sludge slurry can indicate cellular degradation and subsequent utilization [23]. The



Fig. 5. Time course of the variation in the SCOD concentration in A/O and aerobic digester.

SCOD profiles for the A/O digester are considerably different from those for the aerobic digester (Fig. 5). In aerobic digestion, the SCOD rapidly increased in 13 d and reached its maximum value of 315.20 mg/L. Afterward, the value decreased rapidly to 84.33 mg/L during the next 4 d. The content then remained at a low level and barely changed. The SCOD level during A/O digestion was considerably lower than that during aerobic sludge digestion and reached its maximum value of 100.57 mg/L on day 10. However, for both reactors, the SCOD levels decreased to a certain degree; this result is similar to that of Schiener et al. [28]. Moreover, this profile is similar to that of the soluble nitrogen release in both reactors. Therefore, the prolonged starvation and low pH in the A/O and aerobic reactors resulted in significant bacterial lysis on days 10 and 13, respectively, and allowed other bacteria in the reactors to utilize the cell fragments as substrates for maintenance or growth. At the end of the experiment, the SCOD in both reactors were similar (80.33 mg/L for A/O and 86.89 mg/L for aerobic digestion). These results indicate that A/O sludge digestion at 17 d of SRT is optimal for reducing the SCOD and simultaneously increasing the digestion efficiency under similar conditions (temperature, VSS concentration, sludge activity, etc.).

3.3. pH profile during digestion

In this study, the pH in the reactor reflects the variation in alkalinity. Alkalinity balance is reached when the pH ranges cyclically and maintains at a neutral level at the end of each anoxic period. Fig. 6(a) shows the pH in the A/O reactor as monitored at 6 h intervals using an appropriately calibrated pH probe. The pH significantly increased in the anoxic phase and was completely neutralized in the oxic phase in each operational cycle. Fig. 6(b) illustrates that the pH ranged from 6.70 to 7.10 and remained neutral during the 17d of A/O digestion. These results indicate that alkalinity balance was reached during the A/O sludge digestion. However, the pH gradually decreased to 6.32 when the SRT increased after day 17. This finding is attributed to the significantly lower decay rate of autotrophic nitrifying bacteria compared with that of heterotrophic bacteria under prolonged starvation conditions. The pH during aerobic digestion significantly decreased from 7.10 to 5.51 after 5 d of digestion and then remained low until the end of the experiment.

As expected, the pH in the A/O system remained higher than that in the aerobic digester (Fig. 6). The difference became more pronounced as digestion time progressed. The alkalinity could have been complemented by the denitrification during the anoxic



Fig. 6. pH profiles in the digestion reactor: variations in the pH (a) during 1 d of A/O sludge digestion and (b) during sludge digestion.

phase of the A/O digestion. By contrast, the alkalinity was not supplemented during aerobic digestion. Consequently, the alkalinity in the aerobic digester continuously decreased because of nitrification, and the measured pH was lower than that in the A/O digester.

4. Discussions

The SRT, temperature, and operational parameters are the main factors that affect aerobic sludge digestion. Al-Ghusain and Hao [14] used the pH as the control parameter for A/A and aerobic sludge digestion at 20 d SRT. A/A digestion resulted in lower VSS reduction (40-45 vs. 54%) and higher TN removal (45-49 vs. 4-8%) compared with the aerobic sludge digestion. However, compared with the fixed-time control (3 h aeration on and 3 h aeration off), the ORP control for A/A sludge digestion performed slightly better in terms of VSS reduction (18.93 vs. 17.72%) and nitrogen removal (21.61 vs. 20.14%) at 10 d SRT [18]. By comparing three fixed A/A cycles, Al-Ghusain et al. [20] reported that a 50% anoxic cycle length is optimal for sludge reduction. The A/O cycle can achieve 43.70% of VSS reduction and 33.70% of TN removal at 30°C and a 10 d SRT. A combined aerobic/ anaerobic sludge digestion, which consisted of anaerobic digestion for 15 d at 35°C and aerobic digestion for 5 d at 32 to 34°C, achieved approximately 62% of VSS reduction at 20 d SRT [11]. Meanwhile, a VSS removal efficiency of 66% was attained by a system with an anaerobic digestion period of 15 d and an aerobic stage of 12 d at 37°C [25]. Daigger and Bailey [7] reported that pre- and post-thickening and a series of aerobic-anoxic tanks achieved 40 to 50% of VSS reduction at 50 d SRT under ambient temperature. In another experiment involving prethickening and a series of aerobic-anoxic tanks, a VSS reduction of 50–70% was achieved at 25 to 32° C and 45 d SRT. In the present study, the rate control-based A/O cycles achieved a VSS reduction of 51.83% and a nitrogen removal of 65.20% at 25° C and 23 d SRT. These results are considerably better than those obtained by pH and ORP control. However, the combination of aerobic/ anaerobic sludge digestion, particularly when aerobic digestion followed anaerobic digestion [7,11,25], achieved relatively higher VSS and nitrogen removal rates. The mechanisms underlying these processes require further investigation.

The results of the proposed strategy should be compared with other pretreatments (such as thermal, chemical, ultrasound, etc.) that enhance aerobic sludge digestion. Compared with the A/O membrane bioreactor, thermo-chemical wastewater pretreatment increased the sludge reduction by approximately 33% [29-31]. Ultrasound pretreatment improved the sludge reduction efficiency to 42.7-55% [32-35]. Merrylin et al. [36] reported a sludge reduction of 52.4% upon the removal of extracellular polymeric substances. Al-Ghusain and Hao [14] used pH control in the A/O sludge digestion and achieved a sludge reduction of 40%. A/O sludge digestion performed as well as aerobic digestion with these pretreatments. Therefore, the proposed strategy may be a cheaper alternative to aeration, ultrasound, thermal, or chemical input treatments.

An appropriate A/O cyclic control strategy, such as pH [14] or ORP control [18], can sufficiently lengthen the anoxic cycles to denitrify excess NO_x –N. However, the use of these strategies in controlling the anoxic cycles may lead to insufficient VSS and TN removal during A/O digestion. Kumar et al. [5] reported that anaerobic/aerobic digestion can remove 90% of ammonia via nitrification/denitrification. However, in the present study, complete nitrification and denitrification occurred during the A/O digestion. At the same time, nitrate accumulation during the aerobic digestion process further resulted in decrease of pH caused by insufficient alkalinity. Then, incomplete nitrification occurred and high concentration of ammonium accumulated in the aerobic digester.

The small amounts of nitrate and nitrite that remained in the A/O reactor at the end of the anoxic period after 17 d of stabilization should be noted. However, the nitrate level in the leaching sludge increased to 21.67 mg/L at the end of the 23 d stabilization period. These phenomena may be attributed to two factors. The non-biodegradable or slowly biodegradable organic substances derived from the decay and lysis of microbes transformed into readily degradable, soluble substrates after hydrolysis; these substances may have been utilized by other bacteria as a substrate for growth or as a proton donor for denitrification [37,38]. Given that denitrification is a zero-order reaction [14], the decrease in the denitrification rate was more likely due to insufficient substrate utilization. Hao et al. [39] investigated the reduced bacterial activity in activated sludge as a result of the cell death or activity decay. Their findings showed that prolonged starvation reduces the activity of WAS, particularly of denitrifying bacteria.

The strategy proposed in this research was appropriate for a 17 d cyclic A/O digestion. However, the specific rates of ammonification, nitrification, and endogenous denitrification as well as the specific OUR of the activated sludge vary depending on the sludge source. Therefore, the ratio of the aeration time to the non-aeration time must be considered based on the sludge source and the DO concentration in the aerobic period. The viability and activity of sludge decrease as digestion time progresses. The biomass decay rates during sludge digestion also affect the operational cycle. Consequently, the proposed control strategy may be more suitable for continuous or semi-continuous A/O digestion of secondary WAS.

5. Conclusions

The experimental results suggest that perfect equilibria of nitrogen and alkalinity are maintained during the A/O digestion of waste sludge. The process can be controlled using a rate-control strategy, which can be used to optimize the cyclic operations of the A/O process. The SS and VSS removal efficiencies of A/O digestion are higher than those of aerobic digestion. The proposed strategy also achieved higher TN removal compared with that of aerobic digestion. In addition, nitrogen did not accumulate during A/O digestion; whereas a significant amount of nitrate was found in the sludge supernate during aerobic digestion. The experimental results demonstrate that the proposed rate-control strategy is more efficient than other controls for cyclic A/O sludge digestion.

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