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Modelling, Design And Evaluation Of A Continuous Poiseuille Flow Struvite Seed Crystallizer

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Summary of key findings

Poiseuille flow combined with rapid mixing provides a low pressure drop method of continuous seed production. Magnitude and variance of distribution width and volume median diameter (D[50]) measured in line (IL) increase with initial saturation index (SI), whereas filtered and re-suspended samples show little change. These effects are greater for the impinging jet (IJ) mixer than the Roughton (R) mixer. This illustrates that incomplete mixing before nucleation promotes formation of unstable aggregates, creating a more variable product. The IJ mixer also produced a lower phosphorus recovery, which may be attributed to SI gradients caused by incomplete mixing. Differences in mass recovery measurements indicate that scaling is an issue and needs to be further investigated.

Background and relevance

Phosphorus is essential for all food production and global phosphate rock reserves are declining in quality and quantity. Phosphate pollution into waterways from wastewater can cause eutrophication and hypoxia. Fluidized Bed Reactors (FBRs) are common for struvite crystallization (Iqbal, Bhuiyan, & Mavinic, 2008; Shimamura, Ishikawa, Tanaka, & Hirasawa, 2007). FBR operating conditions have been widely discussed but physical processes have not been described in detail. Large pellets often produced in FBRs show that agglomeration is a key growth mechanism. Agglomeration is most likely size dependent as particle size affects velocity, momentum and probability of collision. Understanding how the size of particles entering the fluidized zone is affected by saturation will contribute to understanding agglomeration. Continuous or semi-batch seed production is one way to control particle size entering the FBR (Shimamura et al., 2007). No other work to date has been published investigating continuous struvite seed production. This study investigates how SI and mixing regime affect particle size produced in a tubular reactor. Poiseuille flow is used to achieve a significant residence time and reduce pressure drop over the reactor. Reactor design is informed by modelling of rapid mixing, water chemistry and convective flow. The Poiseuille flow reactor (Figure 1.1) was oriented vertically downwards. P concentration was 0.02M, Mg:N:P was 2:1:1 and NaOH was dosed to achieve oversaturation. Two diaphragm pumps delivered 4L/h of each feed. Mixing was achieved using impinging jets (IJ) and a Roughton (R) mixer. Particle size distribution (PSD) was measured by Laser Diffraction (LD), both in-line (IL) and after filtration and sonication (FS). Outlet pH was measured and crystals were filtered and examined with XRD and microscopy. Filtrate was analysed using spectrophotometry and ICP-OES.

Results

XRD patterns match a pattern given for struvite and ICP-OES showed that primarily struvite had been formed. Experimental determination of the residence time in the reactor showed a value 16% below that theoretically expected, indicating the possible existence of dead zones. Tracer studies showed that particle settling resulted in a 9.6% reduction between hydraulic and crystal residence time and caused a significant degree of PSD spreading. This spreading effect is likely to be less significant during experiments as tracer studies used full sized crystals, whereas during experiments, crystals are smaller earlier in the reactor.

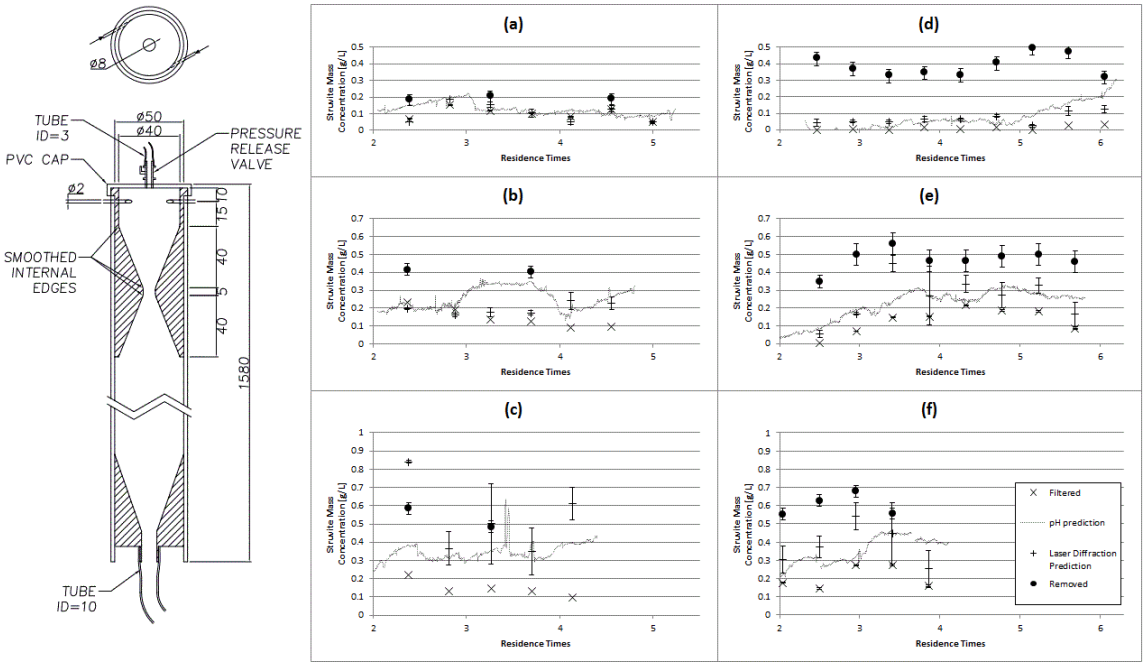

Figure 1.1 – Left: Poiseuille flow crystallizer with Roughton mixer. Right: struvite mass concentration from Poiseuille Flow Reactor using (a) IJ mixer, SI=0.8; (b) IJ mixer, SI=1.0; (c) IJ mixer, SI=1.4; (d) R mixer, SI=0.8; (e) R mixer, SI=1.0; (f) R mixer, SI=1.4.

Measured mass concentration leaving the reactor is significantly less than the amount removed based on residual P concentration, indicating that scaling was an issue (Figure 1.1). LD and pH based mass concentrations lay in the expected range but exhibited high variability. It was also found that particle mass was lost during PSD measurement after re-suspension. Fractional recovery is relatively independent of initial SI at SI=1.0 and 1.4 but lower at SI=0.8. This is true for both mixers. The R mixer achieves greater recovery than the IJ mixer under all conditions.

Table 1.1 -PSD characteristics for Poiseuille flow reactor. R=Roughton; IJ=impinging jet; IL=In-line; FS=Filter and Sonicate. *acceptable samples exclude blockages and LD flow cell scaling.

SI	Mixer Type	Sample Method	Acceptable Samples*	Distribution Width (D[90]-D[10]) [µm]		Volume Median Diameter (D[50]) [µm]		Steady State Filtered Mass Conc. [g/L]		Steady State Obscuration Mass Conc. [g/L]		Steady State pH Based Mass Conc. [g/L]		Steady State Filtrate Based Mass Conc. [g/L]	
				µ	σ	µ	σ	µ	σ	µ	σ	µ	σ	µ	σ
0.8	R	IL	40	58.2	11.2	33.5	0.2	-	-	0.104	0.047	-	-	-	-
	IJ	IL	40	95.9	26.2	44.2	8.1	-	-	0.271	0.053	-	-	-	-
	R	F/S	9	59.1	9.6	30.8	3.7	0.013	0.012	0.068	0.033	0.076	0.079	0.377	0.065
	IJ	F/S	5	52.4	5.5	28.5	2.9	0.100	0.036	0.104	0.056	0.123	0.040	0.189	0.012
1	R	IL	50	111.3	46.3	50.1	8.3	-	-	0.272	0.197	-	-	-	-
	IJ	IL	60	166.5	58.5	76.9	31.8	-	-	1.043	0.910	-	-	-	-
	R	F/S	8	55.2	19.8	25.7	4.6	0.130	0.071	0.255	0.123	0.234	0.071	0.474	0.060
	IJ	F/S	7	59.2	4.0	34.2	2.0	0.146	0.056	0.196	0.032	0.262	0.067	0.410	0.010
1.4	R	IL	30	170.5	89.0	66.6	23.0	-	-	0.562	0.863	-	-	-	-
	IJ	IL	30	189.1	62.9	90.1	34.1	-	-	1.472	1.316	-	-	-	-
	R	F/S	6	60.6	8.1	31.5	5.1	0.205	0.063	0.384	0.115	0.365	0.078	0.604	0.061
	IJ	F/S	6	67.4	3.1	35.0	1.8	0.147	0.045	0.534	0.202	0.366	0.032	0.535	0.071

Filtered samples treated with ultrasound show insignificant changes in distribution width or volume median diameter (D[50]) with SI. Microscopy on crystals not sonicated shows some aggregates with small contact area. Increased SI causes increased distribution width and D[50] for samples measured IL (Table 1.1). At higher SI these increments are less prominent and width and D[50] variance increases.

The R mixer produced particles with a lower D[50] than the IJ mixer at almost all SI. Variance of D[50] measured IL in the R mixer increases with SI. This may be because aggregation and saturation gradients increase with saturation. IL distribution width increase between R and IJ mixer is 65%, 50% and 11% for SI=0.8,1.0 and 1.4 respectively. The variance of distribution width between the two mixers more than doubles between SI=0.8 and 1.0, and increases by 46% between SI=1.0 and 1.4, showing that distribution widths are more variable at higher SI.

Discussion

Design constraints on reactor length and width implemented to achieve in line PSD measurement mean that Poiseuille flow may not have become fully developed, as seen by differences in theoretical and measured reactor residence time. Future investigations will improve this issue using increased saturation to achieve faster crystallization and sample quenching rather than continuous PSD measurement. Variability in pH and LD mass concentration measurements is expected to be due to probe scaling and high particle aspect ratio. An alternative to LD which may overcome uncertainty due to high aspect ratio is single particle optical sizing (SPOS). In-line PSD measurements were also found to deviate from reality due to flow cell scaling over time – only selected data could be examined. Differences between sonicated and in-line sampling techniques show that aggregation played a significant role in particle size enlargement. While this was not intended, results may be analysed using a population balance incorporating aggregation to estimate rate coefficients.

The R mixer achieves greater mass removal at all initial SI values, especially SI=0.8. Poorer recovery in the IJ mixer at SI=0.8 implies insignificant nucleation, a result of poor mixing and low saturation. The R mixer achieves complete mixing before induction time, while the IJ mixer does not. This results in even nucleation and a greater surface area for subsequent crystal growth. Final saturation in the IJ mixer was SI=0.2 for initial saturations of SI=1.0 and 1.4. Crystal growth rate has been observed to decrease significantly below SI=0.2, creating an apparent growth dead zone (S C Galbraith, 2011).

The R mixer also produced a lower IL D[50] and PSD width than the IJ mixer. Differences in IL D[50] and width between mixers are more prominent at lower SI. At higher SI, data variability increases. This is likely a result of incomplete mixing causing uneven nucleation, growth and aggregation. Larger D[50] in the IJ mixer suggests that concentration gradients may promote aggregation. While all available models suggested that mixing time was lower than induction time, it is expected that induction time follows a distribution. Therefore, under continuous operation, lower induction times will eventually be encountered. Greater differences in width between IL and FS techniques at higher SI (for both mixers) show increased formation of weak aggregates. Aggregate bonding is proportional to saturation, but struvite aggregate structure is weaker due to dentritic growth at higher saturation (Ye et al., 2014).

References

Galbraith, S. C., Schneider, P. a, & Flood, a E. (2014). Model-driven experimental evaluation of struvite nucleation, growth and aggregation kinetics. Water Research, 56C, 122–132.

Galbraith, S. C. (2011). A study of struvite nucleation, crystal growth and aggregation. PhD Thesis, James Cook University.

Iqbal, M., Bhuiyan, H., & Mavinic, D. S. (2008). Assessing struvite precipitation in a pilot-scale fluidized bed crystallizer. Environmental Technology, 29(11), 1157–67.

Shimamura, K., Ishikawa, H., Tanaka, T., & Hirasawa, I. (2007). Use of a seeder reactor to manage crystal growth in the fluidized bed reactor for phosphorus recovery. Water Environment Research, 79(4), 406–13.

Ye, Z., Shen, Y., Ye, X., Zhang, Z., Chen, S., & Shi, J. (2014). Phosphorus recovery from wastewater by struvite crystallization: Property of aggregates. *Journal of Environmental Sciences*, *26*(5), 991–1000. doi:10.1016/S1001-0742(13)60536-7

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Is the presenting author an IWA Young Water Professional? (Y)

Bio: Max worked for two years in wastewater operations at Townsville City Council and is currently finishing his PhD on struvite seed production and agglomeration analysis using population balance modelling. Max is interested in system sustainability and resilience and aims to work in the water treatment and resource recovery industry.