

Effect of Silica Sand Addition in Fluidized Bed Reactor on Productivity and Efficiency of Struvite Crystal Formation

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Abstract

This research is motivated by liquid waste containing phosphate and ammonium, which can pollute waters. Struvite crystallization in a fluidized bed reactor is a solution because it can reduce pollutants and produce fertilizer, but still needs to increase efficiency and productivity. Struvite itself is an environmentally friendly slow-release fertilizer. This study aims to examine the effect of the addition of silica sand on the efficiency and productivity of struvite formation. Uniform the size of silica sand using a ball mill operated for 30 minutes at 80 rpm, then sieved using mesh 50. Na₂HPO₄ as a phosphate source, NH₄Cl as an ammonium source, and MgCl₂ as a Magnesium source. NaOH or HCL as acid base solution. There are two factors, namely the addition of silica and fluid flow velocity. The analysis carried out is the productivity and efficiency of struvite crystal formation. The diameter of silica sand is $2,97 \times 10^{-4}$ m. The maximum and minimum velocities of fluid flow with the addition of silica sand are 0.0034 m/s and 0.0102 m/s. The highest productivity and efficiency were 121.9 g/L and 88.15% with the combination of silica sand addition factor and flow velocity of 0.0034 m/s. The addition of silica sand and using minimum speed in the struvite crystal formation process can increase the productivity and efficiency of struvite crystals. Flow velocity determines the transportation of ions to the crystal surface, the maximum speed allows the newly formed crystal nucleus to break.

Keywords: *Fluidized Bed Reactor, Productivity, Silika Sand, Struvite*

Introduction

The global need for sustainable agricultural practices and environmental protection is increasingly driving attention to nutrient recovery from wastewater, particularly phosphorus-an essential but limited element. One promising method to recover phosphorus is through the crystallization of struvite (MgNH₄PO₄·6H₂O), a compound that can be used as a slow-release fertilizer. The use of struvite is not only an environmentally friendly alternative to fertilizer, but also helps reduce nutrient pollution that can cause eutrophication in water bodies. (Ariyanto et al., 2025).

Among the various reactor configurations used, the fluidized bed reactor (FBR) showed superior performance in the struvite crystallization process. This is due to the FBR's ability to provide even mixing, high mass transfer rates, and the potential for continuous operation (Ha et al., 2024; Yan et al., 2024). However, one of the main challenges in this process is controlling the nucleation and crystal growth stages, which

directly affects the yield, purity and size distribution of the resulting struvite crystals.

The use of seed materials such as silica sand has been proposed as an effective strategy to improve crystallization efficiency. Silica sand has chemically inert properties, a suitable specific gravity for fluidization, and a large rough surface that can act as a starting place for crystal formation (heterogeneous nucleation). This is believed to accelerate the crystal growth process, reduce scaling on the reactor walls, and increase product separation efficiency. In addition, the presence of silica sand can also affect the stability and hydrodynamic characteristics of the reactor system, which ultimately impacts the overall process efficiency (Nadagouda et al., 2024; Sinharoy et al., 2024).

Despite the promising role of silica sand, in-depth studies on the quantitative effect of silica sand addition on the struvite crystallization process in FBR systems are still limited, especially in relation to the optimization of operating parameters to achieve maximum results. A better understanding of this interaction is crucial for the development of large-scale FBR technology in wastewater treatment and nutrient recovery systems. Based on this, this study aims to examine the effect of the addition of silica sand in the fluidization reactor on the productivity and efficiency of struvite crystal formation, focusing on the analysis of crystal yield, namely efficiency and productivity. The results of this study are expected to contribute to the development of a more efficient and sustainable struvite recovery system in the future.

Research Method

This study used a laboratory experimental approach with a factorial design to examine the effect of silica sand addition and fluid flow velocity on the productivity and efficiency of struvite crystal formation in a fluidization reactor. The research was conducted in the agroindustry process engineering laboratory from September 2024 to April 2025.

Materials and Preliminary Preparation.

The silica sand used as seed material in the crystallization process was prepared first to obtain a uniform particle size. The pulverization process was carried out using a ball mill operated for 30 minutes at a speed of 80 rpm, then the results were filtered using a mesh 50 sieve to obtain the desired particle size distribution. The chemicals used in this study include Na_2HPO_4 as a source of phosphate ions (PO_4^{3-}), NH_4Cl as a source of ammonium ions (NH_4^+), MgCl_2 as a source of magnesium ions (Mg^{2+}), as well as, NaOH or HCl for pH adjustment of the solution according to crystallization needs.

Research Variables

There are two main variables tested in this study, namely the addition of silica sand (with and without addition), and the fluid flow velocity in the reactor (with a certain value variation with units of m/s.). The response variables observed were struvite crystal productivity (gram product per liter) and struvite crystal formation efficiency.

Experiment Procedure

The reactant solution was prepared by mixing MgCl_2 , NH_4Cl , and Na_2HPO_4 solutions in stoichiometric concentrations of 1:1:1. The reaction was carried out in a fluidization reactor with or without silica sand media, at various flow rates. The pH of

the solution was adjusted using NaOH or HCl to maintain optimal conditions for crystallization (pH 8-9). The reaction was run for 15 minutes, then the product was collected, dried, and weighed.

Data Analysis Technique

Data analysis was carried out quantitatively to calculate crystal productivity, calculated from the mass of crystals obtained per volume of solution. Crystallization efficiency, calculated from the ratio of mol/min of the number of struvite crystals in the reactor compared to mol/min of the total number of struvite crystals in the reactor and those blown out of the reactor.

Results and Discussion

Calculation of minimum and maximum fluid flow velocity

The seed material used is silica sand with a size of 50 mesh. calculations to determine the minimum fluxation speed (v_{mf}) and maximum fluxation speed (vt). The following is the calculation of the minimum fluxation speed (v_{mf}) and maximum fluxation speed (vt).

$$\psi = 0,75 \text{ (sand average)}$$

$$\rho_b = 2650 \text{ kg/m}^3$$

$$dp = 0,297 \text{ mm} = 2,97 \times 10^{-4} \text{ m (No. Sieve: 50)}$$

$$\rho_f = 1000 \text{ kg/m}^3$$

$$\mu = 8,91 \times 10^{-4} \text{ kg/m.s (at } 25^\circ\text{C)}$$

- Maximum Fluidization Velocit (ϵ_{mf})

$$\epsilon_{mf} = 0,586 \times \psi^{-0,72} \times \left(\frac{\mu^2}{\rho_f \times \eta \times dp^3} \right)^{0,029} \times \left(\frac{\rho_f}{\rho_b} \right)^{0,021}$$

$$\eta = g(\rho_b - \rho_f)$$

$$\eta = 9,8(2650 - 1000)$$

$$\eta = 9,8(1650)$$

$$\eta = 1670 \text{ kg/m}^2 \cdot \text{s}^2$$

$$\epsilon_{mf} = 0,586 \times 0,75^{-0,72} \times \left(\frac{(8,91 \times 10^{-4})^2}{1000 \times 1670 \times (2,97 \times 10^{-4})^3} \right)^{0,029} \times \left(\frac{1000}{2650} \right)^{0,021}$$

$$\epsilon_{mf} = 0,5886$$

- Minimum Fluidization Speed (v_{mf})

$$v_{mf} = \frac{(\psi dp)^2}{150\mu} \times \eta \times \frac{\epsilon_{mf}^3}{1 - \epsilon_{mf}}$$

$$v_{mf} = \frac{(\psi dp)^2}{150\mu} \times g(\rho_b - \rho_f) \times \frac{\epsilon_{mf}^3}{1 - \epsilon_{mf}}$$

$$v_{mf} = \frac{(0,75 \times 2,97 \times 10^{-4})^2}{150 \times 8,91 \times 10^{-4}} \times 1670 \times \frac{0,6157^3}{1 - 0,6157}$$

$$v_{mf} = 0,00297 \text{ m/s}$$

- vt value using the formula wh $0,4 < Nre < 500$

$$vt = (1,78 \times 10^{-2} \times \eta^2 / \rho_f \times \mu)^{1/3} \times dp$$

$$vt = (1,78 \times 10^{-2} \times 1670^2 / 1000 \times 8,91)^{1/3} \times 2,97 \times 10^{-4}$$

$$vt = 0,0489 \text{ m/s}$$

- Check Nre

$$Nre = \frac{dp \times \rho_f \times vt}{\mu}$$

$$Nre = \frac{2,97 \times 10^{-4} \times 1000 \times 0,02916}{8,91 \times 10^{-4}}$$

$$Nre = 16,31 \text{ (OK, because in the range } 0,4 < Nre < 500)$$

Based on the above calculations, it is concluded that the u_{mf} (minimum fluid velocity) and u_t (maximum fluid velocity) values for seed material with a size of 50 mesh are 0.00297 m/s and 0.0489 m/s. The variables used in the flow velocity must fall within the range of u_{mf} (minimum fluid velocity) and u_t (maximum fluid velocity) values. So for the three flow velocity variables taken are 0.0034, 0.0068 and 0.0102 m/s. These speeds are taken because they can represent the smallest, medium and largest fluid flow speeds.

Productivity and efficiency of Struvite Crystals Using Fluidized Bed Reactor.

The table 1 presents the productivity data of a fluidized bed reactor in the formation of struvite crystals, both with and without the use of a seeding material, specifically 50 mesh silica. The experiments were conducted at three different flow velocities: 0.0034 m/s, 0.0068 m/s, and 0.0102 m/s, using varying volumes of wastewater. Without silica, the weight of the struvite product ranged from 226.00 grams to 567.00 grams, with the highest productivity being 44.50 grams/L at a flow velocity of 0.0068 m/s. In contrast, when 50 mesh silica was used as a seeding material, there was a significant increase in productivity. The product weight increased up to 1069.20 grams, and the highest productivity reached 121.90 grams/L at the lowest flow velocity of 0.0034 m/s. This indicates that the addition of silica as a seed material greatly enhances the crystallization efficiency. Although flow velocity does have an impact on the outcome, its influence is not as substantial as that of the silica. Therefore, the use of 50 mesh silica as a seeding material in a fluidized bed reactor is proven to be effective in significantly improving the formation of struvite crystals.

Table 1. Productivity of Fluidized Bed Reactor in Struvite Crystal Formation

No	Seed Material	Flow Velocity(m/s)	Weight of Product Obtained (gr)	Waste Water (L)	Productivity (gr product/L)
1	Without Silica	0,0034	226,00	6	37,67
2		0,0068	534,00	12	44,50
3		0,0102	567,00	18	31,50
4	Silica 50 Mesh	0,0034	731,40	6	121,90
5		0,0068	1038,46	12	86,54
6		0,0102	1069,20	18	59,40

Table 2 presents the efficiency of struvite crystal formation in a fluidized bed reactor, considering two main parameters: W grains and W bottom. W grains represents the percentage of struvite crystals that successfully formed and remained inside the reactor, while W bottom indicates the total percentage of struvite formed, including both those retained in the reactor and those ejected from it. In experiments without the use of silica as a seeding material, the efficiency of struvite formation was very low, with total efficiency ranging only from 2.52% to 13.27%. This suggests that most of the struvite was not retained within the reactor. In contrast, the addition of 50 mesh silica as a seeding material significantly increased the efficiency. At a flow velocity of 0.0034 m/s, the efficiency reached 88.15%, with 31.30% of the crystals retained inside the reactor and a total formation of 35.51%. Similar improvements were observed at higher flow velocities, with efficiencies of 84.72% at 0.0068 m/s and 80.77% at 0.0102 m/s. These results demonstrate that the use of 50 mesh silica not only enhances the formation

of struvite crystals but also improves their retention within the reactor, thereby significantly increasing the overall efficiency of the crystallization process.

Table 2. Fluidized Bed Reactor Efficiency in Struvite Crystal Formation

No	Seed Material	Flow Velocity (m/s)	Struvite (mol/minutes)		Efficiency
			W grains	W bottom reactor	
1	Without Silica	0,0034	1,46%	10,97%	13,27%
2		0,0068	1,01%	25,93%	3,88%
3		0,0102	0,69%	27,53%	2,52%
4	Silica 50 Mesh	0,0034	31,30%	35,51%	88,15%
5		0,0068	42,70%	50,40%	84,72%
6		0,0102	41,93%	51,91%	80,77%

The productivity and efficiency of struvite crystal formation in a fluidized flow reactor are strongly influenced by the presence of seed material and fluid flow velocity. The addition of silica as a seed material is proven to significantly increase productivity compared to conditions without silica. Scientifically, this is because silica provides an active surface that serves as a heterogeneous nucleation point, which facilitates the formation of crystal nuclei from the solution (Yan et al., 2024). Heterogeneous nucleation requires lower energy than homogeneous nucleation, so crystallization can take place more quickly and efficiently (Guan et al., 2023). In addition, the silica surface can help stabilize crystal growth as well as keep the crystals in the system to grow larger before they are finally separated. As a result, the number of crystals formed and the size of the crystals produced are optimized, increasing overall productivity and efficiency (Tang, 2016; S. Wang et al., 2023)

Meanwhile, the flow velocity in the reactor also has a major effect on productivity. At low to medium flow velocities, the residence time of the solution in the reactor is long enough to allow the nucleation process and crystal growth to take place properly. However, at excessively high flow velocities, the residence time of the solution becomes shorter so that the crystallization process does not take place optimally. In addition, high velocities can cause large shear forces in the reactor, which runs the risk of damaging newly formed crystals or causing them to detach before reaching the optimal size (Tarragó et al., 2016). This can reduce the efficiency of precipitation and crystal collection. Thus, too high a flow velocity decreases reactor productivity and struvite crystal formation efficiency, both under conditions with and without seed material. Therefore, an optimum flow velocity is required in order to strike a balance between complete mixing and adequate retention time for truvite crystal growth (Wang et al., 2023).

Conclusion

The conclusions of this study are 1). The use of trigger material in the form of 50 mesh silica in a fluidized bed reactor is proven to significantly increase the productivity and efficiency of struvite crystal formation. Without silica, the maximum productivity only reaches 44.50 grams/L, while with silica it can increase to 121.90 grams/L at the lowest flow rate (0.0034 m/s). In addition, the total efficiency of struvite formation without silica was very low (2.52%-13.27%), but increased dramatically to 88.15% with the addition of silica, accompanied by an increase in crystal retention in the reactor. 2). Flow velocity affects the productivity and efficiency of struvite crystal formation in fluidized bed reactors, where the lower the flow velocity, the higher the productivity

and efficiency. The addition of trigger material in the form of 50 mesh silica significantly increases the amount and efficiency of crystal formation. 3). The best combination was obtained at a flow velocity of 0.0034 m/s with the addition of 50 mesh silica, resulting in the highest productivity of 121.90 grams/L and a total efficiency of up to 88.15%. This indicates that the combination is most effective in optimizing the struvite crystallization process.

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