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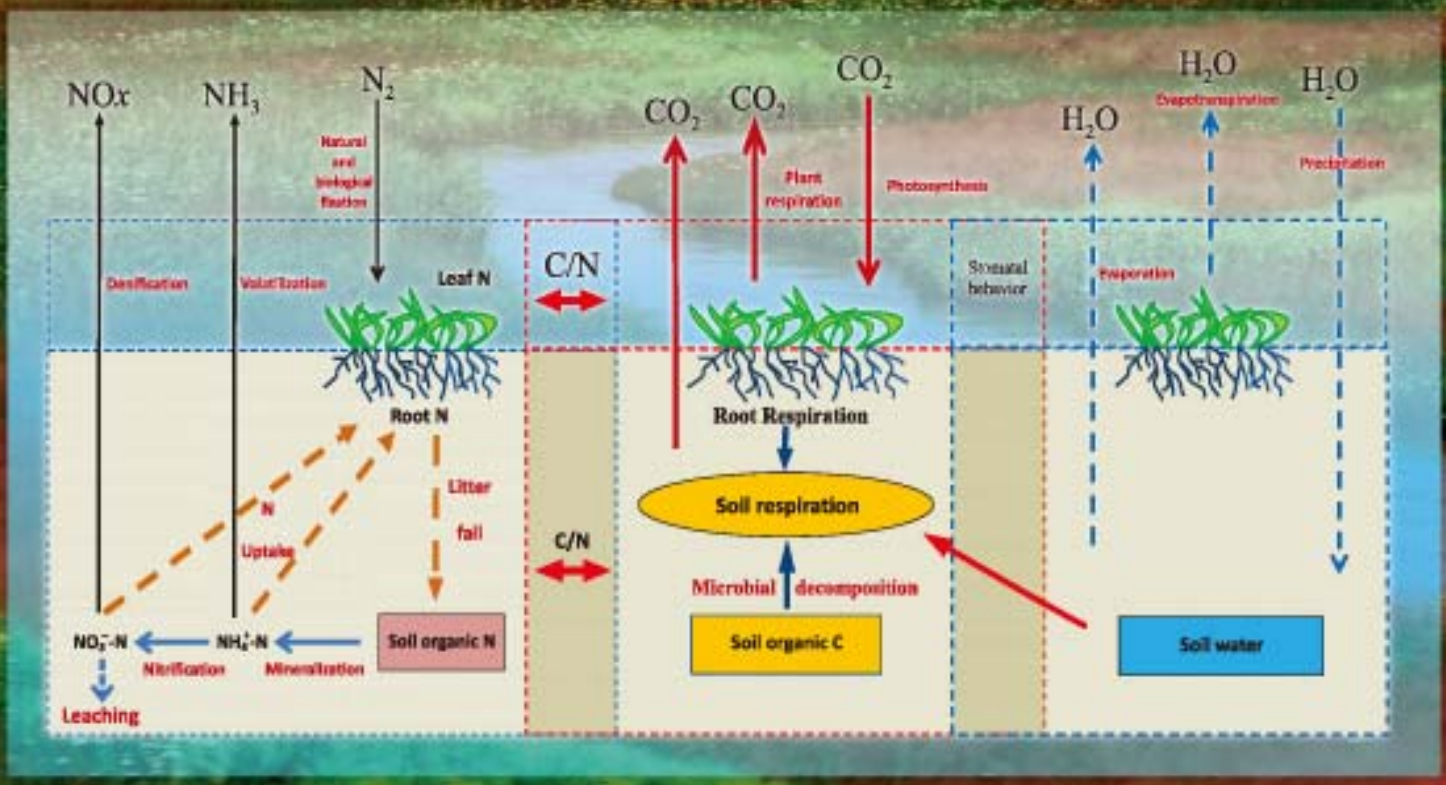

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Enhanced struvite recovery from wastewater using a novel cone-inserted fluidized bed reactor

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ABSTRACT

The feasibility of struvite recovery at low (12.5 mg/L) and high (120 mg/L) phosphorus concentrations was studied by constructing a novel fluidized bed reactor with cones (FBR_{wc}) and without cones (FBR_{woc}). The crystallization process was continuously operated for 133 days under different hydraulic retention times (HRT = 1–10 hr), pH (7.5–10), and molar ratios of Mg/P (0.75–1.75), N/P (1–10) and Ca/Mg (0–2). The optimum operating conditions of HRT, pH, Mg/P and N/P molar ratios were found to be 2 hr, 9, 1.25, and 7.5, respectively. Under these optimum conditions, the phosphorus precipitation efficiencies of FBR_{wc} were 93% for low and 98% for high phosphorus influent; however, the efficiencies were 78% and 81% for FBR_{woc}, respectively. Due to crystal losses at each junction (17%–31%), the crystal recovery efficiency of FBR_{woc} was relatively low (47%–65%) for both influent concentrations. However, the losses were minimal in FBR_{wc}, which showed 75% and 92% crystal recovery for low and high phosphorus concentrations, respectively. At low calcium concentration, crystal chemical analysis showed the product to be pure struvite (> 99%). The scanning electron microscope and X-ray diffraction results further confirmed that the crystal recovered from FBR_{wc} contained pure struvite, which could be considered a high quality fertilizer. Except HRT, all parameters (pH, Mg/P, N/P and Ca/Mg) were found to be influencing factors for FBR_{wc} performance. Overall, inserting cones in each part of the reactor played a significant role in enhancing struvite recovery from a wide range of phosphorus-containing wastewater.

Introduction

Phosphorus and nitrogen are the main nutrients causing eutrophication that lead to biodiversity loss and water quality problems. To maintain a healthy environment, many European Union countries are now establishing strict standards for the discharge levels of phosphorus and nitrogen in wastewater effluents. While excess phosphorus is problematic, it is also a non-renewable limited resource that may be depleted in approximately 50–100 years (Doyle and Parsons, 2002). According to Shu et al. (2006), phosphate rock that can be economically mined will be exhausted within 90 years at an annual extraction rate of 40 million tons and 1.5% increase in demand per

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year. Exhaustion even before 90 years was predicted by Driver et al. (1999). As a result of eutrophication and depletion problems, phosphorus treatment is now getting due attention.

Wastewater treatments that involve phosphorous recovery are believed to be an alternative approach leading to sustainable development (Le Corre et al., 2009). The potential sources for phosphorous recovery include wastewater streams, dairy manure, urine, landfill leachate, bone disposal and industrial wastes (Doyle and Parsons, 2002; Shu et al., 2006). Phosphorus removal can be accomplished by chemical precipitation, biological degradation, crystallization, tertiary filtration and ion exchange methods (Morse et al., 1998; de-Bashan and Bashan, 2004). Crystallization processing (nucleation followed by crystal growth), unlike most other methods, does not produce secondary wastes requiring further management (de-Bashan and Bashan, 2004). Previous studies showed that the best phosphorus crystallization product in wastewater treatment plants is struvite ($MgNH_4PO_4{\cdot}6H_2O$) (Pastor et al., 2010; Huchzermeier and Tao, 2012). The general formula for struvite precipitation is given in Reaction (1) (n = 0, 1, and 2 (Le Corre et al., 2005)).

$$Mg^{2+} + NH_4^+ + H_nPO_4^{n-3} + 6H_2O \rightarrow MgNH_4PO_4{\cdot}6H_2O + nH^+ \quad (1)$$

Struvite crystallization can be either spontaneous or controlled. Spontaneous struvite crystallization and its negative impact in wastewater treatment plants have been widely documented (Rawn et al., 1939; Borgerding, 1972; Battistoni et al., 2001; Britton et al., 2005). It occurs in pipeline locations (such as junctions, elbows, etc.) where carbon dioxide is being stripped (raising pH), which in turn causes clogging of pipelines and pumps that eventually leads to process failure and economic loss (Pastor et al., 2008). When struvite crystallization occurs under controlled conditions, it may be beneficial for sustainable development. Some of the reported benefits include production of a slow release fertilizer (Munch and Barr, 2001), reduction of sludge volume (Woods et al., 1999), simultaneous precipitation of phosphorus and nitrogen (de-Bashan and Bashan, 2004), replacement of natural phosphate used in industries (Pastor et al., 2010), avoiding unwanted pipe clogging and treatment failure (Pastor et al., 2008), and making a marketable product (Ueno and Fujii, 2001). More details of the economic evaluation of phosphorus recovery as struvite are well explained in other studies (Doyle and Parsons, 2002; Shu et al., 2006).

Although struvite crystallization is a promising phosphorus precipitation and recovery method from wastewater, it has not been widely adopted (Adnan et al., 2003; de-Bashan and Bashan, 2004; Pastor et al., 2010). Nowadays, only a few full scale struvite plants exist in the world due to a number of factors including supersaturation, pH, temperature and foreign ions (Ohlinger et al., 1998; Bouropoulos and Koutsoukos, 2000). Struvite crystallization can occur when the concentration of Mg^{2+}, NH_4^+ and PO_4^{3-} exceed the struvite solubility product (K_{sp}) (Ohlinger et al., 1998). Solubility products are generally reported as the negative log of the solubility product or pK_{sp} (Eq. (2)).

$$K_{sp} = [Mg^{2+}][NH_4^+][PO_4^{3-}] \quad (2)$$

Different struvite pK_{sp} values such as 9.42 (Borgerding, 1972), and 13.27 (Ohlinger et al., 1998) have been reported in a wide range of publications. Although the H^+ concentration does not directly enter the solubility product equation for struvite, struvite precipitation is highly pH dependent (Pastor et al., 2008). The relationship between K_{sp} and pH indicates that struvite solubility decreases with increasing pH, which in turn leads to an increase in the struvite precipitation potential. However, this does not to mean continued increases in pH are associated with struvite precipitation, because at higher pH ammonia is evolved as a gas due to dissociation of ammonium ions in the reaction solution. This phenomenon has a negative effect on struvite precipitation (Doyle and Parsons, 2002; Adnan et al., 2004). As a result, an optimum range of pH (8–10) is recommended for struvite formation (Ohlinger et al., 1998; Fattah et al., 2008; Huchzermeier and Tao, 2012). Moreover, the presence of high concentrations of calcium in the reaction solution could hamper struvite formation, because calcium reacts with phosphorus to form calcium phosphate precipitates (Le Corre et al., 2005).

In order to produce struvite, three types of reactors have been tested so far, including a mechanical stirring reactor (MSR) (Pastor et al., 2008), a gas-agitated fluidized-bed reactor (AAFBR) (Munch and Barr, 2001; Bowers and Westerman, 2005), and a water-agitated fluidized-bed reactor (WAFBR) (Battistoni et al., 2001; Adnan et al., 2003). These three reactor systems have their own advantages and disadvantages. The configuration and operation of a MSR is simple, but it consumes a considerable amount of energy for mixing (Doyle and Parsons, 2002). In the AAFBR and WAFBR, struvite crystals can grow rapidly; however, the corresponding energy demand is also high (Battistoni et al., 2005).

Currently, attempts have been made to improve energy demands and phosphorus recovery efficiencies by considering the reactor design (Adnan et al., 2003; Shimamura et al., 2003; Le Corre et al., 2007; Suzuki et al., 2007; Liu et al., 2008). Suzuki et al. (2007) and Le Corre et al. (2007) inserted stainless steel meshes in the upper section of the fluidized bed reactor to reduce energy demand and to minimize fines remaining in solution, thereby enhancing phosphorus recovery. Liu et al. (2008) designed a new internal recycle seeding reactor for struvite recovery, suggesting the internal self-seeding mechanism enhanced recovery efficiency by reducing the energy cost needed for an external recycler. Adnan et al. (2003) also constructed four-part reactors working with an external recycler and

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achieved an average of 57%–82% reactor performance. Adnan and his colleagues in another study (Adnan et al., 2004) stated that the reactor response (i.e. struvite recovery) at low phosphorus concentration (20–30 mg/L) was unsustainable. On the contrary, the reactor designed by Liu et al. (2008) showed encouraging struvite recovery efficiency at low phosphorus concentration (i.e. with 21.7 mg/L PO_4^{3-}-P, 78% recovery efficiency). According to de-Bashan and Bashan (2004), recovery of 10%–80% of phosphorus flowing into wastewater facilities is considered economically feasible. Despite such reactor modifications and recovery efficiencies made so far, a search for a better reactor design that works over a wide range of wastewater strength is still needed for sustainable phosphorus treatment.

Thus, the main objective of this study was to design a novel cone-inserted fluidized bed reactor (FBR_{wc}), and to evaluate its performance at low (12.5 mg/L) and high (120 mg/L) phosphorus concentrations. A fluidized bed reactor without cones (FBR_{woc}) was also used as a control.

1 Materials and methods

1.1 Reactor design and operation

Two equal sized reactors (FBR_{wc} and FBR_{woc}) were designed following the concept of the fluidized bed reactor at Tongji University, China. The reactor designs are shown in **Fig. 1**. The reactors have three distinct parts, bottom (volume 884 mL, diameter 60 mm), middle (volume 4142 mL, diameter 100 mm), and top (volume 4440 mL, diameter 170 mm), depending on the diameter of the column that progressively increased toward the top. The strategy of inserting cone-shape structures at an angle of 45° between the parts was aimed at reducing unwanted crystal loss at each junction (**Fig. 1a**). At the top part of the reactor, a lid was also placed to reduce ammonia stripping.

The entire layout includes eight ports on the FBR (five ports at the bottom for influent supply and product recovery, one port at the middle for sample removal, and two ports at the top for effluent outflow and pH control), an external recycler (volume = 13,000 mL, height = 840 mm), peristaltic pumps, and influent and effluent holding tanks (**Fig. 1c**).

While the whole system was continuously operated, influent and alkaline solutions were supplied to the FBR at the bottom ports using peristaltic pumps (YZ1515X-Longer Pump Co., Ltd., China). As the phosphorus-containing liquid flows upward, its flow velocity decreases leading to segregation of formed crystals. While large crystals are accumulated at the bottom and in the middle part of the FBR, finer particles flow out from the reactor and are recycled back with the effluent through the external recycler. When a sufficient amount of large crystals accumulated in the FBR, they were removed by opening the bottom valve.

1.2 Feed preparation

Synthetic wastewater containing the struvite constituent ions was used as influent. Salts used to prepare the influent feed were commercial-grade magnesium chloride hexahydrate ($MgCl_2{\cdot}6H_2O$), potassium dihydrogen phosphate (KH_2PO_4) and ammonium chloride (NH_4Cl). Depending on the desired experimental conditions, different concentrations of KH_2PO_4 (0.404 and 3.865 mmol/L), NH_4Cl (2.991–34.585 mmol/L) and $MgCl_2{\cdot}6H_2O$ (0.738–6.738 mmol/L) were prepared and fed to the reactor every 1 to 2 days from a 60 L influent holding tank. The terms low (Run 1) and high (Run 2) phosphorus concentrations refer the minimum and maximum KH_2PO_4 concentrations used

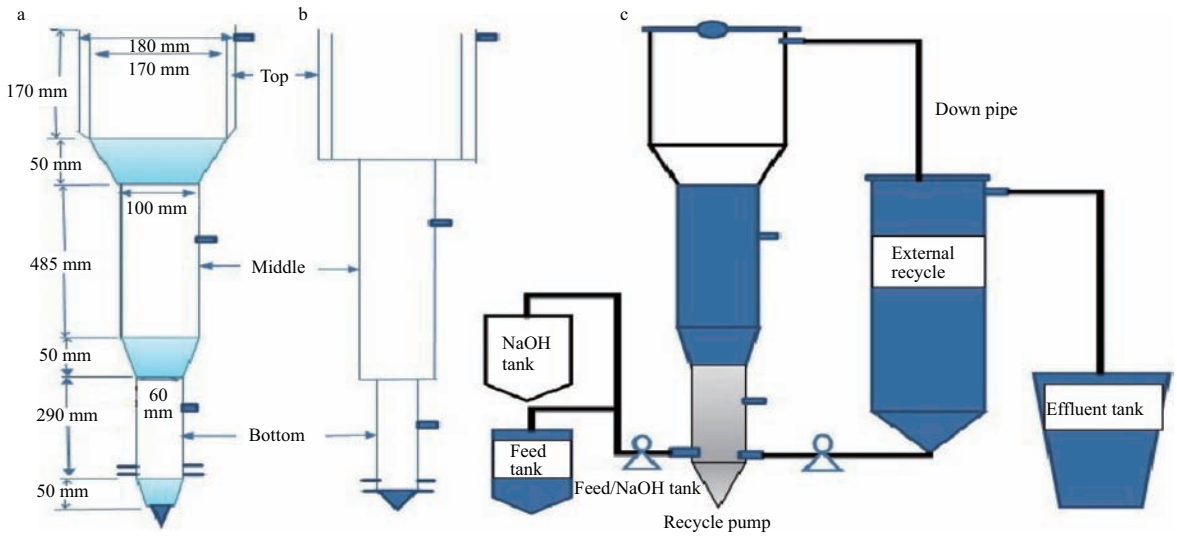

Fig. 1 Fluidized bed reactor (FBR) with cones (FBR_{wc}) (a), without cones (FBR_{woc}) (b), and entire FBR systems (c).

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(12.5 mg/L PO_4^{3-}-P and 120 mg/L PO_4^{3-}-P), respectively.

The pH value was adjusted by adding sodium hydroxide (0.0125 mol/L NaOH) as used by other researchers (Adnan et al., 2004; Britton et al., 2005; Huang et al., 2006; Le Corre et al., 2007; Pastor et al., 2008). The level of pH in the reactor was measured using a PHS-29A meter (HACH, USA).

1.3 Experimental procedures

Two consecutive runs (Run 1 and Run 2) were conducted from February to June 2012. For each run, different experimental conditions were investigated including HRT (1, 2, 4, 6, 8 and 10 hr), pH (7.5, 8, 8.5, 9, 9.5 and 10), Mg/P (0.75, 1, 1.25, 1.5 and 1.75), N/P (1, 2.5, 5, 7.5 and 10) and Ca/Mg (0, 0.5, 1 and 2) molar ratios. Depending on the desired Ca/Mg molar ratios, a range of calcium chloride (0–9.65 mol/L) concentrations was prepared and used as the source of calcium. In each experiment, the parameters were varied at 4 to 6 levels by monitoring the concentration of synthetic wastewater constituents and/or the pump flow rates. When one parameter was varied, the remaining parameters were kept constant. All experiments were carried out at room temperature. A summary of the initial operating conditions is shown in **Table 1**.

1.4 Sampling and analyses

For chemical analysis, influent and effluent samples were collected every two days from the feed tank and external recycler outflow, respectively (**Fig. 1c**). The constituents of orthophosphate-phosphorus (PO_4^{3-}-P), total phosphorus (TP), ammonium-nitrogen (NH_4^+-N), magnesium (Mg), and calcium (Ca) were analyzed following standard methods (APHA, 1998). PO_4^{3-}-P, TP and NH_4^+-N were analyzed using a UV-Visible spectrophotometer (UV 1700, Shimadzu, Japan) whereas Mg and Ca were analyzed using a flame atomic absorption spectrophotometer (PE-AA400, Perkin Elmer, USA). For some of the samples, an inductively coupled plasma-optimal emission spectrophotometer (ICP-OES) (ICP-720ES, Agilent, USA) was used to quantify Mg, Ca, iron (Fe), aluminum (Al), potassium (K), sodium (Na) and other trace metals together.

1.5 Crystal harvesting and analyses

Products were harvested from the reactor after stopping the recycle and feed flows. Crystals were then collected and air dried for further analysis. The quality of the crystals was determined by their composition and morphology. In order to carry out chemical composition tests, 30 mg of crystal sample was dissolved in 50 mL of 0.5% nitric acid solution (Huang et al., 2006). The suspensions were stirred and allowed to stay overnight before analysis. Then, the solution was analyzed for PO_4^{3-}-P, NH_4^+-N, Mg and Ca concentrations.

The morphology of the harvested crystals was examined using a scanning electron microscope (SEM) (XL30, Philips, the Netherlands) at Fudan University, China. The crystals were also examined by X-ray diffraction (XRD). After the dried crystals were crushed to smaller pieces, powdered samples were analyzed on a Bruker D8 Advanced X-ray Diffractometer using CuK-alpha radiation. The XRD patterns were recorded in the scanning range of 2-theta from 10°–90°. Data were collected and processed using MDI Jade 5.0 software. Identification of the phase peaks was accomplished by comparing the observed XRD patterns to that of standard struvite compiled by the Joint Committee on Powder Diffraction and Standards (JCPDS, 1980).

2 Results and discussion

2.1 FBR performance

To evaluate the reactor performance, two types (precipitation and recovery) of efficiencies were calculated on the basis of phosphorus removal from the solution (Pastor et al., 2008). The precipitation efficiency represents the maximum phosphorus precipitation from a thermodynam-

Table 1 Initial operating conditions for Run 1 and Run 2

	FBR_{woc}		FBR_{wc}	
	Run 1	Run 2	Run 1	Run 2
PO_4^{3-}-P (mg/L)	12.5	120.0	12.5	120.0
NH_4^+-N (mg/L)	42.0	363.0	5.65–56.5	48.0–484.0
Mg (mg/L)	12.0	116.0	7.25–17.0	70.0–162.0
Mg/P	1.25	1.25	0.75–1.75	0.75–1.75
N/P	7.5	7.5	1.0–10.0	1.0–10.0
Ca/Mg	–	–	0–2	0–2
pH	9.0	9.0	7.5–10.0	7.5–10.0
HRT (hr)	2	2	1–10	1–10
Recycle flow rate (mL/min)	30	30	30	30
Temperature (°C)	20–26	20–26	20–26	20–26

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ic point of view. It was calculated as the ratio between the difference of the influent and effluent orthophosphate concentration and the influent orthophosphate concentration. In order to consider the recovery efficiency, the precipitation and crystal growth efficiency was taken into account. It was calculated on the basis of total phosphorus concentrations in the influent and effluent streams. The difference between the two efficiencies is related to the presence of fines in the effluent stream and with the quantity of phosphorus lost with the effluent (Pastor et al., 2010).

At low and high concentrations of phosphorus, the FBR_{wc} showed a better efficiency than the control (FBR_{woc}). As shown in **Table 2**, when FBR_{woc} was operated under optimal conditions of pH (9), HRT (2 hr), Mg/P (1.25) and N/P (7.5) molar ratios, phosphorus precipitation efficiencies for Run 1 and Run 2 were 78.0% and 81.2%, respectively. Under the same operating conditions, FBR_{wc} showed 92.8% for Run 1 and 97.5% for Run 2, indicating the advantage of inserting the cones in the reactor. For Run 1 and Run 2, the corresponding recovery efficiencies were found to be 47.0% and 65.0% in FBR_{woc} and 75.0% and 92.0% in FBR_{wc}. The relatively lower values of precipitation and recovery observed in FBR_{woc} than FBR_{wc} were associated with higher crystal loss in the former (31.0% and 16.6% for Run 1 and 2, respectively). The large amount of phosphorus recovered using the new FBR_{wc} might indicate the economic feasibility of recovering phosphorus from low and high phosphorus-containing wastewater. According to de-Bashan and Bashan (2004), recovery of 10%–80% of phosphorus flowing into wastewater facilities is considered economically feasible.

In order to learn more about the advantages of cone insertion, the new FBR_{wc} precipitation (hereafter referred as removal, as used by others in **Table 3**) efficiency was compared with previously reported results. Unlike most of the reports, the efficiency of phosphorous removal in the current study reached about 98%. This is most likely due to the insertion of cones in the reactor, which significantly reduced unwanted crystal wastage at each junction (about 42% for Run 1 and 67.5% for Run 2) (**Table 2**). As shown in **Table 3**, reactor designs that have a larger number of cones achieved higher phosphorus removal than reactors constructed without or with fewer cones. For instance, Ueno and Fujii (2001) used two cones at the base and middle part of their reactor and achieved a better reactor performance than Pastor et al. (2008), who inserted a single cone between the parts. In addition, struvite crystallization

Table 2 Phosphorus precipitation and recovery efficiencies for FBR_{woc} and FBR_{woc}

	FBR_{woc}		FBR_{wc}	
	Run 1	Run 2	Run 1	Run 2
Influent PO_4^{3-}-P (mg/L)	11.8 ± 0.6	118.9 ± 4.4	12.0 ± 0.5	117.1 ± 3.4
Influent NH_4^+-N (mg/L)	39.0 ± 2.5	363.0 ± 1.9	41.0 ± 1.3	362.0 ± 3.7
Influent Mg (mg/L)	12.2 ± 1.1	116.6 ± 1.2	11.2 ± 1.7	115.1 ± 1.8
Effluent PO_4^{3-}-P (mg/L)	2.5 ± 0.5	22.0 ± 4.2	0.9 ± 0.4	2.9 ± 0.7
Effluent TP (mg/L)	6.2 ± 0.2	42.1 ± 9.7	3.0 ± 0.8	9.3 ± 1.3
Precipitation (%)	78.0 ± 4.0	81.2 ± 3.6	92.8 ± 3.0	97.5 ± 0.6
Recovery (%)	47.0 ± 3.1	65.0 ± 8.0	75.0 ± 7.1	92.0 ± 1.1
Changes in efficiency (%)	31.0 ± 3.4	16.6 ± 9.5	18.0 ± 8.0	5.4 ± 0.9

Table 3 Comparison of phosphorous removal efficiencies of the fluidized bed reactor (FBR) used in current and previous studies

Volume	Cones	Parts	Wastewater type	pH control/(value)	P-removal	References
9.5 (L)	+++	3	Synthetic	NaOH (7.5–10)	60%–98%	This study
Ns	++	2	Anaerobic liquor	NaOH (8.3–8.4)	84%–92%	Shimamura et al., 2003
143 (L)	++	2	Dewatering liquor	Aeration/$Mg(OH)_2$ (8.5)	94%	Munch and Barr, 2001
650 (m^3/day)	++	2	Digester liquor	NaOH (8.2–8.8)	>90%	Ueno and Fujii, 2001
21.0 (L)	+	2	Synthetic	NaOH (8.2–9.5)	40%–80%	Pastor et al., 2008
Ns	+	2	Anaerobic liquor	Aeration (7.6–8.2)	68.5%–86.6%	Battistoni et al., 2005
12.8 (L)	+	2	Belt press liquors	Aeration (8.0–8.8)	60%–91%	Battistoni et al., 2001
Ns	–	1	Anaerobic liquor	Aeration (7.9–8.6)	58%–81%	Battistoni et al., 1997
3.2(L)	–	1	Digester liquor	NaOH/Air (8.3)	>80%	Ohlinger et al., 2000
24.4 (L)	–	4	Digester liquor	NaOH (⩾8.3)	>90%	Huang et al., 2006
19.0 (L)	–	4	Digester liquor	NaOH (7.6–8.8)	30%–90%	Britton et al., 2005
24.4 (L)	–	4	Synthetic	NaOH (⩾8.3)	>90%	Adnan et al., 2003

Ns: not specified; –: no cone observed at the reactor; +: one cone, ++: two cones; +++: three cones.

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with and without cones has been extensively investigated by Battistoni et al. (1997, 2001, 2005) and the results for cone-inserted reactors showed better removal efficiencies than reactors used without cones (**Table 3**). This further signifies the positive role of cone insertion in the reactor for enhancing phosphorus removal efficiency.

As can be seen from **Table 3**, the efficiency of phosphorus removal may not only depend on the number of cones inserted but also on the number of reactor parts. Increasing the number of reactor parts help to achieve enhanced phosphorous removal efficiency. Example, Huang et al. (2006), Britton et al. (2005) and Adnan et al. (2003) used four reactor parts without cones and achieved higher performance in terms of phosphorous removal than Battistoni et al. (2001) and others who used a smaller number of parts (Battistoni et al., 1997; Ohlinger et al., 2000; Pastor et al., 2008). However, the results obtained in this study (with three reactor parts) appeared comparable or even higher than some of these reports. Generally, the results observed (60%–98%) in this study indicated that having a larger numbers of cones and parts play a significant role in achieving enhanced phosphorus removal efficiencies.

2.2 Effect of HRT, pH, Mg/P, N/P and Ca/Mg on phosphorus removal efficiency

Struvite crystallization is governed by a combination of key parameters including pH, initial concentration of ionic species in solution, mixing speed and the presence of foreign ions (Le Corre et al., 2007). In the current study, some of these factors were examined for evaluating the feasibility of the FBR_{wc}. As summarized in **Figs. 2** and **3**, under various experimental conditions, the reactor showed better phosphorus removal efficiencies (60%–98%) than for nitrogen (13%–70%).

Hydraulic retention times of 1, 2, 4, 6, 8, and 10 hr were considered and results showed more than 80% phosphorus and 25% nitrogen removal efficiencies in the first 1 hr HRT (**Fig. 2a**). The effect of HRT on removal efficiencies was found to be insignificant. For instance, when the HRT increased from 2 to 10 hr, 90% and 92.5% removal efficiencies for phosphorus were observed for Run 2. Our results agree with other studies (Battistoni et al., 2001; Le

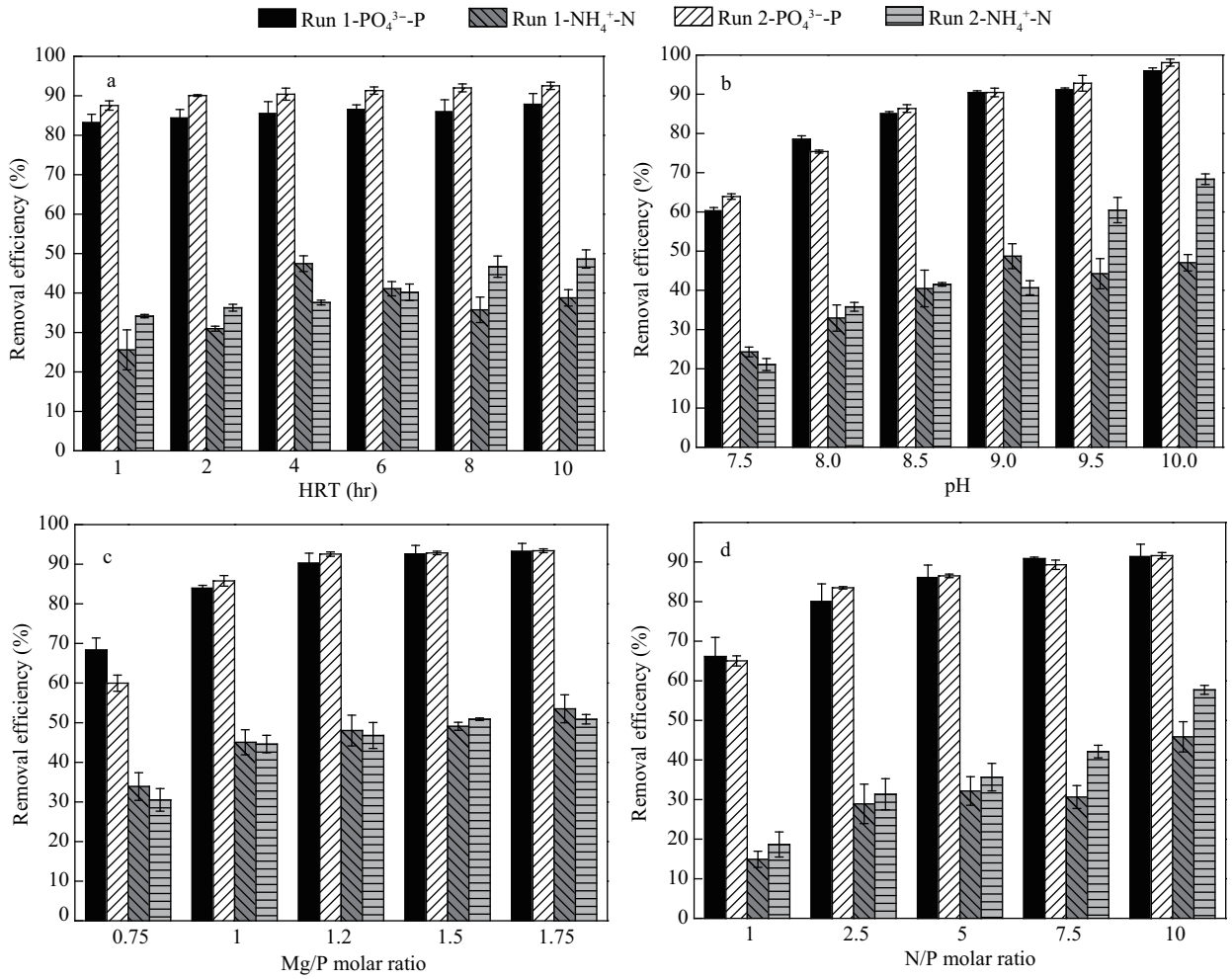

Fig. 2 Effects of hydraulic retention times (a), pH (b), Mg/P ratios (c) and N/P molar ratios (d) on PO_4^{3-}-P and NH_4^+-N removal efficiencies for Run 1 and Run 2.

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Corre et al., 2007; Liu et al., 2008; Pastor et al., 2010). According to Le Corre et al. (2007), the results obtained after 1 and 24 hr were almost the same (79%). Liu et al. (2008) also observed an enhanced phosphorus removal before HRT of 1 hr, indicating the insignificance of HRT in removal efficiencies of phosphorous.

Struvite crystallization requires alkaline pH. Many studies have stated that the operating range of pH for struvite formation is between 8 and 10 (Fattah et al., 2008; Huchzermeier and Tao, 2012). In this pH range, crystallization is known to be metastable (Bhuiyan et al., 2008). When pH is above and below the optimum ranges, supersaturated and unsaturated solutions can be formed (Ohlinger et al., 1998). In this study, the effects of pH from 7.5 to 10 were investigated. As shown in **Fig. 2b**, the phosphorus removal efficiencies increased from 60% to 98% when the reactor pH was raised from 7.5 to 10 (minimum and maximum removals shown related to Run 1 and Run 2, respectively). When the reactor was working at pH $\geqslant$ 9, more than 90% phosphorus removal efficiencies were achieved for both runs. Lower phosphorus removal efficiencies were achieved at pH 7.5, which is as expected based on the precipitation chemistry, because struvite is soluble under acidic condition (Ohlinger et al., 1998; Parsons et al., 2001). Our results are consistent with phosphorus removal efficiencies reported by Britton et al. (2005) (30%–90% at pH 7.6–8.8), Pastor et al. (2008) (40%–80% at pH 8.2–9.5) and Le Corre et al. (2007) (75%–80% at pH 8.5–10).

Compared to phosphorus, nitrogen removal efficiencies in the reactor were lower and showed more variability (20%–70%). In particular, Run 2 showed a sharp increase in nitrogen removal at pH $\geqslant$ 9.5, which could be due to ammonia loss by stripping at these pH ranges rather than struvite formation (**Fig. 2b**). If the improved nitrogen removal efficiency were due to struvite formation, phosphorus removal would not be almost constant. Previous reports showed that ammonium ion (NH_4^+) converted to ammonia (NH_3) at higher pH (Wang et al., 2005; Le Corre et al., 2007).

For struvite crystallization, at least a 1:1 molar ratio of Mg/P is needed (de-Bashan and Bashan, 2004; Shu et al., 2006). Several studies stated that struvite recovery and phosphorus removal efficiencies were increased with increasing Mg/P molar ratio (Le Corre et al., 2005; Huang et al., 2006). In this study, a range of Mg/P molar ratios (0.75–1.75) was evaluated. When the Mg/P molar ratio was adjusted to the theoretical struvite formation (Mg/P = 1) value, the phosphorus and nitrogen removal efficiencies were found to be more than 80% and 45%, respectively. However, when Mg/P molar ratios increased from 1.25 to 1.75, the phosphorus removal efficiencies showed only slight changes (**Fig. 2c**). In the struvite crystallization process, FBR that work optimally at lower magnesium dose (Mg/P = 1.25) could be more desirable in terms of the cost needed for magnesium supply.

The effects of N/P molar ratios were also investigated at constant HRT, Mg/P, and pH (2 hr, 1.25 and 9, respectively). When the N/P molar ratios increased from 1 to 10, ranges of phosphorus and nitrogen removal efficiencies (65%–90% and 15%–60%, respectively) were observed (**Fig. 2d**). When the N/P molar ratios increased from 1 to 7.5, the phosphorus removal efficiencies were increased gradually from 66% to 91% for Run 1 and 65% to 90% for Run 2. For N/P molar ratios above 7.5, the phosphorus removal efficiencies did not show any significant changes for either Run 1 or Run 2. The positive effects of increasing N/P molar ratios for phosphorus and nitrogen removals were consistent with other studies (Fattah et al., 2008; Pastor et al., 2008). When Zhao et al. (2008) increased the N/P molar ratio from 1 to 6, they observed 50% and 80% phosphorus removal efficiency, respectively.

The effects of calcium on struvite crystallization have been studied by many researchers (Le Corre et al., 2005; Wang et al., 2005; Pastor et al., 2008). The impacts of Ca/Mg molar ratios on struvite formation were also studied in our work. Under constant pH = 9, Mg/P = 1.25 and N/P = 7.5 molar ratios, phosphorus and calcium removal efficiencies progressively increased with increasing Ca/Mg molar ratios (**Fig. 3a**). Phosphorus removal efficiencies were 81% without calcium and 98% for the maximum calcium added (Ca/Mg = 2). This high phosphorus removal efficiency might not be totally associated with struvite formation but rather from reaction of phosphorus with calcium (Reactions (3) and (4)) (Wang et al., 2005). The lower NH_4^+-N removal efficiencies observed at higher Ca/Mg molar ratios also confirmed the effect of calcium on struvite formation. The NH_4^+-N removal efficiencies were found to be 42% for Ca/Mg molar ratio of 0.5 and 33% for Ca/Mg molar ratio of 2, which likely indicated less reaction of nitrogen with phosphorus at higher Ca/Mg molar ratios (**Fig. 3a**).

$$3PO_4^{3-} + 5Ca^{2+} + OH^- \longrightarrow Ca_5(PO_4)_3OH \quad (3)$$

$$2PO_4^{3-} + 3Ca^{2+} \longrightarrow Ca_3(PO_4)_2 \quad (4)$$

Investigations performed at constant Ca/Mg molar ratio (1) and variable pH (7.5–10) are also shown in **Fig. 3b**. Phosphorus removal efficiencies increased from 60% to 91% when pH increased from 7.5 to 9. Above pH of 9, phosphorus removal decreased. Nitrogen and calcium removal progressively improved as pH increased from 7.5 to 10. These higher nitrogen and calcium removal efficiencies (while magnesium removal was almost constant) most probably indicated NH_3 loss and calcium not only reacting with phosphorus but also with other substances present in the solution (e.g. carbonate). According to Pastor et al. (2010) and Le Corre et al. (2007), the possible precipitates that can appear when working with solutions containing calcium, magnesium, ammonia, phosphate and carbonate are struvite, hydroxyapatite, calcium phosphate, calcite ($CaCO_3$) and dolomite ($CaMg(CO_3)_2$).

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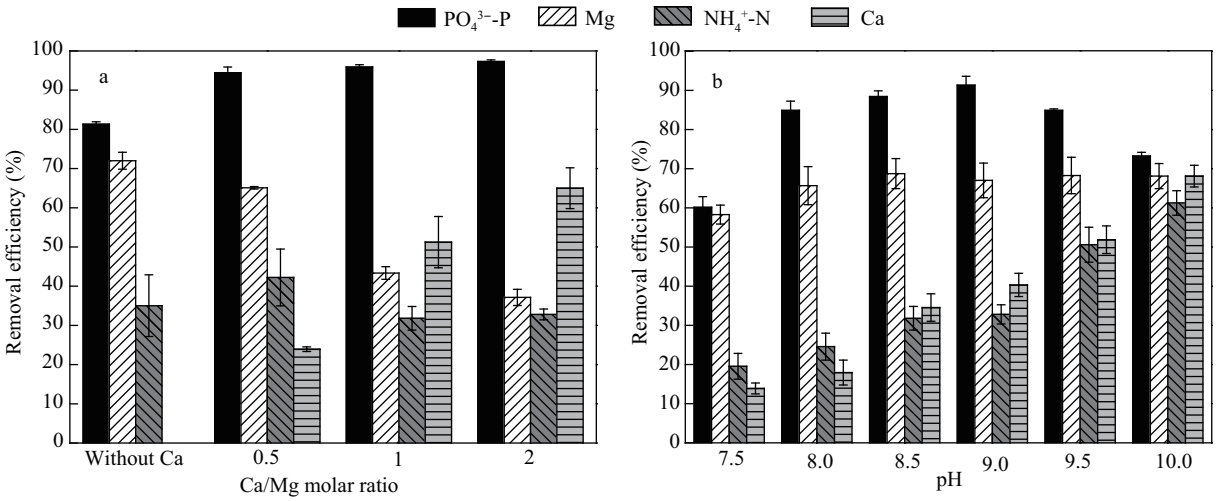

Fig. 3 Effects of Ca/Mg molar ratios on PO_4^{3-}-P and NH_4^+-N removal efficiencies of the FBR_{wc} for Run 1 at constant pH = 9 and different Ca/Mg molar ratios (a), and at constant Ca/Mg molar ratio (1) and different pH (b).

2.3 Crystal product characterization

One of the main advantages of removing and recovering phosphorus from wastewater is tackling eutrophication and its global stock depletion problems simultaneously. Hence, the efficiency of any reactor does not only depend on phosphorus removal but also on the quantity and quality (composition and morphology) of the final product recovered.

2.3.1 Chemical composition

To verify the composition of the crystals obtained in this study, samples were dissolved in nitric acid solution and analyzed for the content of struvite elements. In the absence of calcium, the elemental composition of the crystals was almost the same as the theoretical value (**Table 4**). Thus, the crystal composition results confirmed the product to be pure struvite (> 99%). Impurities (such as aluminum, calcium, iron, etc.) in the analyzed sample were found to be almost negligible, which implied the product could be used safely for agricultural purposes. Owing to its low solubility in pH-neutral solutions, struvite is an excellent slow-release fertilizer (Munch and Barr, 2001) that does not "burn" roots when over-applied, which is common with industrial ammonium-phosphate fertilizers (Shu et al., 2006). This slow-release behavior is ideal for agriculture, since it reduces nutrient run-off and thus reduces the impact of eutrophication in water bodies. Further, when struvite is used as a fertilizer, mining of phosphate rocks can be reduced (Pastor et al., 2010). Overall, recovering high-quality struvite could be a sustainable approach which deals with environmental, societal health and economic issues simultaneously.

2.3.2 Basic crystal morphology

Struvite is a white orthorhombic crystalline substance composed of magnesium, nitrogen and phosphorus in equal molar concentrations (de-Bashan and Bashan, 2004; Shu et al., 2006). The crystals produced in this study were white in color and were further confirmed as struvite by SEM. As shown in **Fig. 4a**, crystals produced under conditions of pH = 9, Mg/P = 1.75 and N/P = 7.5 molar ratios showed typical orthorhombic shapes as observed in other studies (Dunn et al., 2004; Wang et al., 2005). Moreover, the XRD pattern of most of the samples from this study fitted well with the position and intensity of the reference struvite pattern (**Fig. 4b**).

However, in the presence of high Ca/Mg ratios (⩾ 1), the SEM analyses showed relatively low crystal quality, which was consistent with our previous report (Guadie and Xia, 2012). As shown in **Fig. 4c**, the XRD results did not fit with the reference struvite, rather a broad amorphous peak was observed. Indeed, struvite growth inhibition was observed by Le Corre et al. (2005) and Pastor et al. (2008) at Ca/Mg molar ratios greater than 0.5 and 1.6, respectively.

Table 4 Measured elemental composition and theoretical values for struvite

Elements	Composition (mg/L)		Composition (%)	
	Measured	Theoretical	Measured	Theoretical
Mg	24.7 ± 1.2	24.5	9.9 ± 0.5	9.9
N	14.3 ± 0.8	14.3	5.7 ± 0.3	5.7
P	31.6 ± 0.3	31.6	12.6 ± 0.1	12.6
Struvite (estimated)			100	100

3 Conclusions

Enhanced struvite recovery from wastewater was achieved using a novel cone-inserted fluidized bed reactor. Cones

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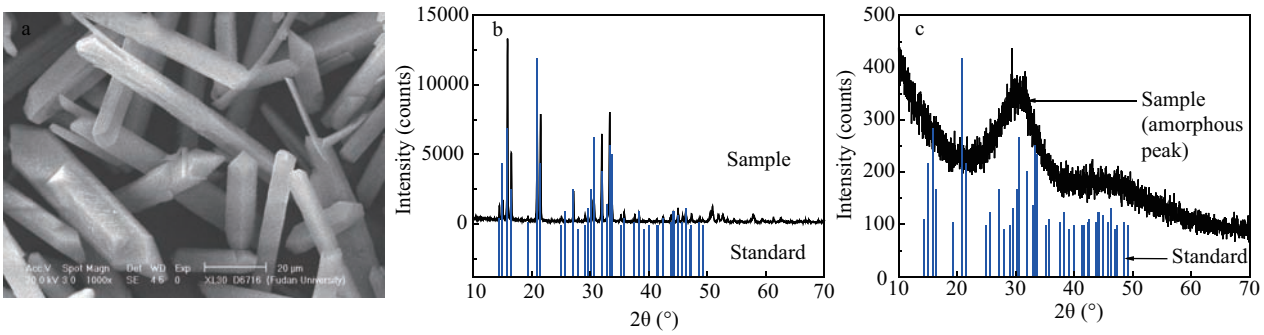

Fig. 4 (a) Scanning electron microscope image of a typical struvite, (b) X-ray diffraction (XRD) for sample and standard struvite peak without calcium and (c) XRD for amorphous product and standard struvite peak with calcium (Ca/Mg = 1).

were sandwiched at bottom, in the middle and in the top part of the FBR, and offered a special advantage in reducing unwanted crystal loss up to 67.5%. Under continuous operating conditions, the reactor showed promising removal and recovery efficiencies at low and high phosphorus concentrations. Phosphorus removal efficiencies were more than 90% under the optimal operating conditions (pH = 9, HRT = 2 hr, Mg/P = 1.25 and N/P = 7.5). The struvite recovery efficiencies for low and high phosphorous concentrations were about 75% and 92%, respectively. In the absence of calcium, the SEM, XRD and chemical composition results confirmed that the recovery product was almost pure struvite. Particularly, at high phosphorus concentration the struvite recovered showed superior quality compared to that at lower phosphorous concentration. The removal and recovery results of this study highlight the feasibility of the new FBR_{wc} for treatment of a wide range of phosphorus-containing wastewaters.

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