Ammonia and phenol removal in an internal-circulate sequencing batch airlift reactor

Qifeng Liu, Cong Du, Ting Yang and Zhimin Fu

ABSTRACT

An internal-circulate sequencing batch airlift reactor (IC-SBAR) has been developed to evaluate the efficiency of phenol and ammonia removal in treating synthetic wastewater. This study examined the effect of operation cycle on this system. Results showed that above 97.0% removal efficiencies of ammonia and phenol were achieved, which indicated that ammonia and phenol removals were not related to operation cycle. The average removal efficiency of 91.7% for chemical oxygen demand (COD) was achieved when the static/aerobic/settling time was 240 min/440 min/40 min. It was found that COD removal efficiency increased due to the time of operation cycle being prolonged. The average removal efficiencies of total inorganic nitrogen (TIN) were 65.8, 69.3 and 68.9% when average TIN concentrations were 78.0, 97.6 and 88.4 mg/L, respectively, in the influent. A cycle study showed that most phenol was degraded by aerobic microbes. Increasing the static time from 120 to 240 min resulted in the accumulation of NO_2^--N, which indicated that the structures of the nitrifying bacterial community were changed.

Key words | IC-SBAR, nitrogen removal, operation cycle, phenol removal

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INTRODUCTION

Several industrial processes such as petroleum refinement, coking (Kim *et al.* 2013), petrochemicals manufacturing (Yang & Jin 2012), and paints and resins production release wastewaters containing both ammonium and phenolic compounds (Milia *et al.* 2012). Phenol and its derivatives are either toxic or lethal to fish at concentrations of 5–25 mg/L. Therefore, it has been listed on the water priority control pollutants blacklist by many countries including China and the USA (Shi *et al.* 2013), due to its toxicity. Various methods of removing phenol in industrial wastewater have been studied such as chemical advanced oxidation (Sano *et al.* 2007), active carbon adsorption (Liu *et al.* 2010) and microbial degradation (Al-Khalid & El-Naas 2012). Among them, microbial degradation, due to complete mineralization and low cost in operation and maintenance, is more environmentally friendly than physico-chemical processes.

During the past decade, the biodegradation of phenol and its derivatives by bacteria has been extensively studied and a large number of phenol-degrading bacteria, such as *Rhodococcus* sp. (Shen *et al.* 2004) and *Pseudomonas* sp. (Polymenakou & Stephanou 2005) have been isolated and characterized at different levels. Usually, the phenol

biodegradation process requires the presence of molecular oxygen to initiate enzymatic attack on the aromatic rings (Al-Khalid & El-Naas 2012).

Ammonia nitrogen contributes to eutrophication and it becomes toxic for the fauna in waters bodies. It can be removed by a biological treatment process. Biological nitrogen removal (BNR) requires the oxidation of ammonia to nitrate through nitrification and the reduction of nitrate to nitrogen gas through denitrification. The nitrification process is performed in two consecutive steps, ammonia oxidation and nitrite oxidation, by chemolithoautotrophic bacteria, ammonia-oxidizing bacteria (AOB) and nitrite-oxidizing bacteria (NOB). Research showed that the nitrification of a full-scale coke manufacturing wastewater treatment plant had been gradually suppressed by the influent phenol concentrations (about 400 mg/L) (Kim *et al.* 2013). A reactor fed at 54 mg C/(L d) of phenol (99 mg C/L) caused a diminishing on the ammonium removal efficiency, from 91 to 77% (Pérez-González *et al.* 2012). The extent of phenol inhibition was correlated with ammonia oxidation rates, with a greater percent inhibition observed with higher initial rates of oxidation (Lauchnor *et al.* 2011;

Lauchnor & Semprini 2013). The effects of phenol on AOB activity have been studied previously. However, the effect of phenol on the BNR process in a sequencing batch reactor (SBR) has not been investigated.

The SBR is considered as the superior method to degrade the pollutants of organic nitrogen and carbon among various biological wastewater treatments methods (Ganigué *et al.* 2012, Gabarró *et al.* 2012). The airlift reactor, usually utilized to treat wastewater using aerobic granular biomass (Jemaat *et al.* 2014), contains a down-comer zone and a riser zone. In this study, a new reactor, named internal-circulate sequencing batch airlift reactor (IC-SBAR) was developed. This reactor was an airlift reactor operated in batches. There are three stages in the treatment process: static/aerobic/settling. In the static stage, first the inlet valve opens and the reactor is filled in 10 min, then the inlet valve closes and no mixing is provided. This stage is aimed at forming an anoxic environment. Aeration of the mixed liquor is performed during the second stage by transferring air into an air disperser fixed to the bottom of the reactor. No aeration or mixing is provided in the third stage and the settling of suspended solids starts. At the end of the third stage, the outlet valve opens and the 'clean' supernatant liquor exits the tank. The laboratory-scale experiment was conducted to evaluate the removal efficiencies of chemical oxygen demand (COD), phenol, NH_4^+-N and total inorganic nitrogen (TIN) in an IC-SBAR. The results will serve to assess the feasibility of IC-SBAR for the simultaneous TIN removal and phenol removal for BNR of complex industrial wastewaters.

METHODS

Experimental setup and operation

A Plexiglas IC-SBAR reactor with a working volume of 2.3 L was utilized in this study (Figure 1). The internal diameter of the down-comer was 0.06 m. The riser had a height of 0.6 m and an internal diameter of 0.04 m, and it was at 0.05 m from the bottom of the down-comer. The IC-SBAR reactor
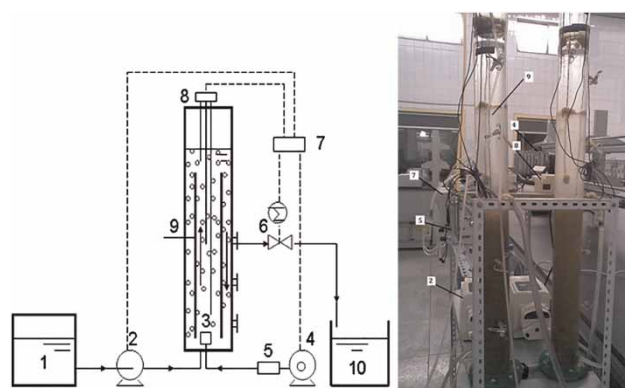

Figure 1 | Schematic diagram of the experimental system. (1) Influent tank, (2) peristaltic pump, (3) air disperser, (4) air pump, (5) gas-flow meter, (6) electromagnetic valve, (7) time controller, (8) liquid indicator, (9) inner pipe, (10) effluent tank.

was inoculated with activated sludge from an anoxic–oxic process treating wastewater for simultaneous carbon and nitrogen removal (Xinxinban, Hohhot). Data were measured after 1-month cultivation.

The operation period consisted of three different phases (Table 1). In phase I, IC-SBAR (0–16 d) was operated sequentially with a cycle time of 8 h, which included static for 120 min (including 10 min of substrate filling), aerobic for 340 min, and settling for 20 min (including 5 min of effluent withdraw). In phase II (17–32 d), the operation cycle was extended to 12 h, including static for 120 min (including 10 min of substrate filling), aerobic for 560 min, and settling for 40 min (including 5 min of effluent withdraw). In phase III (33–51 d), the operation cycle time was 12 h, while the static time was extended to 240 min, and the aerobic stage decreased to 440 min.

Influent was supplied by a peristaltic pump (BT100, Baoding). Air was supplied through a disperser at the reactor bottom with an airflow rate of 30 L/h, which was measured by a glass gas-flow meter (Changzhou, LZB-3WB). Effluent was discharged at 0.48 m height from the bottom of the reactor. The volume exchange ratio was 54%. All experiments were performed at room temperature (20–25 °C). No excess sludge was removed from the system during the operation. The sludge concentration was about 3,300–3,500 mg/L. Figure 1 illustrates the experimental apparatus of the laboratory-scale IC-SBAR installation.

Table 1 | Components and operation conditions of different phases

Phase	Operation days	COD (mg/L)	Phenol (mg/L)	NH₄Cl (mg N/L)	COD/N	Cycle (h)	Operation cycles: static/aerobic/settling (total)
I	0–16 (16)	1,233.3 ± 250.3	133.7 ± 36.0	78.0 ± 11.6	15.8	8	120 min/340 min/20 min (480 min, 8 h)
II	17–32 (15)	1,510.1 ± 132.7	123.5 ± 29.8	97.6 ± 19.4	15.5	12	120 min/560 min/40 min (720 min, 12 h)
III	33–51 (19)	1,047.6 ± 332.4	109.4 ± 2	88.4 ± 13.9	11.9	12	240 min/440 min/40 min (720 min, 12 h)

Synthetic wastewater

In this study, the IC-SBAR was fed with synthetic wastewater, which contained organic carbon (in the form of glucose and phenol), NH_4^+-N (in the form of NH_4Cl), KH_2PO_4 and $NaHCO_3$. The influent concentrations were about 1,000 mg COD/L (0.76 g/L glucose and 0.1 g/L phenol), 100 mg NH_4^+-N/L (0.38 g/L NH_4Cl), 20 mg phosphorus (PO_4^{3-}-P)/L (87.7 mg/L KH_2PO_4) and 700 mg $NaHCO_3$/L. The synthetic wastewater was prepared at room temperature (20–25 °C) and stored for no more than 1 day. Table 1 shows the components of the wastewater and operation conditions of different phases.

Analysis methods

Concentration of COD was measured by the fast airtight catalytic decomposition method (CEPA 2002, p. 216). NH_4^+-N was measured by the Nash reagent photometric method (CEPA 2002, p. 279). NO_2^--N was measured by N-(1-naphthyl)-ethylenediamine spectrophotometry (CEPA 2002, p. 271) and NO_3^--N was measured by phenol disulfonic acid spectrophotometry (CEPA 2002, p. 259). TIN was the sum of NH_4^+-N, NO_2^--N, and NO_3^--N. In addition, pH was measured using a pH meter (Thermo Orion 868, USA). The phenol analysis was performed using the 4-aminoantipyrine colorimetric method (CEPA 2002, p. 460).

RESULTS AND DISCUSSION

COD and phenol removal efficiency

The average COD concentrations in the influent and effluent, during the whole operation period, with different operation cycle and COD/N ratios are shown in Figure 2. During phase I, the influent COD concentration increased from about 1,100 to 1,600 mg/L to evaluate the reactor COD removal performance. Data showed that the effluent COD concentration increased from 198.2 to 604.6 mg/L, which indicates that the COD could not be effectively removed under such operation parameters. Therefore, in phase II, the operation cycle time was extended from 8 to 12 h to increase the COD removal efficiency. It was obvious that the effluent COD concentration decreased from 600 to 100 mg/L. In phase III, the influent COD load was about 1.2 g/L d, and the average removal efficiency of COD was 91.9%.
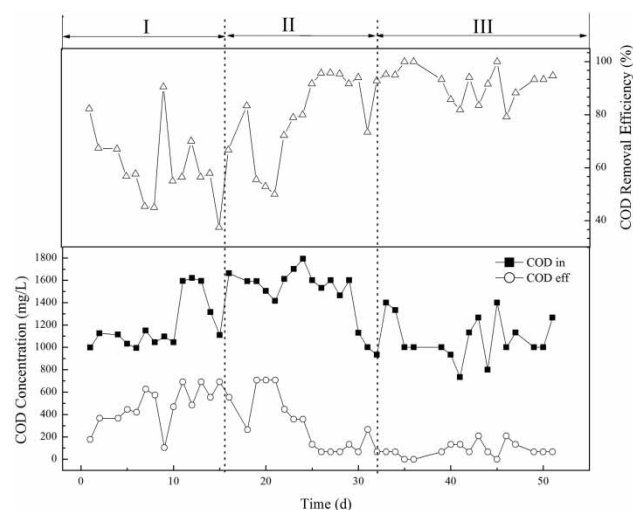

Figure 2 | Variations of COD concentration and COD removal efficiency during experiment.

For the experiment operation period, the average influent and effluent phenol concentrations are shown in Figure 3. The corresponding average influent phenol load was 0.19 g/(L d). These results indicated that the system could provide a consistent high efficiency of phenol removal. In addition, phenol removal was not related to the operation cycle and COD/N ratios. Saravanan et al. (2008) reported that an internal-loop airlift bioreactor could degrade phenol in 6 h with an influent concentration of 100 mg/L. The removal performance of COD and phenol in phase III was better than for a moving bed biofilm reactor (Li et al. 2011) and an anaerobic–anoxic–oxic membrane bioreactor (Kim et al. 2013). A possible reason is that the synthetic wastewater contains easily degraded glucose, while the industrial
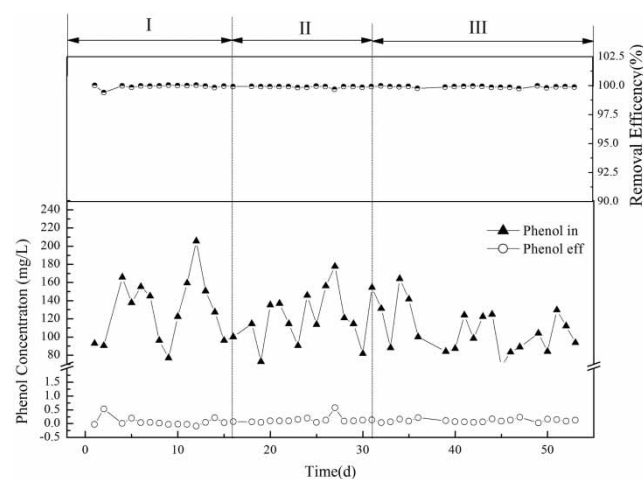

Figure 3 | Variations of phenol concentration and phenol removal efficiency during experiment.

wastewater did not. Therefore, further research on industrial wastewater treatment should be developed.

NH_4^+-N and TIN removal efficiency

For the experiment operation period, the average influent and effluent NH_4^+-N concentrations are listed in Tables 1 and 2 and Figure 4. The removed NH_4^+-N concentration increased from 77.4 (phase I) to 97.0 mg/L (phase II), which indicated that increasing the reaction time is favorable to raise the nitrification reaction. Furthermore, decreasing aeration time from 560 to 440 min (phase II to phase III) led to the effluent NH_4^+-N concentration increase, indicating that the capacity of resisting impact load decreased.

During the whole operation, the removal efficiency of TIN was not as good as NH_4^+-N removal (Figure 5); some NOx^--N accumulation was observed in the effluent, which indicated that the products from nitrification could not be completely denitrified to nitrogen gas. In phase I, the average NOx^--N in effluent was 26.1 mg/L, most of which was NO_3^--N. The average TIN removal efficiency was 64.3%.

In phase II, the average concentration of NOx^--N was 30.1 mg/L in the effluent. The average TIN removal efficiency reached 66.8%. The average removed concentrations of TIN increased from 50.8 to 64.8 mg/L. The results show that extending operation cycle time was favorable to TIN removal.

In phase III, the TIN removal was separated into two parts: during several initial operation days (22–29 d, Figure 5) of phase III the effluent NOx^--N was NO_3^--N, while in the second part (after 29 d) NO_2^--N appeared in the effluent. During initial days (22–29 d), the average TIN concentrations in influent and effluent were 83.3 and 35.1 mg/L; the NH_4^+-N, NO_2^--N and NO_3^--N concentrations were 1.5 mg/L, 0.6 mg/L and 33.0 mg/L in the effluent, respectively; and the average removal efficiency of TIN was 57.9%. After 29 days, the average TIN concentrations were 90.5 and 32.7 mg/L in influent and effluent, respectively, and the average removal efficiency of TIN increased to 63.9%. Researchers had reported that a COD/N ratio

above 10 should be maintained for a nutrient removal efficiency of approximately 90% (Choi *et al.* 2008). In this experiment, the COD/N ratio was above 10, but about 259.4 mg COD was provided by phenol (the theoretical value of 2.4 mg COD/mg phenol). Therefore, the ratio of available COD and NH_4^+-N is lower than 10. The TIN removal efficiency decreased comparing with phase II, which may be due to the decrease of COD/N ratio in the influent.

TIN removal performance increased significantly when NO_2^--N accumulated in the IC-SBAR. There are three possible reasons for this phenomenon. Firstly, the aeration time was decreased from 560 to 440 min in phase III, which indicates that anoxic time was prolonged. In the conventional BNR process, the NO_2^--N and NO_3^--N are produced by nitrifiers under aerobic condition. Then, denitrifiers utilize biological organic substrate as electron donor, and NO_2^--N and NO_3^--N as electron acceptor, to produce nitrogen gas under anoxic conditions. Therefore, decreased aeration time is beneficial for the denitrification process. Secondly, NO_2^--N appears to be the preferred electron acceptor from a biochemical point of view (Moreno-Vivian *et al.* 1999). The maximum specific denitrification rate of NO_2^--N was 0.0275 h^{-1}, which is at the higher level of the range reported in the literature for complete denitrification of NO_3^--N (0.0083–0.0333 h^{-1}) (Gao *et al.* 2012). Furthermore, unlike traditional nitrification, the shortcut nitrification process does not require further oxidization of NO_2^--N to NO_3^--N, which means that up to 25% of the oxygen consumed for nitrification and 40% of the carbon source for denitrification could be saved (Fux *et al.* 2002). Hence, the TIN removal performance improved when NO_2^--N accumulated under the same COD/N ratio in the influent.

Experimental cycle

A cycle study was carried out in order to explore the mechanism of organic matter and TIN removal. Figure 6(a) shows the typical profiles obtained in phase II of the cycle study. During the static stage (0–2 h), phenol concentration

Table 2 │ The summary of different phases

Phase	*n*	COD (mg/L)[a]	Phenol (mg/L)	NH_4^+-N (mg/L)[a]	COD (g/(L d))[a]	Phenol (g/(L d))	NH_4^+-N (g/(L d))	COD (%)	Phenol (%)	NH_4^+-N (%)
I	16	481.6 ± 177.5	0.07 ± 0.15	0.58 ± 0.31	2.0	0.22	0.13	60.8	99.95	99.25
II	15	310.9 ± 248.3	0.14 ± 0.13	0.64 ± 0.40	1.6	0.13	0.11	79.3	99.89	99.34
III	19	93.4 ± 60.2	0.11 ± 0.06	2.03 ± 3.64	1.2	0.12	0.10	91.9	99.90	97.22

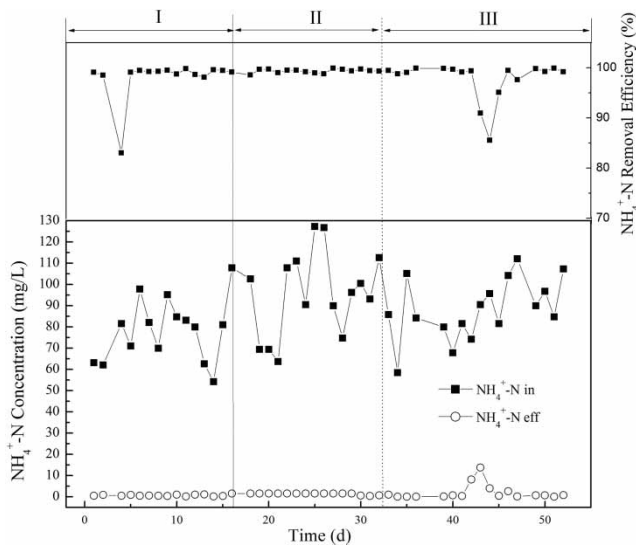*n*: number of analyzed samples; [a]concentration ± standard deviation.

Figure 4 | Variations of NH$_4^+$-N concentration and NH$_4^+$-N removal efficiency during experiment.
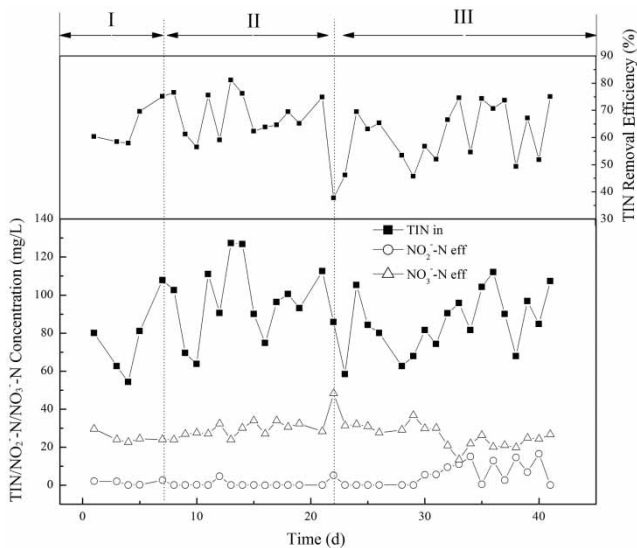

Figure 5 | Variations of TIN concentration and TIN removal efficiency during experiment.

decreased sharply at 0.5 h, then it increased and fluctuated at about 50 mg/L, indicating that phenol was not consumed by heterotrophic organisms. The variation of NH$_4^+$-N concentration was similar to that of phenol. At the end of the static stage it decreased slightly from 48.7 mg/L (0 h) to 42.3 mg/L (2 h). The NO$_3^-$-N concentration increased in the first 0.5 h and then decreased rapidly from 25.0 to 6.9 mg/L; the NO$_3^-$-N reduction rate calculated by Equation (1), was 12.1 mg/(L h), in the last 1.5 h. NO$_3^-$-N decrease was accompanied by NO$_2^-$-N formation; during the operation time, the NO$_2^-$-N concentration increased from

2.4 to 7.3 mg/L. Then, the removed TIN concentration was 14.4 mg/L with a TIN removed rate of 6.7 mg/L h. Results showed that the denitrification process was uncompleted: 36.6% NO$_2^-$-N was kept, indicating that the static time should be prolonged to increased the TIN removal.

$$NO_3^- \text{-N reduction rate} = \frac{(NO_3^- \text{-N})_{t1} - (NO_3^- \text{-N})_{t2}}{t2 - t1} \qquad (1)$$

$$\eta(\text{percent of contribution}) = \frac{(\Delta TIN)_{static} - (\Delta TIN)_{aeration}}{\Delta TIN_{total}} \times 100\% \qquad (2)$$

$$TIN \text{ removed rate} = \frac{(TIN)_{t1} - (TIN)_{t2}}{t2 - t1} \qquad (3)$$

In the aerobic phase, the phenol decreased from 47.2 to 4.9 mg/L after aeration for 1.5 h (2–3.5 h). NH$_4^+$-N increased sharply from 46.4 to 63.7 mg/L, while the NO$_3^-$-N decreased to 2.4 mg/L, at the initial 1.5 h. It was speculated that the presentation of phenol inhibited the activity of nitrifier. It was reported that NH$_4^+$-N oxidation by *Nitrosomonas europaea*, an ammonia-oxidizing bacterium prevalent in wastewater treatment, was inhibited in the presence of phenol (Lauchnor & Semprini 2013). During aeration 1.5–4.5 h (3.5–6.5 h), the NH$_4^+$-N decreased from 63.7 to 1.9 mg/L while NO$_3^-$-N increased from 1.6 to 36.5 mg/L, and the NO$_2^-$-N decreased slightly from 8.9 to 7.7 mg/L. It was obvious that the processes of phenol oxidation, NH$_4^+$-N nitrification to NO$_3^-$-N, and NO$_2^-$-N oxidation occurred in sequence. In total, 41.0 mg/L NH$_4^+$-N and 7.3 mg/L NO$_2^-$-N were removed while 35.7 mg/L NO$_3^-$-N was accumulated. During the aeration period, 12.6 mg/L TIN was removed. In the IC-SBAR, the rising pipe was the aerobic zone and the down pipe was the anoxic zone. Therefore, the NO$_X^-$-N formed in the nitrification process could be denitrified to nitrogen gas. In phase II of the cycle study, both the static phase and the aerobic phase contributed to the nitrogen removal process, with the percent contribution (calculated by Equation (3)) being 53.2 and 46.8%.

Figure 6(b) shows the typical profiles obtained in phase III of the cycle study when the static operation time was prolonged from 2 to 4 h. During the static stage (0–4 h), phenol concentration first decreased then increased and fluctuated at about 60 mg/L. The result shows that prolonging the static time did not benefit the phenol's anoxic degradation. The concentration of NH$_4^+$-N fluctuated at about 50 mg/L. The NO$_3^-$-N
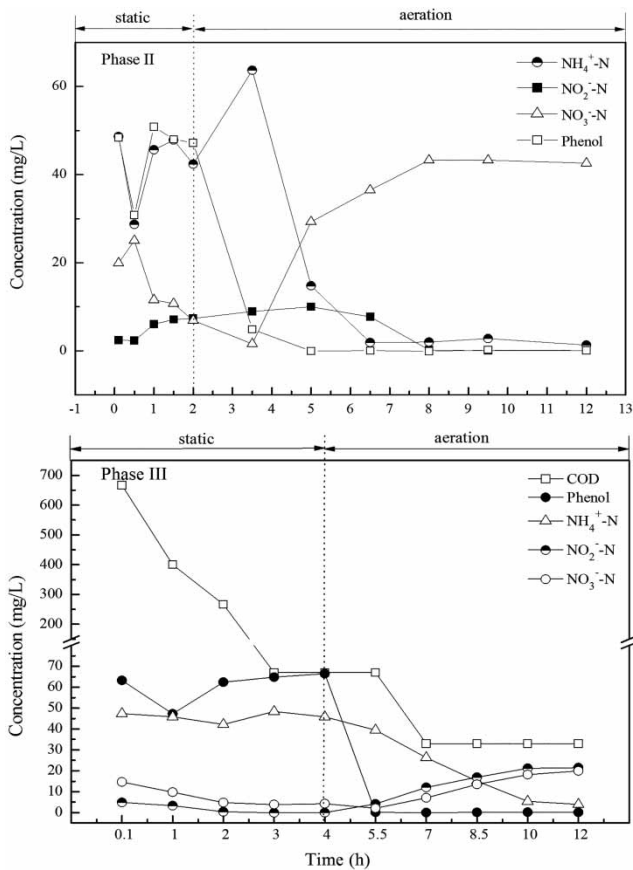
Figure 6 | Typical profiles of cycle study: phase II (top), B phase III (bottom).

and NO_2^--N concentration decreased in first 2 h from 14.6 and 4.8 mg/L to 4.8 and 0.4 mg/L, respectively; the NO_3^--N and NO_2^--N reduction rate was 4.9 mg/L h and 2.2 mg/L h, respectively, in the first 2 h. No NO_2^--N accumulation occurred during the static time. The removed TIN concentration was 15.1 mg/L and the TIN removed rate calculated by Equation (3) was 3.8 mg/L h. Results showed that the denitrification process was almost completed, which indicates that prolonging the static time increased the TIN removal process. However, the NO_3^--N reduction rate as well as the TIN removal rate was lower than that in phase II of the cycle study.

In the aerobic phase, the phenol decreased from 66.5 to 0.1 mg/L after aeration for 1.5 h (4–5.5 h), and the degradation rate was higher than that of phase II. NH_4^+-N decreased from 45.8 mg/L to 4.0 mg/L, while the NO_2^--N and NO_3^--N increased to 21.5 mg/L and 19.9 mg/L, respectively. NH_4^+-N oxidation rate increased obviously after phenol decreased. Similar results were found in phase II. However, the NH_4^+-N oxidation rate decreased significantly. It was speculated that the accumulation of NO_2^--N inhibited the activity of NH_4^+-N oxidation bacteria.

It was obvious that the processes of phenol oxidation, NH_4^+-N nitrification to NO_2^--N, and NO_2^--N oxidation occurred in sequence. It was different from the cycle of phase II. Both AOB and NOB activity decreased in phase III. In total, during the aeration period, 4.7 mg/L TIN was removed, with the percent contribution being 21.9%. Compared with phase II, the aeration period contribution of TIN decreased. It was speculated that the reaction time decreased and the organic carbon was in short supply. The denitrification process required organic carbon as electron donor. Mateju *et al.* (1992) reported that the stoichiometric ratio for denitrification, considering biomass formation, was 4.2 g COD/g N. In phase III of the cycle study, most of the COD was removed in the static stage and the COD/N ratio of the initial aerobic stage was 1.34, which was very low. In the aerobic stage, the average removed COD was 34.0 mg/L, while the average nitrogen concentration removed was 4.7 mg/L. The removed COD/N ratio in the aerobic stage was 7.25, indicating sufficient electron donor for the denitrification process, based on the presumption that the removed COD was totally utilized as electron donor.

CONCLUSIONS

Nutrient removal performance of the IC-SBAR system was influenced by the operation time and influent COD/N ratio. During the operation, the influent COD load changed between 2.7 g/(L d) and 0.8 g/(L d) and the removed COD load changed between 0.7 g/(L d) and 1.8 g/(L d). The average influent phenol and NH_4^+-N load were 0.19 g/(L d) and 0.11 g/(L d) with a high removal efficiency above 90%. The removal of phenol and NH_4^+-N was not related to operation cycle and COD/N ratios. Data indicated that prolonging the cycle time from 480 to 720 min was beneficial for the COD and TIN removal performance. The cycle study showed that phenol was removed by aerobic oxidation. The nitrification process occurred after the phenol degradation. After increasing the static time from 120 to 240 min, the nitrifying bacteria community presented low NOB activity; meanwhile, the nitrification and denitrification activity decreased significantly.

ACKNOWLEDGEMENTS

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