Water



Ethiopian Journal of Water Science and Technology

Special Issue

Proceeding of the 12th Symposium on Sustainable Water Resources Development Held at Arba Minch University from June 26-27, 2012 Arba Minch Proceeding of the 12th Symposium on Sustainable Water Resources Development (June 26-27, 2012)

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Evaluating Factors Affecting Phosphorus Removal and Recovery from Wastewater using Fluidized Bed Reactor

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Abstract

Phosphorus, an important element for life and fertilizer industry is stated to be exhausted shortly in the near future. On the contrary, large quantity of phosphorus in wastewater is considered to be one of the main causes of eutrophication which negatively affects many natural water bodies. Thus, phosphorus removal and recovery from wastewater as struvite is a more sustainable approach that consolidates these issues. Although struvite is a promising phosphorus recovery method, its application is still an infant stage. This is mainly due to various precipitation and crystal growth influencing factors. Therefore, the objective of this research was developed to evaluate the impact of different parameters affecting fluidized bed reactor performance of phosphorus removal and recovery from wastewater. By considering various conditions, laboratory experiments have been performed using appropriate tools and standard methods. The results showed that phosphorus removal was increased with increasing pH and Mg:P molar ratio. To achieve more than 90% of phosphorus removal efficiency, pH ≥ 9 and Mg:P molar ratio $\geq 1.25:1$ was required. Phosphorus removal efficiency was also enhanced when the fluidized bed reactor received feed from an external recycler. In order to achieve more visible precipitate and nearly 90% phosphorus removal efficiency, 12 hours contact time was also required. Furthermore, Scanning Electron Microscope, X-ray diffraction and chemical composition results revealed that the precipitate harvested in the fluidized bed reactor was struvite. The purity of the crystal qualifies the theoretical value of struvite. Overall, the results of contact time, pH, external recycler and Mg:P molar ratio were found to be the potential factors that affect the efficiency of phosphorus removal and recovery in fluidized bed reactor.

Keywords: Efficiency, Recovery, Removal, Struvite, Sustainable

1. Introduction

The simultaneous depletion of natural rock phosphorus (P) resource available for phosphate industry and the increasing awareness of pollution problems due to phosphorus discharge in wastewater effluent (Shu *et al.*, 2006) have led to research in to new process to remove and recover phosphorus from effluents.

Phosphorus which is vital to life and fertilizer industry is reported to be exhausted < 100 years (European Fertilizer Manufacturers Association, 2000; Isherwood, 2000; de-Bashan and Bashan, 2007) with a current exploitation rate of 1.5 % each year (Steen, 1998). It is estimated that there are 7000 million tons of phosphate rocks as P_2O_5 remaining in reserves that could be economically mined and human population consumes 40 million tons of P as P_2O_5 each year (Florida Institute of Phosphate Research, 2005; Steen, 1998). Moreover, 80 % of global phosphate reserve located only in four countries (Morocco, China, South Africa and United State of America) and unable to recycle in natural ecosystem more aggravate the question of phosphorus future sustainability (USGS, 2005; Mavinic, 2011).

Furthermore, the release of phosphorus to surface waters and its consequent contribution to eutrophication have led to increasing concerns about water quality sanitation. Policies are therefore being implemented globally to reduce the levels of phosphorus entering the surface waters from domestic and industrial wastewater for the betterment of ecological health. So far in traditional methods, phosphorus treatment was done fixing it with either by using chemical precipitation (using iron or aluminum salts) or utilizing enhanced biological phosphorus removal processes (Mulkerrins *et al.*, 2004). Both processes are efficient as they are capable of reducing phosphorus concentration in treated effluent below 1mgL⁻¹as required by most regulatory legislations (example, European legislation) (Adinan *et al.*, 2003).

However, the very tricky nature of traditional method is that after treatment has been made phosphate bioaccumulation and high sludge volume becomes a big problem (Woods *et al.*, 1999). The sludge wasted from biological nutrient removal, if anaerobically digested, will rehydrolyse the polyphosphates, consequently releasing magnesium and phosphate ions into the solution (Jardin and Popel, 1996). It is estimated that as much as 80–90% of phosphorus removed during treatment may be released and reintroduced to the process from the digester supernatants. This can lead to potential process failure (Mavinic *et al.*, 1998).

Currently, phosphorus removal and recovery from wastewater as viable sustainable approach gets the attention of many researchers (Driver et al., 1999; Greaves et al., 1999; Dastur, 2001; Burns and Moody, 2002; Doyle and Parsons, 2002, Adinan et al., 2003; Britton et al., 2005). In many developed countries, research is underway to recover phosphorus from the potential wastewater sources such as municipal, agricultural and other biological systems. Japan is a leader in establish full-scale Precovery plants and marketed to fertilizer industries (Doyle and Parsons, 2002). It is mentioned that recovering 10-80% of phosphorus from wastewater is economically feasible and the product quality is superior to currently available phosphate rock (de-Bashen and Bashan, 2007). The most promising compound for recovery of phosphorus from wastewater is struvite.

Struvite is an orthophosphate, containing magnesium, ammonium, and phosphate in equal molar concentrations. The general formula for struvite precipitation is given by equation 1 (with n = 0, 1, and 2) (Corre *et al.*, 2005; Fattah *et al.*, 2008)]:

$$\begin{array}{ll} Mg^{2+} + NH_4^+ + H_n PO_4^{n-4} + 6H_2 O \rightarrow MgNH_4 PO_4.6H_2 O + \\ nH^+ & (Equation \ 1) \end{array}$$

Struvite crystallization can be achieved with various plant configurations but fluidized-bed reactors (FBR) appear to be of particular interest (Dastur, 2001; Adinan, 2003; Britton *et al.*, 2005) and their utilization is widespread in the field of nutrient removal (Fujimoto *et al.*, 1991).

Although struvite crystallization is promising, phosphorus recycling from wastewaters has not been widely adopted. This is mainly due to a number of factors including pH control, magnesium concentration, retention time, presence of foreign ions and seed materials (Ohlinger, 1999; Corre *et al.*, 2005). Moreover, lack of proven technologies currently available for phosphorus recovery at low cost also a challenge (Yaffer *et al.*, 2002). The focus of this work was considering these issues.

2. Research Objectives

The main objective of this work was to **evaluate factors which affect the removal and recovery of phosphorus from wastewater using FBR**. In order to achieve this goal, FBR was designed and evaluated under different pH, Mg:P molar ratio and external pump flow rate.

3. Materials and Methods *Reactor design and operation*

To study struvite nucleation and growth, FBR was designed at Tongji University, College of Environmental Science and Engineering. The reactor has three parts made of transparent polyvinyl chloride plastic. The bottom, middle and top parts have different length, volume and diameter in which the later increased from bottom to top (Table 1 and Figure 1). The total volume and height of the reactor is about 9.5 liter and 1.1 meter.

Since the reactor design follows the concept of fluidized bed, the cross-sectional area and upflow velocity differ in the three parts (Table 1). Area and upflow velocity of the reactor was competed by applying equation 2 and equation 3 (Dastur, 2001). While the liquid moves up from bottom to top, upflow velocity were created which progressively decrease in FBR (Adinan *et al.*, 2003).

$$A = \pi r^2 \qquad (Equation \ 2)$$

$$Upflow \ velocity = \frac{Q}{A} \qquad (Equation \ 3)$$

Where,

A = area, r^2 = Radius and Q= Flow rate (in this case considered constant 72 ml⁻¹)

As part of FBR system, an external recycler (height 84 cm and volume 13 liter) constructed from the same transparent material and different tanks (60 liter each) were used during this study. Wastewater was fed into the bottom of the reactor along with the recycle stream using pumps which could be adjustable up to 100 revolutions per minute (rpm). Before, a continuous mode of operation was functional, the flow rate capacity of each pump was checked (1rpm=3ml⁻ min⁻¹) by allowing the liquid drop to measuring cylinder for one minute.

Parts	Length	Diameter	Volume	Area	Upflow Velocity
	(mm)	(mm)	(ml)	(mm ²)	$(mm^{-}min^{-1})$
Тор	220	170	4440	22686.5	3.2×10^{-3}
Middle	535	100	4142	7850	9.2x10 ⁻³
Bottom	340	60	884	2826	2.55×10^{-2}

Total volume = 9.5 L

Table 1: The dimensions and working condition of FBR



Figure 1: Fluidized bed reactor units

The whole system was operated in a continuous mode in that feed and NaOH supplied to the FBR at the bottom ports. Then, the liquid moves-up (fluidized) on different cross-sectional areas where crystals progressively getting larger and larger. As time elapsed, the bigger precipitates withstanding the turbulence get down and accumulated at the bottom section.

The liquid plus very fine precipitates were down flow from FBR to external recycler through plastic tube fitting. Finally, the system was allowed external recycler return portion of the effluent to FBR with pump and the remaining liquid volume to the supernatant tank connected by tube at the top part of the external recycler (Figure 1).

Chemicals

Synthetic feed water containing the constituent ions of struvite was used as an influent. The reason why we used synthetic waste is due to the need for optimizing the working condition of the new FBR.

The salts used to make the synthetic feed were commercial-grade magnesium chloride hexahydrate (MgCl₂.6H₂O), potassium dihydrogen phosphate (KH₂PO₄) and ammonium chloride (NH₄Cl). The feed was prepared by using tape water. Summary of synthetic feed and tape water constituent is given in Table 2.

pH control

Since struvite formation is highly dependent on pH in the reactor, commercial grade sodium hydroxide (NaOH) was separately prepared and feed to the FBR. However, different pH increasing methods were stated NaOH, selected in this study was due to its more effectiveness recommended by different scholars (Fujimoto *et al.*, 1991; Adinan *et al.*, 2003, Fattah *et al.*, 2008).We used low concentration of NaOH (0.0125N) based on our preliminary work of pH control achieved easily to the required range. When we used 0.1 and 0.2 N NaOH recommended concentration, manipulating the pump head to the required range founds difficult. Samples were taken at each part of the reactor zone and measured using pH meter. Whenever, pH deviation was observed, the pump head rpm was adjusted to raise or lower the value.

Sampling and analyses

Influent and effluent samples were collected daily for chemical analysis. The constituents of magnesium (Mg), ammonia-nitrogen (NH₄⁺-N) and orthophosphate (PO₄³⁻-P) were analyzed following standard methods. While NH₄⁺-N and PO₄³⁻-P were analyzed using UVvisible spectrophotometer (UV 1700, Shimadzu), magnesium was analyzed using flame atomic absorption spectrophotometry (PE-AA400, Perkin Elmer, USA).

The concentration of aluminum, calcium, iron, potassium and sodium was measured using an Inductively Coupled Plasma-Optimal Emission Spectrophotometer (ICP-OES) (ICP-720ES, Agilent, USA) (Table 2).

Table 2.Synthetic feed and tape water ICP-OES measured average results.

Table 2: Synthetic feed and tape water ICP-OES measured average results

	Feed				Tape water			
Chemical	KH ₂ PO ₄	MgCl ₂ .6H ₂ O	NH ₄ Cl	Ca	Al	Fe	Na	Κ
Value(mg ⁻ L ⁻¹)	55	62-145	160	2.65 <u>+</u> 0.75	*	*	13.2 <u>+</u> 0.05	2.6 <u>+</u> 0.01

Phosphorus removal efficiency

The primary objective of this research was evaluating the phosphorus removal efficiency of the FBR. For monitoring the removal efficiency, the idea of mass balance was used to compute removal percentage (Adinan *et al.*, 2001, Brittion *et al.*, 2005; Fattah *et al.*, 2008):

$$P-removal(\%) = \frac{(P_{ifluent} - P_{effluen})}{P_{influent}} \times 100 \quad (eq. 4)$$

Where *P* influent= Concentration of $PO_4^{3-}P$ at the inlet and *P*effluent = Concentration of $PO_4^{3-}P$ in the effluent

Crystal harvest procedure and analysis

Products were harvested from the reactor after the feed, recycle and chemical feed flows were stopped and then crystals were allowed to settle on the bottom part. To remove the crystals for harvesting, the valve was opened and the crystals were allowed to fall into a beaker. The harvested crystals were then air dried at room temperature before analysis.

Crystal product analyses

Samples of harvested crystals were dissolved in a 0.5% nitric acid solution to determine the composition and purity of the crystals grown in the reactor. These solutions were subsequently analyzed for the components of struvite, as well as calcium, aluminum, iron and potassium. For each sample analyzed, a 0.03g of precipitate was dissolved in 50 ml of 0.5% nitric acid solution (Fattah *et al.*, 2008).

Morphology of the harvested crystals was examined using Scanning Electron Microscope (SEM) (XL30, Philips, the Netherlands) and X-ray Diffraction (XRD) (D/ MAX-rB, Rigaku, Japan).

3. Results and Discussions

The main objective of this study was constructing reactor working an excellent p-removal which is applicable for wastewater treatment. The reactor operational conditions and summary of the result is given in Table1 and Table 3. The description of each run is given in the following sections.

Parameters	Run1	Run 2	Run 3	Run 4
Influent PO_4^{3-} -P concentration (mg·L ⁻¹)	19.98~21.79	21.01~21.9	19.89~21.9	18.30~21.87
Effluent $PO_4^{3-}P$ concentration (mg ⁻ L ⁻¹)	1.41~7.48	1.41~21.00	0.87~9.46	1.48~7.07
P-Removal efficiency (%)	65.1~93.5	70.77~93.46	48.71~95.96	54.85~93.23
Mg:P molar ratio	1.25:1	1.25:1	1.25:1	0.75-1.75:1
N:P molar ratio	7.44:1	7.44:1	7.44:1	7.44:1
Temperature (°C)	21~25	20-25	20-23	21-26
Conductivity (mS ⁻ cm ⁻¹)	0.87~0.93	0.75~0.90	0.84~0.93	0.68~0.87

Table3: Range of the operational conditions and summary of results of the FBR

Run 1= Recycler supply, Run 2= Contact Time, Run 3= pH, Run 4=Mg:P molar ratio

Reactor performance

A preliminary FBR design was made and evaluated for its precipitation potential. After we made minor modification, we came up the best design of FBR which had an efficient precipitation potential of phosphorus without wasting at each junctions that was the problem for the first reactor we made (Figure 2A).

As shown in Table1, the reactor design purposely

varied the area. The rationale behind we employed the varying cross-section areas and, hence, the different upflow velocities was to avoid the washout of the tiny crystals in the effluent. The high upflow velocity is in the bottom section of the reactor which progressively decreases to top. As the crystals increase in size, they are able to overcome the higher upflow velocities and move towards the lower section. Different studies said that higher upflow velocity at the bottom section enhance crystal growth because of the high turbulence (Ohlinger, 1999; Dastur, 2001).



A) Precipitation in FBR **Figure 2:** Struvite precipitation in FBR

B) Precipitation in FBR with Recycler

We collected products (precipitates) at the end of every experimental runs. Most of the time products were collected after 3-4 days. The settle bed height was measured from the bottom section of FBR before the product was drawn. The bed height results were found variable (6.25-15 cm) depending on conditions considered. We obtained the minimum bed height when the system runs without external recycler and at lower pH and Mg:P molar ratio (Data not shown).

As visually seen in Figure 2B, the reactor precipitation potential becomes more enhanced when the external recycler employed in the system. This is because of wasted fine particle from the FBR in the first pass get a chance to stay in the recycler and return back to the reactor. Phosphorus removal efficiency with and without external recycle also investigated (Figure 3) to confirm this issue. The percentage p-removal efficiency improved from 72 to 93% when the volume of liquid from external recycler returned increased from 15 to 45 ml (1rpm=3ml per minute). However, removal efficiency increased with increasing volume of return, the visible precipitate was not encouraging when recycle pump rpm \geq feed pump rpm. We critically observed very promising precipitate when our FBR received equal amount of liquid from feed and recycle. This might be due to the dilution effect which inhibits crystal growth ..



Figure 3: Effect of Recycle on P-removal

Effect of contact time on p-removal

The detention time required to fill the whole volume of FBR (V= 9466 ml) is about 130 minutes (2:20 hours) at flow rate of 72 ml min⁻¹. The external recycler with a volume of 12970 ml capacity also need 180 minutes (3:00 hours) to be full and released its effluent to the super-

natant tank. In general, both (FBR and Recycler) container to be fully operational until effluent flow to the supernatant tank, 5:20 h total hydraulic retention time was required. Often, visible precipitates were formed at the middle part of the reactor after 3:30-4:00 h.

In order to investigate contact time as a factor of premoval, analysis was done by taking sample at different interval (Figure 4). To achieve more than 90% of premoval, the reactor required ≥12 hours. P-removal was also greater than 80% after 6 h but efficiency was low before this time. Moreover, having more elapsed time (48 hours) did not show significant removal efficiency but crystal bed volume growth become high. It is therefore, important to consider time factor to achieve very good removal efficiency and crystal growth.

The current result agrees with results obtained in other studies (Adinan *et al.*, 2003; Britton *et al.*, 2005; Huang *et al.*, 2006; Liu *et al.*, 2008). According to Liu *et al.*, (2008) mentioned that the reactor they constructed enhanced phosphorus recovery with increasing total hydraulic retention time. They also noted that further increasing highly improved reduction of effluent concentration.



Figure 4: Effect of contact time on p-removal

The effect of pH on p-removal

As shown in Figure 5, experiment without and with NaOH supply was made. The pH value investigated here ranged 7-10 which was made by adjusting the flow rate of NaOH pump. To achieve over 90% P-removal, the operating pH had to be raised to 9. Compared to without NaOH adjustment (<50%), the efficiency of p-removal increased progressively from 55 to 96% with adjusting the pH from 7 to 10.

Similar studies were made on pH by other researchers (Ohlinger, 1999; Celen and Turker, 200; Munch and Barr 2001; Stratful *et al.*, 2001). All these studies have shown that there is an increase in P-removal with an increase in pH as we succeeded. They also stated struvite is soluble at acidic pH conditions and highly insoluble at alkaline pH.

The effect of Mg:P molar ratio on p-removal

It is stated in many literatures that high premoval accompanied with struvite formation required a theoretical Mg:N:P molar ratio of 1:1:1.Therefore, magnesium ion supplementation of at least the stoichiometric requirement would be required to ensure that magnesium ions did not become a limiting factor (Adinan *et al.*, 2003). However, this does not imply that the process of struvite crystallization and p-removal can proceed only if Mg:P has a molar ratio of 1:1.We achieved almost 55% of p-removal without any addition of magnesium in the system.

As shown below in Figure 6 at lower Mg:P molar ratio (0.75), removal efficiency was low (78%). When the Mg:P molar ratio increases from 0.75 to 1.75, the FBR P-removal improved from 78 to 93%. The reactor also achieved more than 80% of P-removal at theoretical (1:1) Mg:P molar ratio. After 90% removal efficiency achieved at 1.25:1 Mg:P molar ratio further increasing the ratio (1.75:1) did not show significant percentage reduction, however, the condition contribute lower effluent concentration (< 1.5 mgL⁻¹) in the reactor.



Figure 5: Effect of pH on P-removal



Figure 6: Effect of Mg:P ratio on P-removal

This study result is in consistent with other studies (Hirasawa *et al.*, 1997; Munch and Barr, 2001; Stratful *et al.*, 2001; Huang *et al.*, 2006). They stated as an increased in Mg:P molar ratio increase the P-removal efficiency.

Struvite crystal composition

To verify the composition of the crystals grown in this study, 6 samples were analyzed. Table 2 shows the average crystal compositions compared with the expected theoretical composition of pure struvite. Results showed that the crystal contains struvite reacting species more than the theoretical value. Deviation of the results was observed which could be errors during analysis. However, the deviation does not mean the product do not qualify struvite property rather indicates our FBR was able to produce high-grade crystals. Adinan *et al.*, 2003 achieved 99.8 % of crystal purity. The crystal samples were also analyzed for content of Fe, Al, K, and Ca. The main impurity found in the crystals was Ca followed by Na and Al (data not shown).

Basic crystal morphology

In this study, precipitates with various size, density and hardness were harvested. In order to understand whether the precipitate formed was struvite or not, products were observed under the Scanning Electron Microscope (SEM). The results showed that products collected at various conditions have different morphology. Some of the precipitates were found amorphous in shape and unacceptable as struvite (Figure 7A). The amorphous shape was resulted when the system was operated at lower pH and Mg:P molar ratio. When the pH in the system was operated above 9 and the molar ratio of Mg:P \geq 1:1, the crystal product was very hard (Figure 7 B) which qualify the rhombic shape of struvite. These struvite products were confirmed with X-ray Diffraction analysis (Figure 8).

Struvite reacting species	Weight (mg^{-1})		Composition (%)		
/Element	Theoretical	Measured	Theoretical	Measured	
Mg	24.5	25.550 <u>+</u> 1.265	9.9	10.4	
Ν	14.3	14.525 <u>+</u> 17.160	5.7	5.9	
Р	31.6	31.709 <u>+</u> 3.223	12.8	12.9	
Struvite (estimated)			~100	~100	

Table4: average results of purity composition analysis and theoretical values (Dastur, 2001; Fattah et al., 2008)

4. Conclusions

Phosphorus is the growth limiting factor in aquatic ecology which causes eutrophication. Its source is direct disposal of untreated effluent to water bodies. Currently, for the betterment of environmental and aquatic health stringent laws are set for nutrient (phosphorus) reduction in wastewater. On the other hand, global phosphate reserve depletion leads a question of sustainability which forces the modern society to think phosphorus recovery instead of removal from wastewater. The present study was considering both issues and results were found promising even at lower PO_4^{3-} -P concentration (18.30-21.90 mg·L⁻¹). As to our current knowledge, (except Liu *et al.*, 2008 did at 21.7 mg·L⁻¹ PO₄³⁻-P concentration and

achieved 78% p-removal efficiency) no one tried to recover phosphorus at lower concentration and achieved high removal efficiency.

The new FBR we constructed effectively remove and recovery phosphorus from synthetic wastewater. Performance of the reactor was optimal (≥ 90 %) when higher pH (≥ 9) and Mg:P molar ratio (≥ 1.25 :1) used. The reactor efficiency was also enhanced when higher recycle flow rate and longer contact time maintained in the FBR. The SEM, XRD and chemical composition results showed that the crystal harvested qualify the characteristic of struvite. The overall finding showed that FBR will be successfully used in phosphorus removal and recovery (in the form of struvite) from wastewater by monitoring pH, Mg: P molar ratio, time and recycle conditions.



A. Amorphous Precipitate



B) Struvite





Figure 8: Struvite sample and standard XRD results

Acknowledgement

The authors would like to thank Tongji University and Chinese Government Scholarship for providing financial support. This work was supported by the National High Technology Research and Development Program of China (No.2009AA062902),National Water Pollution Control Program (No. 2009ZX07317-008-5), and Shanghai Shuguang Tracking Program (No. 10GG12).

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