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Enhancing phosphorus recovery by a new internal recycle seeding MAP reactor

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Abstract

Phosphorus is a depleting resource that needs recovery from wastewater streams. The magnesium ammonium phosphate (MAP) crystallization process could simultaneously recover ammonium nitrogen and phosphorus at equal molar basis to yield slow-release MAP fertilizer. However, the present MAP processes are not efficient in recovering phosphorus at low P concentrations. This work presented and tested the performance of a newly proposed MAP reactor, the internal recycle seeding reactor (IRSR) that comprised of a reaction zone and a settling zone connecting with an internal recirculation loop. Owing to the enhanced secondary nucleation rates of MAP crystals in reaction zone under controlled circumstance, the proposed IRSR recovered 78% of phosphorus from wastewater at a low level of 21.7 mg-P L^{-1}. The optimal operation parameters for the IRSR were investigated with synthetic wastewater and determined as that the Mg/PO_4^{3-}-P molar ratio was 1.3–1.5:1, THRT was up to or longer than 1.14 h, the seed concentration of reaction zone was 0.40–1.0 g L^{-1}. Further needs for the proposed IRSR strategies were also discussed.

Keywords: MAP; Internal recycle seeding reactor; Phosphorus recovery; Seed

1. Introduction

Phosphorus is a depleting resource (Steen, 1998). In addition, as a result of its consumption, it returns the environment as the waste or through wastewater, which has potential to cause eutrophication or blue green algal blooms of receiving waters. Thus, it is obligatory and necessary to remove and recover phosphorus from waste or wastewater in order to contribute toward sustainable development, alleviate the environmental pressure and meet the increasingly strict regulations for phosphorus discharge. Recovery and removal of phosphorus from wastewater streams can be achieved via various processes, such as metal precipitation, constructed wetland systems, biological nutrient removal (BNR) processes, enhanced biological phosphorus removal (EBPR) processes, MAP (magnesium ammonium phosphate) crystallization process, and others (de-Bashan and Bashan, 2004). Among these processes, the magnesium ammonium phosphate (MAP, mineralogically as struvite) crystallization has been regarded as a promising method, because it can simultaneously recover ammonium nitrogen and phosphorus at equal molar basis to yield slow-release MAP fertilizer (Li and Zhao, 2003).

Process parameters and mechanisms of MAP crystallization were conducted and summarized (Battistoni et al., 2002; Doyle and Parsons, 2002; Jaffer et al., 2002). Studies had been performed to realize effects of numerous process

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parameters on MAP performance, including pH (Ohlinger et al., 1998; Mijangos et al., 2004), reactant origins (Chimenos et al., 2003; Yang and Sun, 2004), molar ratios of Mg/N/P (Altinbas et al., 2002), temperature (Mijangos et al., 2004), the presence of foreign ions (Le Corre et al., 2005; Kabdaşli et al., 2006), and mixing energy (Ohlinger et al., 1999). MAP crystallization is applied to treat phosphorus or ammonium nitrogen laden wastewaters (Battistoni et al., 2002; Li and Zhao, 2002, 2003; Lee et al., 2003; Kim et al., 2004; Quintana et al., 2004; Tunay et al., 2004; Wu and Bishop, 2004) and separated human urine (Ban and Dave, 2004). Three types of batch reactors were adopted with sufficient phosphorus recovery: mechanically stirring reactor (MSR) (Stratful et al., 2004; Yoshino et al., 2003), air agitated fluidized bed reactor (AAFBR) (Jaffer et al., 2002; Le Corre et al., 2007a), and water agitated fluidized bed reactor (WAFBR) (Battistoni et al., 2000, 2001; Adnan et al., 2003). The configuration and operation of MSR is simple, but consumes considerable amount of mixing energy (Wu and Bishop, 2004). Struvite crystals can grow rapidly in the AAFBR and WAFBR, however, the corresponding energy demand is also high (Battistoni et al., 2005). Additionally, large MAP crystals thus formed are not only poorly fluidized in the reactors, but also reduce MAP recovery owing to low surface area (Shimamura et al., 2001). Suzuki et al. (2007) and Le Corre et al. (2007b) recently inserted stainless steel meshes in the upper section of AAFBR to reduce energy demand and to minimize fines remaining in solution, thereby enhancing phosphorus recovery. Shimamura et al. (2003) devised a two-tank reactor to keep MAP crystal size constant in the reaction tank and phosphorus recovery efficiency stable.

Difficulties were noted to operate these MAP reactors. Suzuki et al. (2007) and Le Corre et al. (2007b) demonstrated the quantity of struvite crystal needed in the reaction zone was hard to control. Suzuki et al. (2007) noted shortcut flow between reaction zone and precipitation zone in the reactor reduced recovery efficiency. Moreover, reactor proposed by Le Corre et al. (2007b) and Shimamura et al. (2003) required external recirculation loops to fluidize and recycle seed crystals, respectively. To overcome the mentioned difficulties, in this study, a new MAP reactor, namely, the internal recycle seeding reactor (IRSR), was proposed and its effect and operation performance in phosphorus recovery from wastewater were tested and determined, in order to provide a potential and practical reactor used in the future for phosphorus recovery from wastewater. Burns et al. (2003) and Adnan et al. (2004) claimed that crystal seeding strategy insignificantly affected the MAP reactor performance, but Wu and Bishop (2004), Lee et al. (2005) and Wang et al. (2006) reported seeding could enhance the MAP reaction rate, increased crystal size and improved crystal settleability. Thus, the other objective of this work is to investigate into whether crystal seeding affects the performance of the proposed IRSR.

2. Methods

2.1. Reactor design and preliminary test

The IRSR consisted of two concentric columns (Fig. 1), with an internal column of 0.35 L effective volume for reaction, and an external column of 2.5 L effective volume for crystal sedimentation. Seed were lifted continuously by air to reaction zone to contact with wastewater for forming new MAP crystals. Then the agglomerates were settled in the external settling zone. The crystal concentration, including primary and new MAP crystals in the reaction zone, could be controlled by adjusting the circulation flow rate and the quantity of crystals at the bottom, respectively or both. The circulation flow rate of air lift pipe depended on the flow rate of main air pipe, while the crystal quantity in crystal zone was kept by discharge amounts of crystal. Three sub air pipes fixed on the inner wall of the internal column in proportional spacing provided blending power to avoid potential shortcut.

All chemicals were in analytical reagent grade and were used without further purification. The synthetic wastewater was made by mixing tap water, NH_4Cl, and $Na_2HPO_4 \cdot 12H_2O$ salts, whose pH was adjusted to 9.2–9.7 using NaOH. The synthetic wastewater was settled for 8–10 h before use to minimize the effects of Ca^{2+}, Mg^{2+} and Fe^{3+} originated potentially from tap water by spontaneous precipitation at base condition. The magnesium solution was prepared using $MgCl \cdot 6H_2$ salt due to its superiority in MAP crystallization (Le and Li, 2006). Both magnesium solution and synthetic wastewater were injected by peristaltic pump (YZ1515w, Baoding Longer

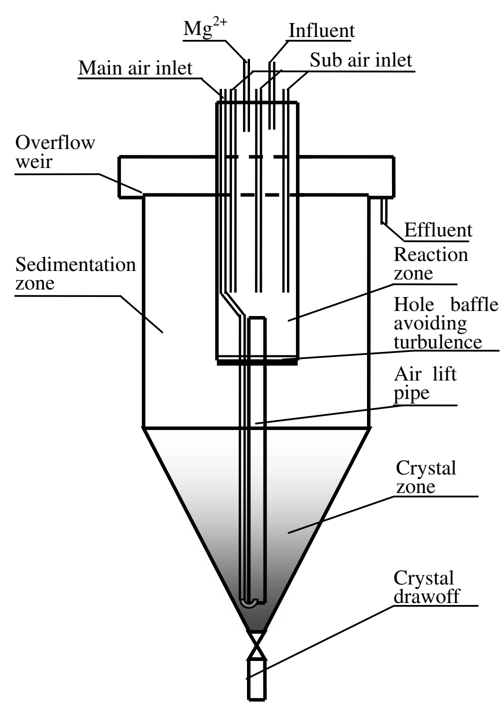

Fig. 1. Configuration of IRSR.

Precision Pump Co., Ltd., China) quantificationally depending on the experiment design.

Table 1 lists the tested experimental conditions. Three different phosphorus concentrations were adopted and the influent flow rate was fixed at 1.25 L h^{-1}. The molar ratios of Mg/PO_4^{3-}-P were achieved with magnesium solution of 0.25 mol L^{-1}.

2.2. Performance tests of IRSR

Three operating conditions were investigated to determinate the performances of IRSR, respectively. In all experiments, the flow rate of main air pipe and each sub pipe were kept at 11–12 L h^{-1} and 1–2 L h^{-1}, respectively. All tests were conducted in triplet and the mean and standard deviation of collected data were reported.

2.2.1. Seed concentration in reaction zone

The concentrations of PO_4^{3-}-P, NH_4^+-N and pH of synthetic wastewater were 60.4 ± 1.48 mg L^{-1}, 81.6 ± 0.99 mg $L^{-1}$and 9.63 ± 0.02, respectively. The operating temperature ranged 16.2–17.5 °C and the molar ratio of Mg/PO_4^{3-}-P was at 1.54 ± 0.04 with magnesium solution of 0.25 mol L^{-1}. The influent flow rate was fixed at 1.25 L h^{-1}. Four seed concentrations, 0.12 ± 0.03, 0.30 ± 0.05, 0.5 ± 0.1, and 1.20 ± 0.1 g L^{-1}, were kept in the reaction zone.

2.2.2. Molar ratio of Mg/PO_4^{3-}-P

Six Mg/PO_4^{3-}-P molar ratios, 1.03:1, 1.12:1, 1.22:1, 1.31:1, 1.49:1 and 2.05:1 were tested herein. The PO_4^{3-}-P, NH_4^+-N concentration and pH was 53.0 ± 0.14 mg L^{-1}, 81.2 ± 0.79 mg L^{-1} and 9.59 ± 0.02, respectively. The operating temperature was 12.0–16.5 °C, the seed concentration in reaction zone was kept in 0.4–1.0 g L^{-1}, the influent flow rate was fixed at 1.25 L h^{-1}, and the molar ratio of Mg/PO_4^{3-}-P was achieved accurately to some extent with magnesium solution of 0.04 mol L^{-1}.

2.2.3. Total hydraulic retention time (THRT)

THRT included reaction time and precipitation time, the ratio of reaction time to precipitation time was 0.14. Six THRTs of 4.56, 2.28, 1.71, 1.14, 0.76 and 0.57 h were obtained by adjusting the flow rate of influent, with the respective PO_4^{3-}-P, NH_4^+-N concentrations and pH of 52.4 ± 0.40 mg L^{-1}, 81.6 ± 0.59 mg L^{-1} and 9.56 ± 0.03. The operating temperature was 16.2–18.2 °C, the seed concentration in reaction zone was kept in 0.4–1.0 g L^{-1}, and the molar ratio of Mg/PO_4^{3-}-P of 1.73 ± 0.05 was achieved with magnesium solution of 0.25 mol L^{-1}.

2.3. Sampling and analysis methods

Four samples were taken at 1.0 h interval in each batch test and measured up to steady-state being reached. The concentrations of NH_4^+-N and PO_4^{3-}-P were analyzed according to Standard Methods (APHA, 1998). The suspension pH was measured using PHS-3C type pH meter (Shanghai Weiye Instrument Plant, China). Seed concentration in reaction zone was sampled and filtered through 0.45-μm filter paper. The filtered crystals were dried at 55 °C before weighing.

3. Results and discussion

3.1. Preliminary test

Table 2 lists the phosphorus and ammonium recovery by IRSR under different conditions.

The tests with seeds (runs 1, 3, and 5) removed more phosphorus than those without seeds and their effluent concentrations of phosphorus were lower than the corresponding control tests (runs 2, 4, and 6). Hence, seeding crystal via internal loop enhanced phosphorus recovery, since the higher the supersaturation ratio of solution was, the shorter induction time needs and the higher growth rate of struvite was (Kofina and Koutsoukos, 2005). In addition, once a seeded struvite crystallization reaction was underway in a supersaturation ratio solution, the new smaller MAP crystals could be used as the seed (Adnan et al., 2004). Thus the enhancement with seeding was 19% at low (comparison of runs 1 and 2), 6% at medium (comparison of runs 3 and 4) and less than 1% at high phosphorus and ammonium feeding levels (comparison of

Table 1
Characteristics of synthetic wastewater and operating conditions in the experiments of determination on the effect of IRSR

Run	Characteristics of synthetic wastewater			Operating conditions				
	PO_4^{3-}-P (mg L^{-1})	NH_4^+-N (mg L^{-1})	pH	Molar ratio		T/°C	Air flow[a]/L h^{-1}	
				Mg/P	N/P		Main pipe	Sub pipes[b]
1	21.7	40.0	9.54	2.85	4.07	9–10	11–12	(1–2) × 3
2	21.7	40.0	9.54	2.85	4.07	9.5–10	2–3	(3–4) × 3
3	54.1	83.6	9.68	1.71	3.42	10.5–11	11–12	(1–2) × 3
4	54.1	83.6	9.68	1.71	3.42	9.5–10	2–3	(3–4) × 3
5	153.4	4560	9.23	1.61	65.8	12–12.5	11–12	(1–2) × 3
6	153.4	4560	9.23	1.61	65.8	14–15	2–3	(3–4) × 3

[a] When the air flow of main pipe was above 10 L h^{-1}, the crystals could be lift continuously in this IRSR.
[b] There were three sub pipes in IRSR.

Table 2
Different phosphorus recovery effects between seeding crystals or not in IRSR

Run	Effluent of IRSR (mg L^{-1})		Removal amounts (mmol L^{-1})[a]		
	PO_4^{3-}-P	NH_4^+-N	pH	PO_4^{3-}-P	NH_4^+-N
1	4.71 ± 0.09	31.9 ± 0.38	9.13 ± 0.03	0.55 ± 0.003	0.57 ± 0.03
2	8.82 ± 0.14	34.1 ± 0.40	9.28 ± 0.04	0.41 ± 0.004	0.42 ± 0.03
3	4.22 ± 0.24	55.2 ± 0.46	8.97 ± 0.04	1.61 ± 0.008	2.02 ± 0.03
4	7.49 ± 0.23	56.2 ± 0.44	9.09 ± 0.02	1.50 ± 0.007	1.95 ± 0.03
5	0.32 ± 0.08	4460 ± 30	9.18 ± 0.005	4.94 ± 0.003	7.30 ± 2.17
6	1.05 ± 0.26	4450 ± 28	9.18 ± 0.005	4.92 ± 0.008	8.08 ± 2.02

[a] Removal amount (mmol L^{-1}) = (influent concentration–effluent concentration)/molecular weight of P&N.

runs 5 and 6), which represented low, medium and high supersaturation ratio solution, respectively. Adnan et al. (2004) claimed that the MAP process was not cost effective at low phosphorus influent concentration (20–30 mg L^{-1}). The proposed IRSR, on the contrary, revealed a high phosphorus recovery rate of 78% at influent phosphorus concentration of only 21.7 mg L^{-1} (run 1) because that the recycle seeding could enhance the MAP reaction rate, increase crystal size and improve crystal settleability. Such an observation indicates the significant potential to apply IRSR in phosphorus recovery from domestic wastewater. The advantage of applying IRSR, however, diminished at high supersaturation ratio solution, such as higher PO_4^{3-}-P concentration, higher molar ratio of N/P and Mg/P etc., like the synthetic wastewater used in runs 5 and 6.

3.2. Operation performance

Seeding enhances MAP crystals growth in the reaction zone, meanwhile, it also costs energy (Wu and Bishop, 2004). As Fig. 2 shows, the residual phosphorus concentrations were all less than 7 mg L^{-1} over seeding concentration of 0.15–1.2 g L^{-1}, with the minimum residual phosphorus concentration was achieved at seed concentration of 0.50 g L^{-1}. However, the noted differences from tests of different seeding concentration were not significant. A low recirculation rate in the loop that acquires minimum energy input but still performs well is considered. In addition, considering the difficulty of controlling a constant seed concentration of 0.50 g L^{-1} in practical use, the seed concentration of 0.40–1.0 g L^{-1} was suggested as optimum amount in reaction zone of IRSR.

High molar ratio of Mg/PO_4^{3-}-P yields high phosphorus recovery based on chemical equilibrium calculation, but produces high chemical cost. The residual phosphorus concentrations from IRSR were less than 10 mg L^{-1} at molar ratio of Mg/PO_4^{3-}-P greater than the stoichiometric ratio of 1:1 (Fig. 3). However, again, increasing this molar ratio only yielded incremental increase in phosphorus recovery. A molar ratio of Mg/PO_4^{3-}-P of 1.3–1.5:1 could be selected for IRSR operation considering minimum use of chemicals.

The phosphorus recovery increased with increasing total hydraulic retention time (THRT) of IRSR. No further improvement was achieved at THRT > 1.14 h (8.4 min for reaction and 60 min for settling) (Fig. 4). Even with a reduced TRHT of 0.57 h, the residual phosphorus

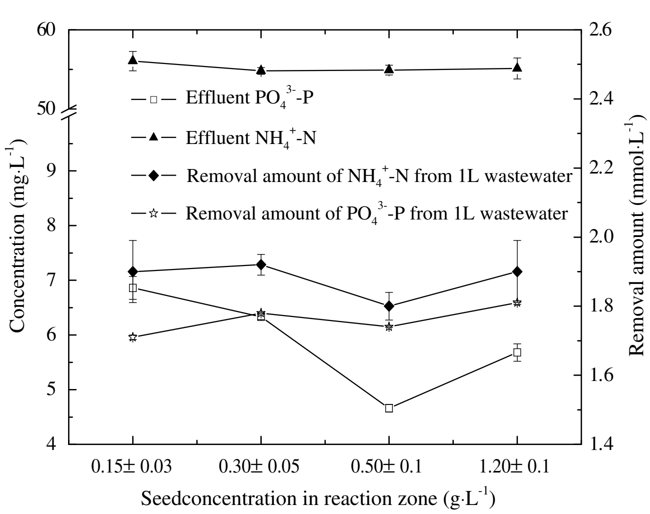

Fig. 2. Correlation between seeds quantities and effluent concentrations and removal amounts from IRSR. (THRT = 2.28 h, initial PO_4^{3-}-P=60.4 ± 1.48 mg L^{-1}, NH_4^+-N = 81.6 ± 0.99 mg L^{-1}, pH = 9.63 ± 0.02, temperature = 16.2–17.5 °C, Mg/PO_4^{3-}-P = 1.54 ± 0.04.)

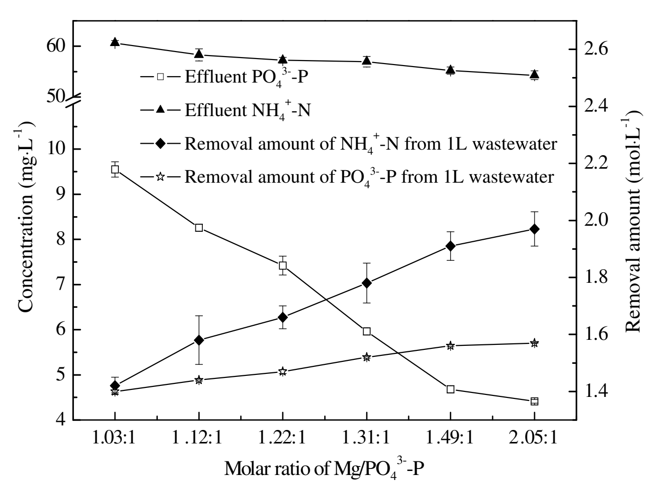

Fig. 3. Correlation between Mg/PO_4^{3-}-P molar ratio and effluent concentrations and removal amounts from IRSR. (THRT = 2.28 h, initial PO_4^{3-}-P = 53.0 ± 0.14 mg L^{-1}, NH_4^+-N = 81.2 ± 0.79 mg L^{-1}, pH = 9.59 ± 0.02, temperature = 12.0–16.5 °C, seed concentration in reaction zone = 0.4–1.0 g L^{-1}.)

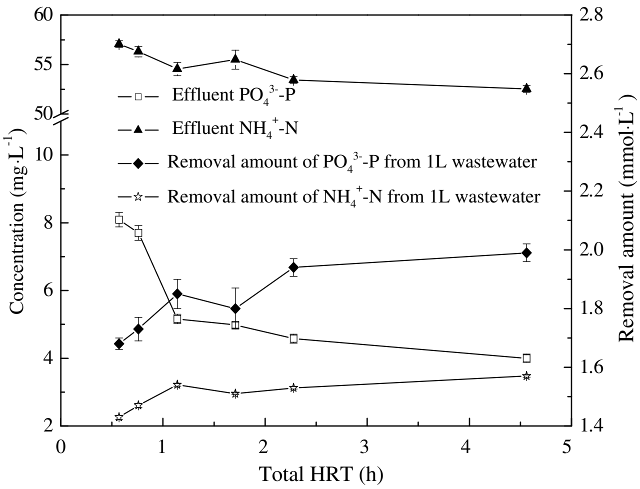

Fig. 4. Correlation between THRT and effluent concentrations and removal amounts from IRSR. (Initial PO_4^{3-}-P = 52.4 ± 0.40 mg L^{-1}, NH_4^+-N = 81.6 ± 0.09 mg L^{-1}, pH = 9.56 ± 0.03, temperature = 16.2–18.2 °C, seed concentration in reaction zone = 0.4–1.0 g L^{-1}, Mg/PO_4^{3-}-P = 1.73 ± 0.05.)

concentration was less than 10 mg L^{-1}. The proposed IRSR could thereby be designed in a compact manner in domestic wastewater treatment plant.

Two disadvantages were noted in the tests for proposed IRSR. Firstly, in all herein reported tests, the removal quantities of NH_4^+-N exceeded those of PO_4^{3-}-P based on the stoichiometric ratio of 1:1 for MAP. Since the loss of ammonium increased with increasing THRT and initial ammonium concentrations (Fig. 4, Table 2), such an occurrence may be attributable to stripping loss at alkaline condition. The stripping loss of NH_4^+-N may be minimized with the revised feeding strategy by Kim et al. (2007). Secondly, during the tests, certain amount of fine crystals would attach to the reactor's wall and could be flushed out with effluent. Le Corre et al. (2007a) coagulated the fines from their reactor to reduce this loss. A collection device as proposed by Suzuki et al. (2007) and Le Corre et al. (2007b) may be applicable to the present IRSR. Further studies are needed to justify these improvement strategies.

4. Conclusions

A new MAP reactor, the IRSR, which comprises a reaction zone and a settling zone, was proposed to enhance phosphorus recovery from wastewater stream containing low levels of phosphorus. A recirculation loop providing seed crystals from reaction zone promote secondary nucleation rate of MAP. This proposed MAP reactor increased phosphorus recovery efficiency by 19% at low phosphorus influent concentrations, but revealed similar performance at high phosphorus influent concentrations compared with the non-seeded reactor. Performance tests showed that the proposed MAP reactor could achieve sufficient phosphorus recovery using seeding concentration of 0.4–1.0 g L^{-1}, molar ratio of Mg/PO_4^{3-}-P of 1.3–1.5:1, and THRT > 1.14 h. On the other hand, two disadvantages, including ammonium stripping loss and fine MAP crystals loss remain unresolved for the proposed IRSR.

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