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THE EFFECT OF STIRRER ROTATION ONCRYSTALIZATION OF STRUVITE THAT CAN BE USED AS FERTILIZER

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ABSTRACT

Stirring has been used as an efficient method for crystallization so as to maintain the crystal suspension and to achieve uniformity of all suspension properties in the crystalliser. The addition of tartaric acid can significantly affect the crystallization kinetics and crystal morphology even in small amounts. The purpose of this study was to study the effect of stirring and the addition of tartaric acid on the crystallization of struvite which can be used as fertilizer. $MgCl_2$, $C_4H_6O_6$ and $NH_4H_2PO_4$ crystals were dissolved in distilled water to prepare struvite crystallization solution. All solutions were then mixed in a beaker glass with stirring at 200 and 300 rpm at 40ºC with the addition of 0, 10, 20 ppm tartaric acid using the bath process. The results showed that the stirring speed affected the growth of struvite crystals, the higher rotation speed of the stirrer resulted in an increase in struvite crystals so that the constant rate value became greater so that it could be used as a good parameter for the manufacture of struvite fertilizer. Material characterization used Scanning electron microscopy (SEM-EDX) and crystal phase analysis using X-ray powder diffraction (XRPD).

Keywords: Stirrer rotation, ,struvite, SEM-EDX, tartaric acid, XRPD

INTRODUCTION

Stirring has been used as an efficient method for crystallization so as to maintain the crystal suspension and to achieve uniformity of all suspension properties in the crystalliser. Stirring in the solution could helped to thin out the adsorption layer so it could affected the mass transfer between the solution and the crystals. Thus, the crystal growth rate would increased with increasing stirring speed (Sung et al, 2002). However, increasing the stirring speed caused the fluid velocity to increase and collisions occur between the crystals and the stirrer. Stirring speed affected the growth of struvite crystals, the higher the rotation speed of the stirrer results increased in struvite crystals so that the constant rate value became greater, namely 2.6; 3.7 and 5.8 h^{-1} for agitator rotation of 50, 100 and 120 rpm (Ariyanto, 2013). Likewise, according to Rahaman and friends, the value of the cash rate rose by 1.902; 2.034 h^{-1} for agitators 70 and 100 rpm (Rahaman et al, 2008). But at a stirring speed of 500 rpm the value of the constant rate began to decrease, 0.90; 1.38; 0.72; 0.48 h^{-1} for 200, 300, 500 and 700 rpm (El-gawad et al, 2017).

Struvite crystallization has been found to be a promising technique for phosphorus recovery because the precipitate obtained can be used as a slow release fertilizer (Bing Li et al, 2019). Recently, struvite crystals have became attractive because the final product had a potential market for the fertilizer industry. Struvite crystals could impaired equipment performance and lead to increase maintenance costs. However, struvite crystallization is now considered a prospective technology for phosphate recovery from wastewater treatment processes, municipal sewage, industrial effluents and liquid fertilizers (Mohajit et al, 1989; Doyle, J.D., Parsons, S.A, 2002).

Additives could significantly affected crystallization kinetics and crystal morphology even in small amounts (Muryanto and Bayuseno, 2014). Research on the effect of additives on the formation of struvite crystals has been widely carried out, but the effect generally depended specifically on the type and concentration of additives and the characteristics of each type of crystal (Muryanto et al, 2002). Reduced struvite crystals as crystalline crust in general used inorganic additives Cu^{2+}, Pb^{2+} and Zn^{2+}(Perwitasari et al, 2018), or organic additives citric acid (Perwitasari et al, 2017), and maleic acid (Perwitasari et al, 2018),which could inhibited the growth of struvite crystals, but struvite itself contains phosphate which could be used as fertilizer material.

Many organic and inorganic additives could inhibited the crystallization process, but the most effective organic additive was tartaric acid (Prisciandaro et al, 2003). Therefore, it was necessary to do research on the effect of stirring and the addition of tartaric acid on the crystallization of struvite which can be used as fertilizer.

RESEARCH METHODS

The materials and equipment used in the research were distilled water, KOH, $MgCl_2.6H_2O$, $NH_4H_2PO_4$ and additives Tartaric Acid ($C_4H_6O_6$) with analytical grade chemicals (MerckTM), analytical balance, stirrer, measuring cup, magnetic stirrer, filter paper.

Research Stages

$MgCl_2$, $C_4H_6O_6$ and $NH_4H_2PO_4$ crystals were dissolved in distilled water to prepare struvite crystallization solution. The pH of the initial solution was made equal to the addition of 0.5 N KOH. Additive concentrations of tartaric acid (0; 10; and 20 ppm) were dissolved and added to the $MgCl_2$ solution. All solutions were then mixed in a beaker glass with stirring at 200 and 300 rpm at 40ºC using the bath process. The resulting solution is then filtered and dried. After drying, it was weighed to get the mass of struvite crystals.

Material characterization

The dried precipitate was analyzed for morphology and struvite crystal elements used Scanning electron microscopy (SEM-EDX) and crystal phase analysis using X-ray powder diffraction (XRPD).

RESULTS AND DISCUSSION

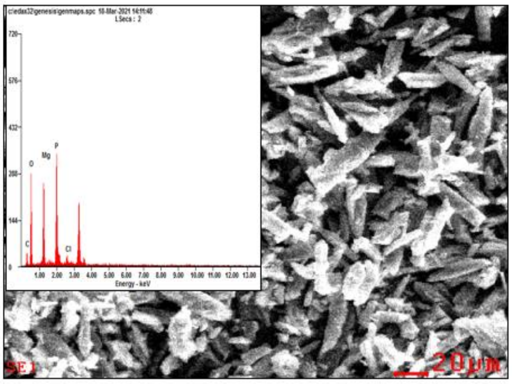
The stirring speed affected the growth of srtuvite crystals, the higher rotational speed of the stirrer results was an increase in the deposition of struvite crystals so that the value of the constant rate became greater, as has been done by previous researchers (Ariyanto Eko, 2013; Rahaman MS et al. 2006), so that the more struvite crystals. which could be used as fertilizer. The highest constant rate value was at 300 rpm with a constant rate of 1,614 h^{-1}. However, with the addition of tartaric acid concentration the value of the constant rate decreased, this indicated that the growth of struvite crystals is inhibited. As shown in table 1 below.

Table 1. First-order rate constant for struvite crystallization

Temperature and stirrer rotation	Tartaric acid concentration (ppm)	Regression equation	Constant rate	R^2
40ºC, 200 rpm	0	y= -0,0254x – 3,4428	1,524	0,9879
	1	y= -0,0238x – 3,5212	1,428	0,9678
	10	y= -0,0195x – 3,3390	1,170	0,9604
	20	y= -0,0163x – 3,4168	0,978	0,9363
40ºC, 300 rpm	0	y= -0,0269x – 3,3253	1,614	0,9611
	1	y= -0,0241x – 3,4890	1,446	0,9608
	10	y= -0,0205x – 3,2913	1,230	0,9679
	20	y= -0,0169x – 3,3904	1,014	0,9430

Mineral characterization

From the results of the SEM-EDX analysis obtained as shown in Figures 1a and 1b for a stirrer rotation of 200 rpm with the addition of 10 and 20 ppm of tartaric acid. The more the concentration of tartaric acid added, the number of struvite crystals decreased. Likewise in Figures 2a and 2b for a stirrer rotation of 300 rpm, the more the concentration of tartaric acid added, the number of struvite crystals decreased. Meanwhile, Figure 1a (200 rpm) and Figure 2a (300 rpm) with different stirrer rotations showed that the higher the stirrer rotation, the greater the number of struvite crystals.



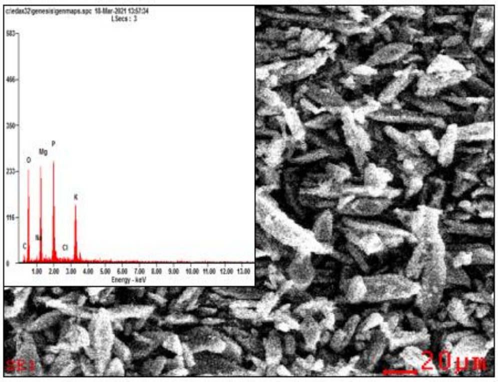
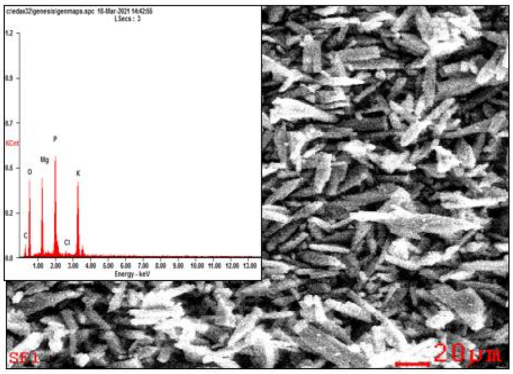


Figure 1a, at 200 rpm, 40ºC, 10 ppm   Figure 1b, at 200 rpm, 40ºC, 20 ppm



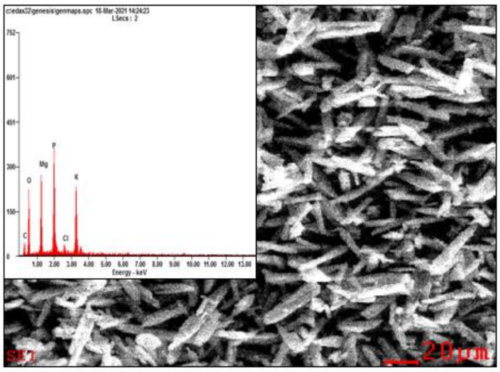


Figure2a, at300 rpm, 40ºC, 10 ppm   Figure2b, at300 rpm, 40ºC, 20 ppm

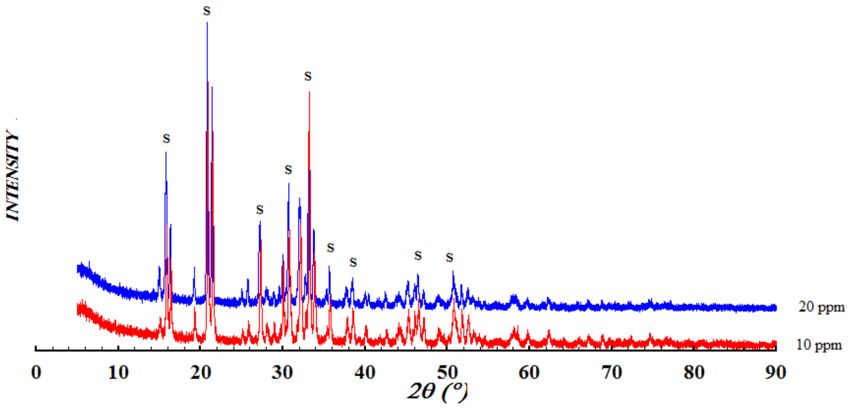


Figure 3, XRPD pattern at 200 rpm, 40ºC (10, 20 ppm)

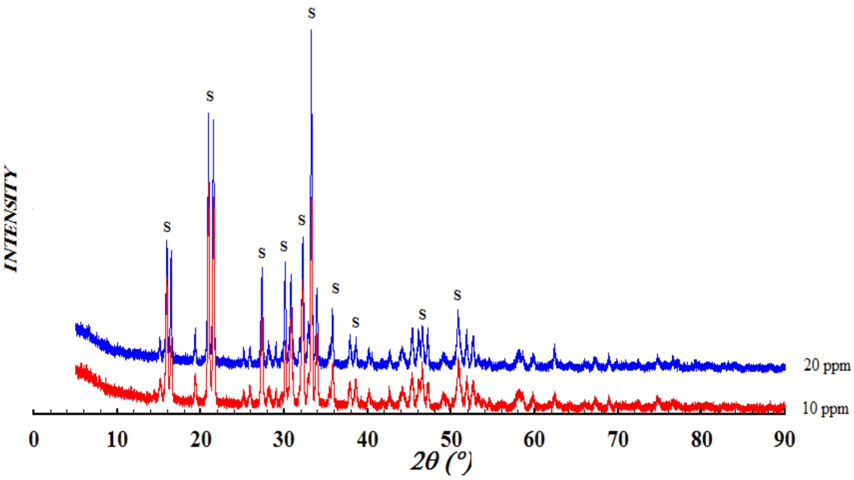


Figure 4, XRPD pattern at 300 rpm, 30ºC (10, 20 ppm)

Figure 3 and Figure 4 are X-ray diffractograms of the crystal samples where struvite crystal peaks were obtained at 200 rpm and 300 rpm at 40ºC with the addition of 10 and 20 ppm tartaric acid concentrations.

CONCLUSION

From the results of the study, it can be concluded that the stirring speed affected the growth of struvite crystals, the higher the rotation speed of the stirrer results was an increase in struvite crystals so that the constant rate value became greater so that it could be used as a good parameter for the manufacture of struvite fertilizer.

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REFERENCES

1. Sung, M.H., Kim, J.S., Kim, W.S.,and Izumi Hirasawa, W.S.K. 2002, Modification of crystal growth mechanism of yttrium oxalate in metastable solution,*Journal of Crystal Growth*, **235**, 529 - 540.
2. Ariyanto, Eko. 2013, '*Crystallisation and dissolution studies of struvite in aqueous solutions*,' Ph.D, Curtin University, Department of Chemical Engineering, Division of Science and Engineering, Australia.
3. Rahaman, M.S., Mavinic, D.S., Ellis, N. 2008, Effects of various process parameters on struvite precipitation kinetics and subsequent determination of rate constants, *Water Science&Technology*,**57**, 647-654.
4. El-Gawad, H.A., Kattab, I.A., Mahdy, A.N., Moselhy, H., Ibrahim, O.A. 2017, Cementation of lead from mono-metallic nitrate solution using a modest agitated reactor, *Research Journal of Pharmaceutical, Biological and Chemical Sciences*, **8**, 1366 - 1380.
5. Bing Li., Irina Boiarkina., Wei Yu., Hai Ming Huang., Tajammal Munir., Guang Qian Wang., Brent R. Young. 2019. Phosphorous recovery through *struvite* crystallization: Challenges for future design. Science of the Total Environment, vol 648, pp. 1244-1256
6. Mohajit, X., Bhattarai, K.K., Taiganides, E.P., Yap, B.C. 1989, Struvite deposits in pipes and aerators, Biological Wastes, **30**, 133 - 147.
7. Doyle, J.D., Parsons, S.A. 2002, *Struvite* formation, control and recovery, Water Research, 36, 3925 - 3940.
8. Muryanto, S., Bayuseno, A.P, (2014), Influence of Cu^{2+} and Zn^{2+} as additives on crystallization kinetics andmorphology of struvite,*Powder Technology, 253*, 602 - 607
9. Muryanto, S. 2002, '*The role of impurities and additives in the crystallisation of gypsum*,' Ph.D, Curtin University, Department of Chemical Engineering, Division of Science and Engineering, Australia.
10. Perwitasari. D.S., Muryanto. S., Jamari. J., Bayuseno. A.P. 2018, Kinetics and morphology analysis of *struvite* precipitated from aqueous solution under the influence of heavy metals: Cu2+, Pb2+, Zn2+, Journal of Environmental Chemical Engineering, 6, 1, 37 – 43
11. Perwitasari, D.S., Edahwati, L., Sutiyono, S., Muryanto, S., Jamari, J., Bayuseno, A.P. 2017, Phosphate recovery through *struvite*-family crystals precipitated in the presence of citric acid : mineralogical phase and morphology evaluation, Environmental Technology, 38, 22, 2844 - 2855.
12. Perwitasari. D.S., Muryanto. S., Jamari. J., Bayuseno. A.P. 2018, Modeling and Optimization of *Struvite* Crystal Scaling Using Experimental Design Methodology For Maleic Acid, Atlantis Highlights in Engineering (AHE), volume 1, 253 – 258
13. Prisciandaro, M., Lancia, A., and Musmarra, D. 2003, The Retarding Effect of Citric Acid on Calcium Sulfate Nucleation Kinetics, *Industrial & Engineering Chemistry Research.*, **42,** 6647 - 6652.