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Startup operation and process control of a two-stage sequencing batch reactor (TSSBR) for biological nitrogen removal via nitrite

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ABSTRACT

The startup operation and process control of a two-stage sequencing batch reactor (TSSBR) was investigated to improve the efficiencies of organic substrate degradation and nitrification via nitrite from a chemical industrial wastewater with high COD and nitrogen concentrations. A control strategy using process variables as dissolved oxygen (DO), oxidation–reduction potential (ORP) and pH was implemented. The conventional SBR test results showed that based on DO and pH breakpoints at the transition of COD removal and nitrification, organic substrate degradation and nitrification could be separated and occurred in two different reactors termed TSSBR. For the purpose of improving the process flexibility and saving aeration energy, the variations of DO, ORP and pH in TSSBR were characterized. The developed control strategy for TSSBR was that in the SBR1, DO and ORP breakpoints indicated the end of COD removal; in the SBR2, the DO breakpoint and ammonia valley on the pH profile represented the end of nitrification; a nitrate knee on the ORP profile and a nitrate apex on the pH profile indicated the completion of denitrification. A stable nitrite-type nitrification was achieved in the SBR2 with nitrite accumulation rate above 95%. The TSSBR demonstrated an improved organic substrate degradation rate by 40% and nitrification rate by 60% in comparison with conventional SBR. The TSSBR consisting of SBR1 and SBR2 was a two-sludge system, i.e., heterotrophs and autotrophic nitrifiers in the different reactors, which is favorable to improve the treatment efficiency and increase the proportion of nitrifiers in the SBR2 biomass.

Keywords: Two-stage SBR; Nitrite-type nitrification; Nitrogen removal; Process control

1. Introduction

Organic substrate degradation and nitrification is performed by two groups of microorganisms, i.e., heterotrophs and autotrophic nitrifier, respectively. In a biological nitrogen removal process, the nitrifier abundance and activity is a key factor to enhance the nitrification performance. For nitrogen removal, a longer sludge retention time (SRT) is required due to the slow growth rate of nitrifier. However, a high influent COD concentration or high organic loading will promote the growth of heterotrophs and shorten SRT. Thus, there is a SRT conflict in the system treating high strength COD and nitrogen wastewater. Moreover, the previous studies also proved that a relatively lower influent COD concentration is favorable for increasing the proportion of nitrifiers in the biomass. In a carbon-limited autotrophic nitrifying biofilm (rotating disk reactor), the proportion of nitrifying bacteria was about 50% according to fluorescent in-situ hybridization (FISH) [1]. Contrastively, the proportion of nitrifying bacteria in a rotating biological contactor with an influent COD concentration of

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300 mg/L was only about 12.8% [2]. Many processes are modified to increase the SRT and nitrifier abundance, e.g., biofilm process [1–3], two-sludge system [4].

The sequencing batch reactor (SBR) has become increasingly popular in engineering applications for industrial wastewater treatment, such as poultry processing wastewater [5], brewery wastewater [6] and reject water from anaerobic sludge digester [7]. One of advantages of SBR process is its ability to perform multiple biochemical processes in only one tank, e.g., carbon substrate degradation, nitrogen and phosphorus removal [8–11]. One main characteristic of the chemical industrial wastewater is the high COD and nitrogen concentrations. As mentioned above, the higher influent COD concentration will cause nitrification rate to decrease. Using one single SBR for treatment of high strength COD and nitrogen industrial wastewater, it is very difficult for the effluent quality to conform to the discharge standards, especially for nitrogen removal. However, by controlling the operational conditions, organic substrate degradation and nitrification can occur sequentially in the different reactors. The dominant microorganisms grow in respective reactor, which avoids the negative impact of high organic loadings on nitrification and maintains system stability. That is more important to improve the treatment efficiency and effluent quality in spite of the variations of influent characteristics. The previous study investigated the effect of temperature, influent COD concentration and NH_4^+-N concentration on the performance of TSSBR, and proved that TSSBR effectively improved the rates of organic substrate degradation and nitrification with compared to conventional SBR [4].

The drawback of SBR is the complex operation and management with various sequences for carbon, nitrogen and phosphorus removal [12]. The SBR performance and treatment efficiency can be improved if the process control is developed [12–14]. The earliest control method used in SBR was conventional steady-state time procedure control, that is, a prefixed duration of each phase in one cycle. Even now, many SBR engineering applications still use this control strategy. In fact, a SBR system often receives the shock loadings of flow rate and wastewater compositions. In this case, the steady-state time procedure control is difficult to regulate optimal conditions and results in the performance deterioration or plant failure. For the purpose of controlling and optimization of processes, Charpentier et al. used oxidation-reduction potential (ORP) set-points as the process control parameters [15]. However, absolute ORP values are affected by many factors, e.g., pH, temperature, DO concentration and MLSS. Subsequently, some researchers also found that real-time control, using the DO, ORP and pH-time profiles, was more practical and useful for process control of the activated-sludge processes [16–20]. These studies demonstrated that the DO, ORP and pH-time profiles were strongly correlated to organic substrate degradation, nitrification and denitrification. Although the previous study has proved that TSSBR is an efficient process for the treatment of wastewater with high COD and nitrogen concentrations [9], there are no literatures related to startup operation and process control of TSSBR, and how to achieve a two-sludge system.

The objective of this study was twofold. Firstly, the DO, ORP and pH-time profiles during the organic substrate degradation, nitrification and denitrification phases were characterized to provide a direction for the startup operation of TSSBR, i.e., the startup of two-sludge system. Secondly, a process control strategy based on online monitoring of the DO concentration, ORP and pH-time variations was developed to improve the organic substrate degradation and nitrification rates, save aeration energy and to establish a simple and cost-effective TSSBR process.

2. Materials and methods

2.1. Wastewater and seed sludge

The characteristics of the chemical industrial wastewater used as the feed are shown in Table 1. The 144 wastewater samples were measured with the minimum and maximum concentrations. The wastewater was mainly composed of soluble organic acids (acetic acid) and a small amount of aromatic compounds, e.g., benzoic acid, benzoic anhydride, and aromatic hydrocarbons. Sodium hydroxide (NaOH) was added to neutralize the influent. In order to investigate the nitrogen removal performance at the varying influent NH_4^+-N and HCO_3^- concentrations, ammonium chloride (NH_4Cl) of 7–28 g and sodium bicarbonate ($NaHCO_3$) of 10–30 g were added in the influent to change the initial ammonia nitrogen (NH_4^+-N) concentration and bicarbonate alkalinity (calculated as $CaCO_3$).

70% of the seed sludge was obtained from a domestic wastewater treatment plant with conventional nitrogen removal via nitrate. The remaining 30% of the seed sludge with partial nitrification to nitrite was taken from lab-acclimated sludge, which had been operated for one month at 32°C. The temperature range of 30–35°C is favorable to achieve partial nitrification via nitrite due to a

Table 1
Influent wastewater characteristics (mg/L)

Contents	Concentration range
Total COD	400±20–2000±60
Soluble COD	390±15–1960±55
BOD_5	240±9–1400±28
Total nitrogen	53±2–212±6
Ammonia nitrogen	50±1.5–200±5
Total phosphorus	2.5±0.06–15±0.3

higher specific growth rate of ammonia oxidizing bacteria (AOB) than nitrite oxidizing bacteria (NOB) [21]. Therefore, AOB became the dominant nitrifying bacteria with a nitrite accumulation rate above 95%. MLSS concentration in conventional SBR was 3000–3200 mg/L. In the SBR1 and SBR2 of TSSBR, MLSS concentration was 2000–2100 mg/L and 3000–3100 mg/L, respectively.

2.2. Experimental set-up

2.2.1. Conventional sequencing batch reactor (SBR)

A bench scale conventional SBR with the working volume of 38 L was used to investigate the startup of TSSBR, i.e., one-sludge SBR transferred to two-sludge TSSBR. Air diffusers were placed at the bottom of the reactors for oxygen supply, and airflow meters controlled the aeration rate to achieve the desired DO concentration. A mechanical stirrer was used to provide mixing during the anoxic phase. Temperature sensors and electric heaters were used to maintain the wastewater temperature at 30±2°C and pH throughout the nitrification was maintained at 7.5–8.0, which is the optimal temperature and pH range for growth of AOB to achieve nitrogen removal via nitrite [21]. At the temperature below 25°C, nitrification process tends towards the complete oxidation of ammonia to nitrate rather than nitrite-type nitrification [22]. Sludge retention time (SRT) was about 18d calculated as the following Eq. (1):

$$\mathrm{SRT} = \frac{VX}{\left(V_w X_w / \mathrm{cycle}\right) \times \left(\mathrm{cycle/d}\right)} \quad (1)$$

where V = reactor volume, L; X = concentration of MLSS in reactor, mg/L; V_w = waste sludge volume, L; X_w = concentration of MLSS in waste sludge, mg/L.

2.2.2. Two-stage sequencing batch reactor (TSSBR)

A laboratory-scale TSSBR consisting of two reactors (SBR1 and SBR2) was operated with fed-batch sequences for the organic substrate and nitrogen removal (Fig. 1). The working volume of each reactor was 38 L. Airflow meters controlled the aeration rate to achieve the desired DO concentration. A mechanical stirrer was used to provide mixing during the anoxic phase in SBR2. Temperature sensors and electric heaters were used to maintain the wastewater temperature at 30±2°C and pH throughout the nitrification was maintained at 7.5–8.0. The SRT in SBR1 and SBR2 was 8 d and 45 d, respectively, which was also calculated by Eq. (1).

2.3. Experimental procedure

One cycle of conventional SBR was operated as the following procedures: 1) influent loading of 1 min; 2) aeration for COD removal and nitritation, and the aerobic duration of each cycle is different depending on the influent COD and nitrogen concentrations; 3) anoxic agitation for denitritation, and the anoxic duration of each cycle is varying depending on the NO_x^--N concentrations from nitritation; 4) sludge settling of 30 min; 5) effluent discharge of 5 min. The batch time of each cycle was not constant due to on-line regulating of aerobic and anoxic durations based on different influent composition, e.g., varying influent COD and NH_4^+-N concentrations.

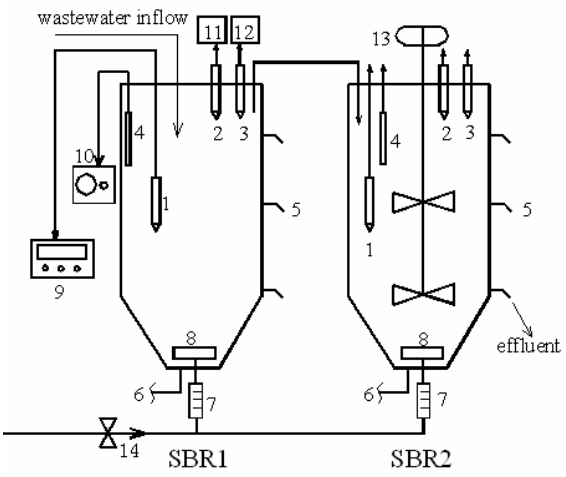

Fig. 1. TSSBR system. 1 ORP probe; 2 DO probe; 3 pH probe; 4 temperature sensor; 5 outlet; 6 waste sludge; 7 airflow meter; 8 diffusers; 9 ORP meter; 10 temperature controller; 11 DO meter; 12 pH meter; 13 stirrer; 14 compressed air.

The operational patterns of one TSSBR cycle are shown in Fig. 2. Most of the organic substrate was removed in the SBR1 under aerobic condition. After then, transferring of the effluent from the SBR1 to the second reactor (SBR2) occurred in sequence. The SBR2 was operated under aerobic condition for simultaneous nitrite-type nitrification and removal of a small amount of residual organic substrate. Then denitrification occurred under anoxic condition. The durations of aerobic phase in SBR1 and aerobic, anoxic phases in SBR2 were varying to accommodate the different influent COD and nitrogen concentrations. As the explanation in conventional SBR, the batch time of TSSBR each cycle was not constant due to on-line regulating of aerobic and anoxic durations based on different influent composition. Acetate was used as the external carbon sources for denitrification in the SBR2.

The experimental period including 3 phases is shown in Table 2. The seed sludge was firstly acclimated in a conventional SBR to achieve a stable performance of organic substrate degradation and nitrogen removal via nitrite. During this operational period of 20 d, DO, ORP and pH were characterized. Then based on DO, ORP and pH-time profiles, organic substrate degradation and nitrification was separated, and two-sludge TSSBR was started up. Lastly, a control strategy using DO, ORP and

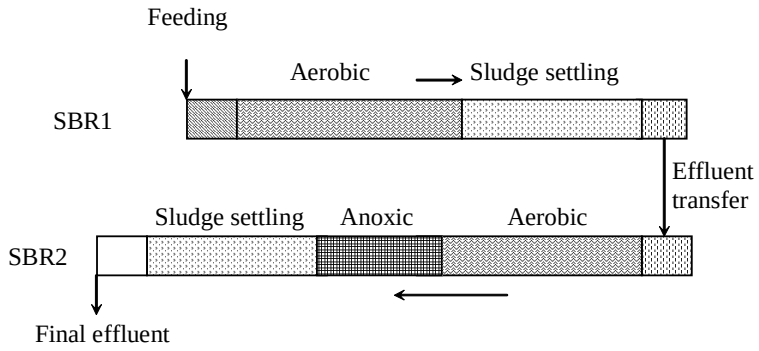

Fig. 2. Operational patterns of one TSSBR cycle.

Table 2
Experimental phases

Phase	Duration	Process	Contents	Objectives
1	20 d	Conventional SBR	Achieve stable performance of COD and nitrogen removal; characterize DO, ORP and pH variations	Provide a direction for TSSBR startup
2	21 d	TSSBR	Based on DO, ORP and pH-time profiles in SBR, one-sludge SBR was converted to two-sludge TSSBR	Successful startup of TSSBR.
3	30 d	TSSBR	Characterize DO, ORP and pH-time variations in TSSBR	Establish control strategy for TSSBR operation

pH as process variables was established for stable performance of TSSBR.

2.4. Analytical methods

The DO concentration was continuously measured in the conventional SBR and TSSBR using an YSI 5739 oxygen probe connected to a transmitter (YSI 52B). Continuous monitoring of the pH and ORP was carried out using pHS-3C meters with a pH probe E-201 and an ORP electrode E-414Q.

Samples for COD, NH_4^+-N, nitrite nitrogen (NO_2^--N), nitrate nitrogen (NO_3^--N), alkalinity analysis were directly collected from the reactor. The concentrations of MLSS, COD, total nitrogen, NH_4^+-N, NO_2^--N, NO_3^--N and alkalinity were measured according to Standard Methods [23].

3. Results and discussion

3.1. Conventional SBR performance and characteristics of process variables

In a conventional SBR, heterotrophs for organic substrate degradation and autotrophic nitrifiers grow in only one reactor. The key problem concerning TSSBR startup is to find an effective method achieving the separation of heterotrophs and autotrophic nitrifiers, i.e., one-sludge conventional SBR converted to two-sludge TSSBR. Consequently, the DO, ORP and pH variations in a conventional SBR were investigated to provide a direction for the startup operation of TSSBR.

In the organic substrate degradation, aerobic nitrification and anoxic denitrification of one typical cycle, the DO, ORP and pH-time variations and wastewater quality dynamics are shown in Figs. 3–4 with an initial COD concentration of 500 mg/L and NH_4^+-N concentration of 60 mg/L. The cycle time at this condition was 176 min including the following sequences: influent feeding of 1min, organic substrate degradation and nitrification of 120 min, denitrification of 20 min, sludge-settling of 30 min and effluent discharge of 5 min. Because this research focused on organic substrate degradation and nitrification-denitrification, the phases of feeding, sludge-settling and effluent discharge was insignificant, which were not included in the figures.

As shown in Figs. 3–4, organic substrate degradation first occurred at 0–40 min. The slow decrease of ammonia nitrogen was mainly resulted from biosynthesis of heterotrophic bacteria rather than nitrification. Very few nitrites below 2 mg/L was produced, and thus nitrification should be negligible. During this period, DO was relatively stable, meanwhile ORP and pH gradually in-

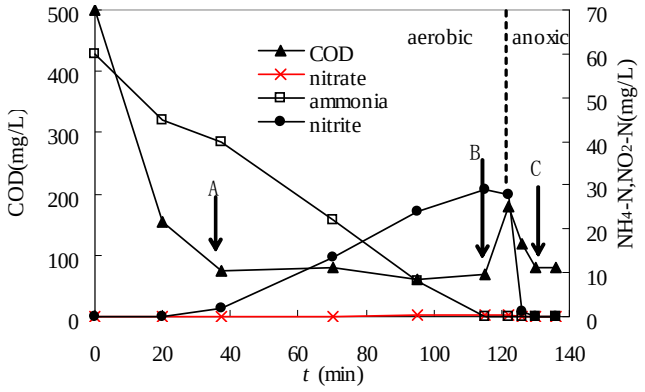

Fig. 3. COD, NH_4^+-N, NO_2-N variations in SBR.

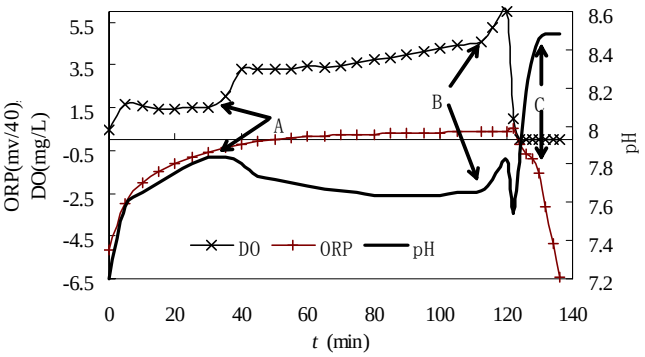

Fig. 4. DO, ORP, pH variations in SBR.

creased. When organic substrate was not further utilized, DO showed a strong increase (arrow A in Fig. 4) due to a quick decline of the oxygen uptake rate (OUR). Thereafter air-on continued and nitrification began. During nitrification, DO increased slowly as well as the pH changed from its previously increasing state to a decrease because of the consumption of alkalinity by nitrifying bacteria. A breakpoint (arrow A in Fig. 4) was detected on the pH profile. Because the DO rising rapidly and the breakpoint on the pH curve at the transition of organic substrate degradation and nitrification identified the sequence of these two biological reactions in a conventional SBR process, it is feasible to separate two biological reactions and acculturated the dominant microorganisms in different reactors.

The effluent qualities during the phase 1 of 20 d are shown in Table 3. After one week, the stable performance of COD and nitrogen removal was achieved.

3.2. Startup operation of TSSBR based on conventional SBR

According to the DO and pH-time variations in the conventional SBR, aeration was shut down at the 40 min as soon as DO increased quickly and the breakpoint on the pH profile was monitored, corresponding to the detection of arrow A at the 38 min in Fig. 4. Then after quiescent sludge settling, transfer of the effluent from this reactor (SBR1) to the second reactor (SBR2) occurred. As shown in Fig. 3, at the 40 min COD and NH_4^+-N concentration was 75 mg/L and 40 mg/L, respectively, with a C/N ratio of 1.9:1. Therefore, the effluent from SBR1 at the 40 min has a character of low COD and high NH_4^+-N concentrations. The conditions being favorable for the nitrifiers were maintained in SBR2, e.g., the temperature was set to 30±2°C and the DO was above 2 mg/L. The pH was kept at a proper level to assure the bicarbonate alkalinity (HCO_3^-) sufficient for nitrification. With a relative lower influent COD concentration below 100 mg/L, the SRT in SBR2 was 45 d, which was long enough for growth of nitrifiers due to their lower specific growth rates. Aeration was not shut down until the end of NH_4^+-N oxidizing to nitrite, and then anoxic denitrification occurred. With the above operation, organic substrate degradation and nitrification was separated as well as SBR2 start-up for nitrification-denitrification was achieved.

In phase 2, the variations of COD, NH_4^+-N and NO_x-N in SBR1 influent and effluent during 21 d operational period (one cycle per day and total 21 cycles) are shown in Fig. 5. As shown in Fig. 5, with 15 d under the above operational conditions, most of organic substrate was removed in SBR1 and no NO_x^--N was detected in the SBR1 effluent, which verified that nitrification did not occur in SBR1. The ammonia removal efficiency of approximately 50% occurred in SBR1 as a result of biosynthesis of heterotrophic bacteria. With the high strength COD influent, the heterotrophs were more competitive and dominant. The rapid reproduction of heterotrophs caused the percentage of autotrophic nitrifiers in biomass to decrease. When COD concentration was decreased to a lower level, aeration was shut down and nitrification could not continue. As a result, the residual nitrifying bacteria in SBR1 were gradually washed out with

Table 3
The effluent qualities during phase 1 of 20 d (mg/L)

	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20
COD	102	98	95	90	87	80	75	75	70	72	70	68	65	62	60	58	60	63	61	59
NH_4^+-N	22	19	15	11	6	4.3	2	1	1.5	1.2	1.0	0.8	0.5	0.9	0.7	0	0	0	0	0
TN	29	24	19.3	14	9.3	6.7	4	1.5	1.8	1.5	1.4	1	1	0.9	0.7	0	0	0	0	0

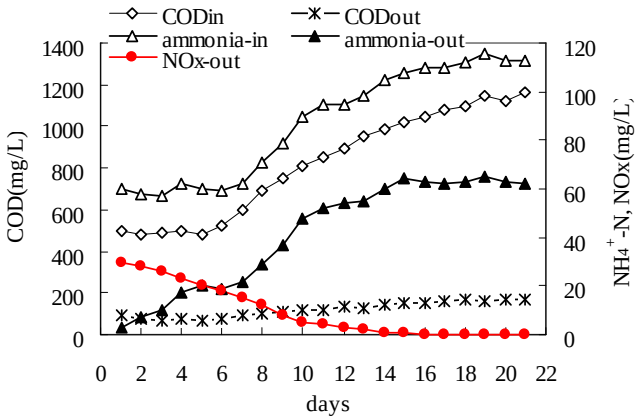

Fig. 5. Variations of COD, NH_4^+-N and NO_x-N in the SBR1 influent and effluent during phase 2.

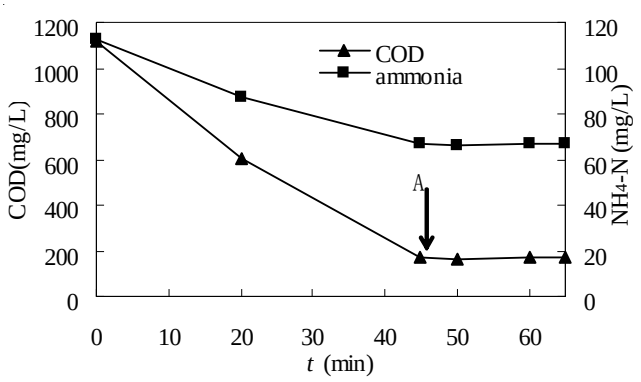

Fig. 6. COD and NH_4^+-N variations in SBR1.

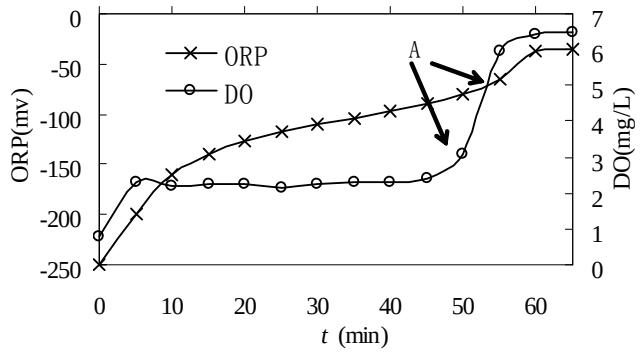

Fig. 7. DO and ORP variations in SBR1.

the increase of waste sludge in two weeks. SBR2 was mainly for nitrogen removal; moreover, a small amount of COD that was left in SBR1 effluent was also removed to make a further reduction in the final effluent COD. The rates of organic substrate biodegradation and nitrification were obviously improved after two biological reactions were successfully separated and characteristic microorganisms grew in different reactors. With the influent COD concentration of 1150 mg/L and NH_4^+-N of 105 mg/L, the specific organic substrate degradation rate and specific nitrification rate in the TSSBR was 8.7 kg COD/kg MLSS.d and 0.31 kg NH_4^+-N/kg MLSS.d, respectively, which is obviously higher than those in the conventional SBR; the corresponding rate in the conventional SBR was 6.1 kg COD/kg MLSS.d and 0.19 kg NH_4^+-N/kg MLSS.d, respectively.

3.3. Process control of TSSBR system

After the successful startup operation of TSSBR, the key problem concerning the engineering application of TSSBR was process control of organic substrate degradation, nitrification and denitrification. The characteristic variations of DO, ORP and pH corresponding to the dynamics of COD, NH_4^+-N, NO_2-N and NO_3-N during one typical TSSBR cycle are shown in Figs. 6–9 (SBR1 in Figs. 6–7 and SBR2 in Figs. 8–9), which provided a basis for the real-time control of TSSBR process. The initial COD and NH_4^+-N concentrations were 1120 mg/L and 113 mg/L, respectively.

3.3.1. Process control of SBR1 for organic substrate degradation

Figs. 6–7 show the experimental results in SBR1. Ammonia nitrogen was removed by microbial assimilation during organic substrate degradation. No NO_x^--N was detected in the SBR1 effluent. The ratio of utilized COD to removed NH_4^+-N was about 100:5. DO concentration maintained stable, meanwhile ORP increased gradually due to the oxidizing of organic substrate. At the end of organic substrate degradation (arrow A in Fig. 6), DO increased suddenly and quickly (arrow A in Fig. 7) as a result of rapid decline of the OUR. The DO rising caused a strong increase on the ORP profile (arrow A in Fig. 7). Consequently, the significant breakpoints on the DO and ORP profiles signified the transition of organic substrate degradation in SBR1 and nitrification in SBR2, and could be used as process control information to terminate aeration in SBR1.

3.3.2. Process control of partial nitrification to nitrite in SBR2

Figs. 8–9 show the experimental results in SBR2. The effluent from SBR1 was fed into SBR2 and aeration was continued. During nitrification, DO concentration gradually increased because the nitrification rate and OUR of nitrifying bacteria descended with the decrease of NH_4^+-N concentration. When NH_4^+-N was essentially exhausted, the beginning of endogeneous respiration of nitrifying bacteria caused DO a rapid increase identifying the end of nitrification (arrow B in Fig. 9). The pH value changed from its slowly decreasing during nitrification to a sudden increase at the end of nitrification,

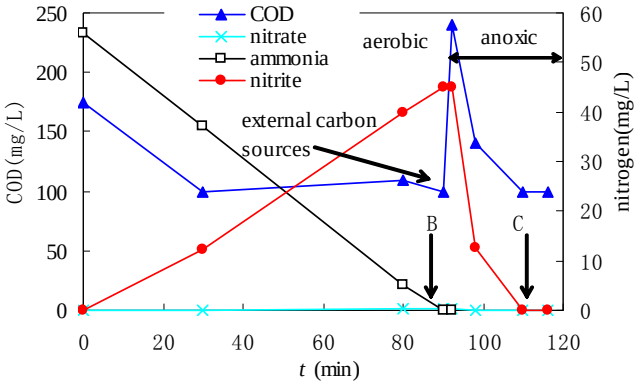

Fig. 8. COD, nitrogen variations in SBR2.

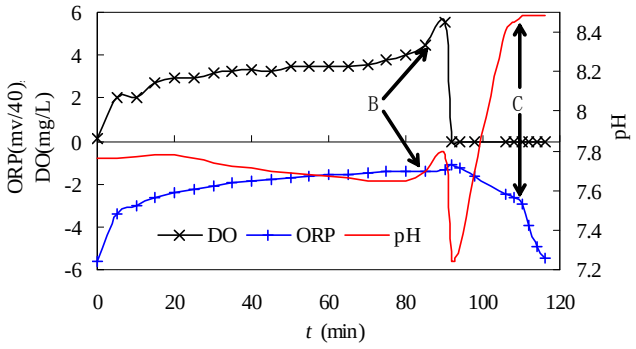

Fig. 9. DO, ORP, pH variations in SBR2.

termed ammonia valley (arrow B in Fig. 9). Consumption of alkalinity and production of H^+ caused pH to decrease slowly during nitrification. After the end of nitrification, CO_2-stripping produced by extended aeration resulted in pH increase [24]. Therefore, breakpoints on the DO and pH profiles (arrow B in Fig. 9) indicated the end of ammonia oxidizing to nitrite and could be used for process control of aerobic duration in SBR2. As can be seen in Figs. 3–4, in a conventional SBR process the DO and pH data showed the same variations (arrow B) at the end of nitrification. If a plant is operated with DO set point control, the only control option for nitrification would be the identified pH breakpoint because DO set point control would mask the breakpoint in the DO profile.

The aerobic duration in SBR2 was controlled, based on the on-line monitoring of the DO and pH breakpoints, and not based on the fixed hydraulic retention time (HRT). In fact, nitrification was incomplete. Ammonia oxidation to nitrite by AOB was complete, but nitrite oxidation to nitrate by NOB (nitrite oxidizing bacteria) was incomplete. If aeration was extended, the accumulated nitrite would be oxidized to nitrate and the levels of NOB would increase in SBR2. Therefore, by the process control strategy and temperature set to 30±2°C, aeration was terminated as soon as NH_4^+-N levels were essentially exhausted, which inhibited the growth of NOB. After TSSBR system had been operated for one month under the above conditions, the average nitrite accumulation rate (NO_2-N/ NO_x-N) was above 95%, which indicated the successful performance of partial nitrification to nitrite (Fig. 8).

3.3.3. Process control of denitrification in SBR2

With denitritation occurring, ORP significantly dropped because aeration stopped and system converted from the aerobic condition to the anoxic respiration. During denitritation, ORP gradually descended with nitrite reduced to nitrogen gas. When NO_x^--N in the system was completely removed, the system shifted into a true anaerobic condition resulting in a further strong descent on the ORP profile. A nitrate knee (arrow C in Fig. 9) on the ORP profile indicated the end of denitritation. The pH gradually increased due to the yielding of alkalinity during denitrification, and then maintained constant after the end of denitrification. A nitrate apex (arrow C in Fig. 9) on the pH profile was detected, corresponding to the nitrate knee on the ORP profile. Consequently, both ORP and pH variations could be used for real-time control of anoxic duration in SBR2 [25,26]. As can be seen in Figs. 3–4, in a conventional SBR process the ORP and pH data showed the same variations (arrow C) at the end of denitrification.

4. Conclusions

Based on the breakpoints on the DO and pH-time profiles during the transition of organic substrate biodegradation and nitrification in conventional SBR, two biochemical reactions were separated and occurred sequentially in two different reactors. The start-up operation of TSSBR system was successfully achieved by identification of DO and pH characteristic variations, which avoided the negative impact of higher influent COD concentration on nitrification and the treatment efficiency was improved, especially nitrification rate was obviously improved.

The process control strategy for TSSBR operation based on online monitoring of the DO concentration, ORP and pH-time variations was developed. The results demonstrated that the real-time control strategy effectively controlled organic substrate degradation, nitrification and denitrification. It was significant to stabilize the effluent quality and save energy. Moreover, by the real-time control strategy and controlling temperature at 30±2°C, partial nitrification to nitrite was successfully performed. The operational pattern (Fig. 2) and real-time control strategy provide universal principles applying to all the relevant processes for the control and optimization of biological systems.

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