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Effects of chemical phosphate precipitation in the sidestream process on biological phosphorus removal at the anaerobic stage in an anaerobic—aerobic sequencing batch reactor

Jinghua Lv, Linjiang Yuan*

School of Environmental and Municipal Engineering, Xi'an University of Architecture and Technology, Xi'an, Shaanxi 710055, P.R. China, Tel. +86 29 82201354; Fax: +86 29 82201354; email: yuanlinjiang@xauat.edu.cn

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ABSTRACT

This study investigates the effects of chemical phosphate precipitation on the performance of a sequencing batch reactor (SBR) operated in anaerobic—aerobic mode. Supernatants enriched with 50% phosphorus were removed from the SBR (i.e. sidestream) system at the end of the anaerobic stage and were placed into a reaction-sedimentation basin to recover the phosphorus. Results indicate that phosphorus removal efficiency increased from 95.26 to 97.22% during the first 40 d and then declined over the next 20 d. Intracellular polyphosphate (poly-P) storage remained low during sidestream stripping. The sludge was not dominated by organisms that accumulate poly-P, as confirmed by the low anaerobic $\text{phosphorus}_{\text{release}}/\text{HAc}_{\text{uptake}}$ ratios, the phosphorus content in the sludge, and the high ratios of volatile suspended solids of mixed liquor to suspended solids of mixed liquor. These results imply the possibility of changes in population structure given the low level of intracellular poly-P storage during long-term stripping. Intracellular poly-P content may be considered a factor that affects biological phosphorus removal in the future work.

Keywords: Chemical precipitate; Phosphorus removal; Sidestream stripping; Polyphosphate-accumulating organisms (PAOs); Polyphosphate

1. Introduction

Phosphorus (P) is an important non-renewable resource. Much P exists in municipal wastewater and it is a main cause of eutrophication [1] in China. To limit P discharge in effluents from municipal wastewater treatment plants in China, discharge requirements are increasingly stringent.

P can be removed from wastewater by chemical removal, biological treatment, or a combination of both. As an alternative to chemical removal, biological processes are cost-effective and environment-friendly. Biological phosphorus removal from wastewaters is dependent upon the polyphosphate accumulating organisms (PAOs) under particular operational conditions by accumulating P in cells in the form of polyphosphate (poly-P) granules. This accumulation is beyond the normal metabolic requirements of these organisms. Therefore, net P is removed from the wastewater through the discharge of waste-activated sludge with high poly-P content [2–5]. However, the system of enhanced biological phosphorus removal (EBPR) can be unstable and unreliable; thus, meeting discharge regulations can be difficult. P recovery can

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*Corresponding author.

effectively remove P gradually. To meet the P requirements in effluent, the biological treatment must be supported by chemical precipitation if the chemical oxygen demand (COD)/P ratio is low or if the concentrations of nitrogen and P are high in the influent [6,7]. Thus, increasing numbers of wastewater treatment plants integrate chemical precipitates into biological treatment [8–10].

P recovery is mainly used to treat excess sludge and in the stream of return sludge in the system of biological P removal. Supernatants from disposed excess sludge are typically treated further in wastewater treatment plants, and this process increases the load on treatment plants, as well as the total costs of wastewater treatment [11–13]. Another alternative to P recovery involves sidestream removal from the anaerobic part of the activated sludge [14]. A portion of the supernatants enriched with P is removed from mainstream biological treatment by chemical precipitation. As a result, the phosphate load on the processes of biological treatment and waste sludge disposal is alleviated, and P can be recovered. In the sidestream process, P removal is much lower than the requirements for acetate in a mainstream process [15].

The sidestream process combines both biological and chemical P removal processes [16]. The first example of this is the Phostrip process [8]. The biological-chemical P and N removal (BCFS) process [8] was developed to enhance the efficiency of the combined processes of chemical and biological P removal. Barat [17] reported that the BCFS process can meet effluent requirements and maximize the release and recovery of anaerobic phosphate. However, the process of stripping P affects nitrification, and poly-P concentration in the sludge decreases. This phenomenon can induce significant problems in the treatment of downstream sludge. Szpyrkowicz [7] noted that the fraction of poly-P in the sludge decreases as a result of lime dosing; however, the enzymatic system of the biological luxury uptake is reversible when chemical dosing is stopped. In Hao's study [18], P concentration in the effluent was not affected by regular P recovery. Biological P activity may have been negatively affected by the reduced efficiency of P removal, which was in turn induced by the stimulation of dynamic inflow.

Previous studies suggest that P recovery from the anaerobic portion of the activated sludge is beneficial and stimulates the removal of nutrients by a biological treatment system to a certain degree. Nevertheless, the potential negative effects of P recovery on a biological treatment system must be investigated further. In this study, therefore, a sequencing batch reactor operated in anaerobic—aerobic mode (AO-SBR) is adopted, and a portion of supernatants enriched with P is stripped for phosphate recovery in the anaerobic stage. This study examines the effects of sidestream P recovery on the system of biological P removal in the above-mentioned operating mode.

2. Materials and methods

2.1. Experimental setup

The experiments were conducted in an AO-SBR under a working volume of 10 L. The SBR system operated continuously with a cycle time of 8 h (filling: 1 min, anaerobic: 120 min, aerobic: 210 min, settling: 135 min, and drawing: 15 min) under a temperature of 20 ± 1℃. After each cycle, 5 L of the effluent was exchanged with 5 L of influent. The resultant hydraulic retention time was 16 h. The sludge was removed from the reactor at the end of the aerobic phase to control solid retention time (SRT) for approximately 11 d. Oxygen concentration in the aerobic phase was maintained at above 2 mg/L. System pH, which fluctuated between 7.1 and 8.3, was recorded as it is. A schematic diagram of the experimental setup is depicted in Fig. 1.

2.2. Experimental procedure

The experiments were conducted in two operational periods, each of which lasts for 60 d.

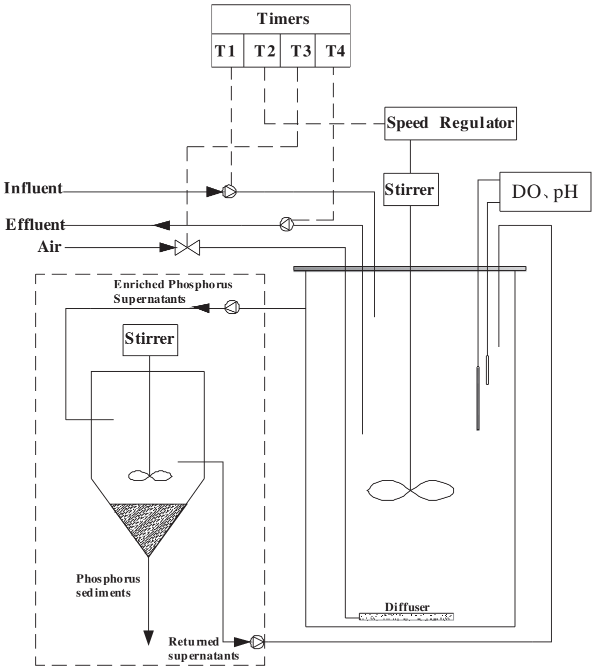

Fig. 1. Schematic of the SBR system with sidestream chemical precipitation.

(I) The sidestream stripping phase lasted from 1 to 60 d. Five liters of supernatants enriched with P were separated from the SBR system and placed into a chemical basin for chemical P precipitation (ferric chloride was used as a coagulant) after a settling phase of 30 min at the end of the anaerobic stage. After chemical precipitation, the clear supernatants on top of the basin were returned to the SBR for the subsequent aerobic stage. With chemical precipitation, the average efficiency of P removal exceeded 96%, and no more than 1 mg/L residual ferric was returned to the SBR system to prevent the ferric iron from affecting the biological treatment as reported in previous studies [16,19–23].

(II) The recovery phase started upon the termination of the stripping operation, that is, on the 61st day, and it lasted until the 120th day. This phase determines whether the AO process can replenish its P removal capacity without stripping.

Hydrochloric acid (HAc), COD, orthophosphate (PO_4^{3-}-P), and pH were measured in samples collected every 15 or 30 min during detailed cycles. The P content in the sludge was monitored every 3 d. At the end of the aerobic phase, total phosphate (P_T), mixed liquor suspended solids (MLSS), and mixed liquor volatile suspended solids (MLVSS) were measured. Once a day, coagulation/flocculation experiments were conducted. The samples of sludge and liquid were taken simultaneously. For most samples, replicate analyses were carried out.

2.3. Wastewater composition

Synthetic wastewater containing sodium acetate (organism cultures in activated sludge), ammonia, and P were pumped into the reactor in the first minute of the anaerobic treatment. The reactor contained the following at the beginning of the cycle: CH_3COONa (400 mg/L as COD basis), NH_4Cl (20 mg/L as NH_4^+-N basis), KH_2PO_4 (10 mg/L as PO_4^{3-}-P basis), 180 mg/L of $MgSO_4 \cdot 7H_2O$, 14 mg/L of $CaCl_2 \cdot 2H_2O$, and 0.5 mL of nutrient solution per liter. Each liter of the nutrient solution consisted of the following compounds [24]: 10 g of ethylenediaminetetraacetic acid, 1.54 g of $FeSO_4 \cdot 7H_2O$, 0.15 g of H_3BO_3, 0.03 g of $CuSO_4 \cdot 5H_2O$, 0.18 g of KI, 0.12 g of $MnCl_2 \cdot 4H_2O$, 0.06 g of $Na_2MoO_4 \cdot 2H_2O$, 0.12 g of $ZnSO_4 \cdot 7H_2O$, and 0.15 g of $CoCl_2 \cdot 6H_2O$.

2.4. Analytical methods

PO_4^{3-}-P, COD, MLSS, MLVSS, and sludge volume index (SVI) were all analyzed according to standard methods [25]. Dissolved oxygen was measured using HACH-HQ40d (USA). Once a week, the activated sludge was microscopically analyzed using an OLYMPUS-BX51 microscope.

To determine the total iron, samples were immediately filtered using a 0.22 µm filter membrane and then acidized by HAc (1 M) prior to atomic absorption spectroscopy. P_T was converted to PO_4^{3-}-P through digestion with potassium persulfate; then, the method for PO_4^{3-}-P determination was applied. The SVI value of the sludge mixture, which evaluates the settleability and compressibility of the sample, was measured using a 100 mL cylinder after 30 min of settlement.

The optimal condition of phosphate precipitation was determined with the aid of Design-Expert 8.0 software. The molar ratio of ferric iron to phosphate is 1.40:1, and stirring intensity is 275 r/min with 30 s and 60 r/min with 18 min for coagulation and flocculation, respectively. Precipitation time is 20 min.

3. Results and discussion

The laboratory-scale SBR utilized synthetic wastewater as the feed for the anaerobic—aerobic cycles. During the sidestream P stripping experiments, the biomass activity was stabilized. The average P removal efficiency exceeded 95.26%, and the P level in the effluent was 0.37 mg/L. The PAO phenotype was clearly displayed. Then, the sidestream stripping experiments were conducted under similar operational conditions.

3.1. Studies of the system cycle

3.1.1. P release and uptake ratios

Table 1 summarizes the rates and ratios obtained on different days in the two periods. P release rate declined gradually from the 1st d to the 40th d under anaerobic conditions until sidestream stripping was terminated. This decline accompanied the reduced phosphate concentration in the anaerobic stage during Period I (Fig. 2(a)). As a result of sidestream stripping, intracellular poly-P storage decreased. At the end of the aerobic phase, P was completely removed in all tests except in those conducted on the 40th d. In this case, P release declined sharply and immediately. Therefore, the P released in the anaerobic stage was 18.81 mg/L. The concomitant uptake of aerobic P was incomplete, given the high P concentration of 3.27 mg/L in the effluent (Fig. 2(a)).

Nevertheless, the uptake rate of aerobic P increased in the nine cycles of the first three days.

Table 1
Variations in P release and uptake rates and $P_{release}/HAc_{uptake}$ in different periods

Time (d)	0	3	9	15	21	40	71	84	96	102	114
Anaerobic P release rate (mg/g VSS/h)	24.13	11.36	10	9.45	5.01	2.78	1.76	1.82	3.01	2.61	4.40
Aerobic P uptake rate (mg/g VSS/h)	13.69	16.18	11.41	8.87	8.93	1.20	1.29	2.06	3.36	5.56	6.91
$P_{release}/HAc_{uptake}$ (P mmol/C mmol)	0.41	0.46	0.16	0.12	0.11	0.05	0.03	0.05	0.10	0.10	0.20

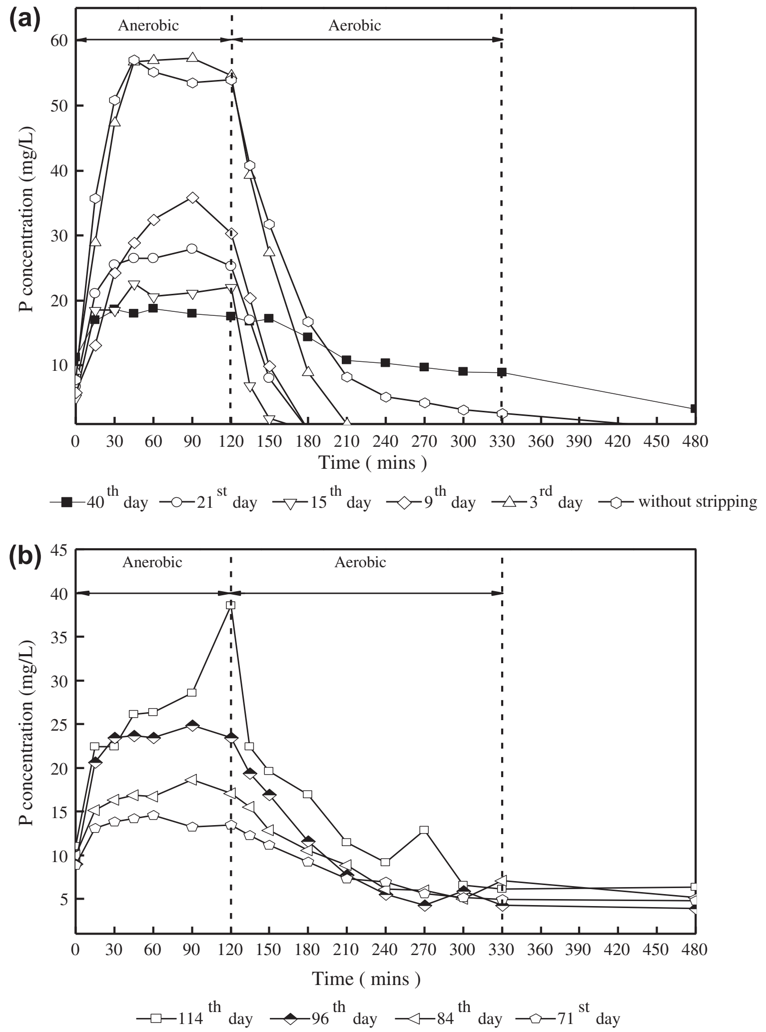

Fig. 2. Cycles of P transformation during the different periods.

Biological P removal improved to some extent in the early days of Period I; thus, additional phosphate accumulated in the system to satisfy the demand for excessive P absorption. This trend may be a short-term adaptive response to sidestream stripping to maintain the energy balance in cells. For every three cycles, 150 mg of P was added to the SBR system in the influent. In the following two cycles, the stripped P was quickly replenished during short-term stripping. The P in the sludge maintained its balance, and biological removal preceded chemical precipitation. The additional P was thus removed by the interaction of biological and chemical effects.

This study examined whether the AO-SBR could restore P removal activity in Period II when sidestream stripping was terminated. According to Fig. 2(b), the uptake rate of aerobic P was significantly lower than that of the process without sidestream stripping. The average P concentration was 5.18 ± 1.11 mg/L in the effluent. This value indicates that biological P removal was ineffective even after side-stream stripping had been terminated for 60 d. Furthermore, the amount of anaerobic P released in the liquid gradually increased as the P release rate increased (Table 1). Moreover, the uptake rate of aerobic P on the 114th d was half that of the process without sidestream stripping approximately 60 d after stripping was terminated. The deterioration in P removal suggests that PAO activity was reduced because of P stripping.

3.1.2. Anaerobic $P_{release}/HAc_{uptake}$ ratio

The ratio of anaerobic $P_{release}/HAc_{uptake}$ is a good indicator of the relative populations of PAOs and of glycogen-accumulating organisms (GAOs) [26–29]. Increased proportions of PAOs can maximize the amount of P released per acetate obtained. An increase in GAOs in the sludge can lower the uptake ratio of P release to acetate because less P is transformed for the same amount of acetate taken up. The factors affecting $P_{release}/HAc_{uptake}$ ratio include pH [30,31], SRT [32], and temperature [33,34]. During the experiments, the temperature remained constant at 20 ± 1°C, and the SRT was controlled for approximately 11 d, which is beyond the range that affects the competition between PAOs and GAOs. In this competition, PAOs are advantageous over GAOs at $pH > 7.0$. Acetate uptake rate remained nearly constant between 7.0 and 8.5 [31,35]. In this study, pH fluctuated between 7.1 and 8.3. Thus, the $P_{release}/HAc_{uptake}$ ratio can reflect the relative levels of PAOs and GAOs in the proposed system.

Without sidestream operation, the $P_{release}/HAc_{uptake}$ ratio of the sludge was 0.41 P mmol/HAc C mmol, which is within the range of values (0.37–0.50 P mmol/C mmol) reported in the studies on PAO metabolic models [36–38]. This value indicates an almost complete uptake of aerobic P (PO_4^{3-}-P concentration in the effluent < 0.5 mg/L). After nine cycles with sidestream stripping, P removal improved on the third day of Period I, with a high $P_{release}/HAc_{uptake}$ ratio of 0.46. On the contrary, P release was low and HAc uptake increased from the 40th d to the 60th d of Period I. The resultant $P_{release}/HAc_{uptake}$ ratio, 0.05, is lower than the theoretically predicted ratios. This finding suggests that the GAOs in the sludge competed for HAc utilization but did not contribute to P release. EBPR activity deteriorated during Period I, and the $P_{release}/HAc_{uptake}$ ratios ranged from 0.46 to 0.05. These values are in accordance with the cycles of P transformation on the 3rd, 9th, 21st, and 40th d.

Throughout Period II, similar operational conditions were maintained in the system without stripping for 60 d. However, the high P concentration in the effluent and biological P removal was unchanged. In this period, the $P_{release}/HAc_{uptake}$ ratio was distinctly low (0.03–0.20 P mmol/C mmol). This range of values is similar to the values reported in studies on reactors with an abundance of GAOs [5].

The diminished Poly-P content and the decreased ratio of anaerobic $P_{release}/HAc_{uptake}$, indicated that the sludge was not dominated by PAOs [27,28,39–41]. Thus, the results with sidestream stripping suggest that the possibility of population shift from the PAO phenotype to the GAO phenotype.

3.2. P and COD removal

The reactor was monitored daily by measuring the COD and the P in the effluent. Fig. 3 shows the phosphate and COD concentrations in the effluent of the AO-SBR during the two periods. In Period I, the average COD concentration in the effluent was approximately 29.01 ± 5.60 mg/L, with a removal efficiency of 85.70 ± 3.15%. The corresponding values for Period II were 31.22 ± 5.33 mg/L and 83.58 ± 3.46%. In this period, the overall efficiency of COD removal was maintained at 87.08 ± 3.26% in the AO-SBR system without sidestream stripping. The level of COD in the effluent was maintained at 30.12 ± 5.55 mg/L in both periods, thus indicating that unlike the AO-SBR system, sidestream stripping had little effect on COD removal. The results suggest that the effect of such operating conditions on COD removal is insignificant.

By contrast, P removal improved slightly in the first 40 d of Period I. The average efficiency of P removal increased from 95.26 to 97.22%. The level of P in the effluent was 0.14 ± 0.16 mg/L, which shows that sidestream stripping benefited the biological treatment to some extent. The supernatants that were eliminated from the coagulation/flocculation process were returned to the AO-SBR for the downstream biological process. The chemical precipitates could not mix with the activated sludge because they were isolated from the basin and were thus recovered as either iron phosphate or its compounds. Therefore, half of the anaerobic supernatants enriched with P were removed as chemical precipitants. Given these findings, sidestream stripping is thus a potential method of P recovery.

Despite a lack of operational error, EBPR efficiency deteriorated suddenly after 40 d of stripping. In this situation, P concentration in the effluent also increased to 5.36 ± 1.27 mg/L. During Period II, the system continued to operate poorly given the same influent

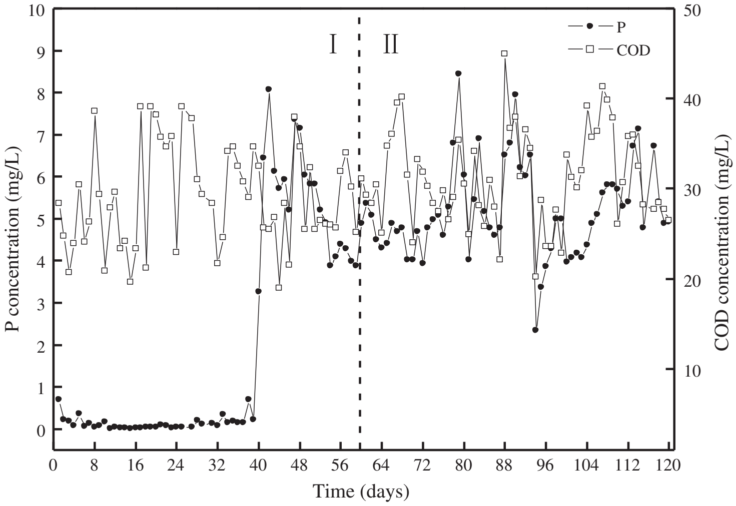

Fig. 3. Profile changes in effluent P and COD at various periods.

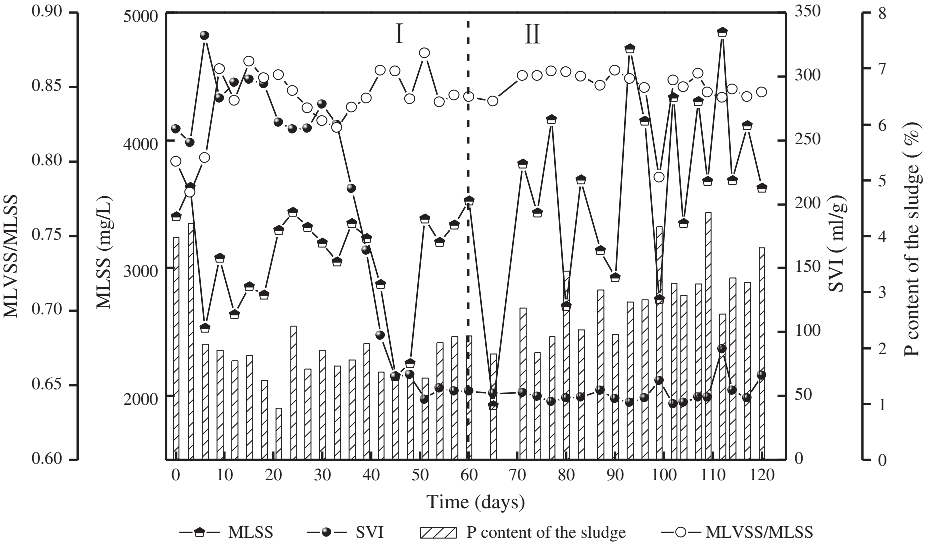

Fig. 4. Variations in SVI, MLSS, and P content (%) in the AO-SBR at various periods.

substrate and under the same operating conditions as the system without stripping. To recover P in sidestream, half of the volume of the anaerobic supernatants was used in this study. The available P with which PAOs replenish their intracellular poly-P pools was reduced, thereby inhibiting biological P removal. This result suggests that even the partial P that was liberated by PAOs in anaerobic conditions is stripped by chemical precipitates, thus limiting P accumulation in the subsequent aerobic condition. As a result, the entire metabolism process of P is restrained in the PAOs. Given these findings, the effects of intracellular poly-P on P removal efficiency merit further research.

To maintain the balance between chemical and biological P removal, a suitable deprivation ratio must be determined for P recovery and EBPR in anaerobic—aerobic SBRs in the future.

3.3. Sludge characteristics

3.3.1. Variations in MLVSS/MLSS, and P/MLSS ratios

The ratio of MLVSS to MLSS can be a rough indicator of inorganic matter storage, assuming that the intracellular poly-P, in the form of poly-P granules, is non-volatile. In the experiments without sidestream

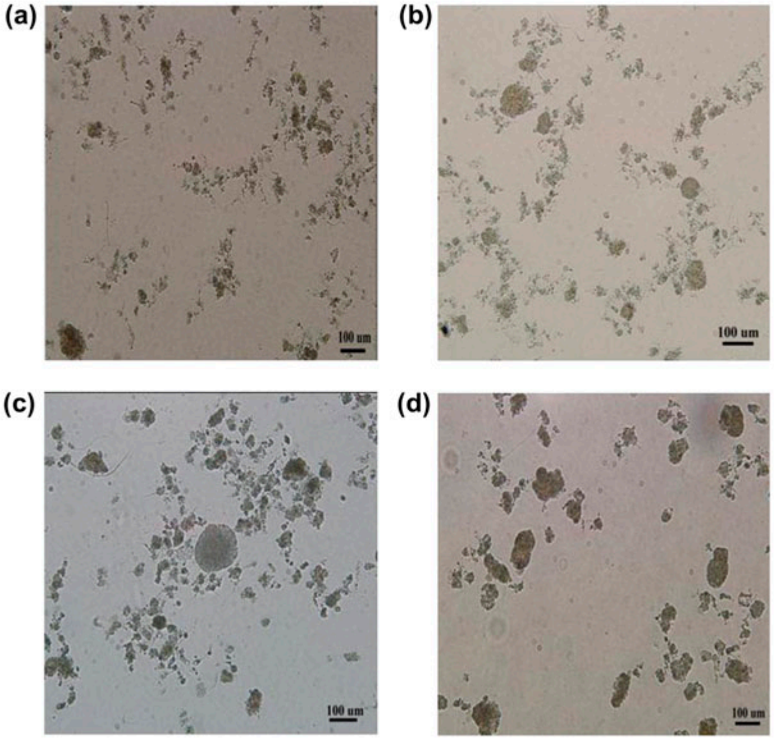

Fig. 5. Microscopy images (100×) of activated sludge during different periods: (a) Without stripping, (b) 15th d, (c) 40th d, and (d) 60th d.

stripping, MLVSS was 2,858 ± 154 mg/L, and the MLVSS/MLSS ratio was 0.77 ± 0.03. This low MLVSS/MLSS ratio suggests that PAOs were dominant in the sludge. During Periods I and II, MLSS and MLVSS fluctuated from 2,200 to 3,630 mg/L and from 1,900 to 2,830 mg/L, respectively, at the end of the aerobic phase. The corresponding MLVSS/MLSS ratio was 0.85 ± 0.02, which was relatively higher than that of the sludge that did not undergo sidestream stripping. A portion of P was removed as a chemical P compound; thus, the biosynthesis of intracellular poly-P was insufficient.

In Period I, the P content [42] in the sludge (based on MLSS) decreased with continuous stripping and remained at a low level (0.95–2.21%) (Fig. 4). This trend is comparable with that of typical activated sludge, which has a normal P content of 2.3%. Furthermore, the maximum phosphate concentration in liquid gradually declined from 55.29 to 32.6 mg/L (from the 0th d to 10th d), 31.25 to 20.42 mg/L (from 11th d to 40th d), and 19.63 to 9.59 mg/L (from 41st d to 60th d) in the anaerobic stage. By contrast, no significant release of anaerobic P was observed after 40 d of stripping. These findings indicate that the storage of intracellular poly-P content in the PAOs is deficient as a result of continuous stripping. The stored energy pool of the PAOs was insufficient under anaerobic conditions, and P uptake was restrained in the sequent aerobic stage. These results are consistent with previous observations [43] that poly-P content in anaerobic—aerobic activated sludge is limited without EBPR.

During the recovery period, P concentration in the sludge gradually increased to 4.44% along with orthophosphorus release (39.42 mg/L) in the anaerobic stage. The intracellular poly-P pool apparently recovered during biological treatment. When sidestream stripping was terminated for 60 d, however, effluent P concentration remained high, and EBPR could not be recovered. This finding suggests that PAOs may have been washed out of the system. Although microbial population structure was not investigated in this study, the metabolic model predictions of PAOs, $P_{release}/HA_{cuptake}$ ratios, low poly-P content, and low MLVSS/MLSS ratios confirm that PAOs were not dominant in the sludge.

3.3.2. Sludge sedimentation

SVI, which is an indicator of sludge settleability, varied at different operational periods as shown in

Fig. 4. The SVI declined gradually from 284.72 to 53.87 mL/g during stripping in Period I. The SVI then remained at approximately 52.43 ± 9.78 mL/g during residual operation in Period II, thus indicating that the stripping process significantly enhances sludge settleability. With stripping, high floc diameters (Fig. 5(c) and (d)) in the form of compact aggregates simultaneously improved with reduced SVI in AO-SBR. The result suggests that the settleability of the sludge samples with stripping was higher than that of sludge samples without stripping.

4. Conclusions

In short-term operations, sidestream stripping benefits biological P removal and chemical P recovery. Throughout the entire process, sludge settleability is significantly enhanced. During the later days of stripping, however, the reduced concentration of intracellular poly-P collapses EBPR, and it cannot be recovered even if sidestream stripping is terminated for 60 d. Another contributor to EBPR collapse is the dominance of GAOs rather than PAOs. Therefore, levels of intracellular poly-P storage should be regarded as an important factor that affects the performance of EBPR systems.

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