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Optimization Of Struvite Crystallization With Addition Of Tartaric Acid In Liquid Waste As Fertilizer Materials

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Abstract-This paper presents experiments of struvite crystallization ($MgNH_4PO_4.6H_2O$)on a stirred-batch lab. The optimizing parameters, namely solution temperature (30, 35 and 40°C), tartaric acid concentration (1, 10 and 20 ppm),stirring speed (200, 250 and 300 rpm), were using Response Surface Methodology to provide the optimum yield of the mass scales. The tartaric acid concentration was the most significant factor for scale yield, while the temperature and the stirring speed were insignificant factors determining the optimal condition of the mass scale yields in the Response Surface Methodology. The optimum mass scale response of 10.483 mg was obtained at a temperature of 36°C, stirring speed of 260 rpm and tartaric acid concentration of 18 ppm, respectively. This gave the best combination of process parameters for struvite crystallization in liquid waste as fertilizer materials.

Keywords: Mass scale, Response surface methodology, Struvite crystallization,Tartaric acid

Introduction

Struvite crystals (magnesium ammonium phosphate hexahydrate) are hydrated phosphates comprising equimolar amounts of magnesium (Mg), ammonium (NH_4^+) and phosphate (PO_4^{3-}) [1]. Wastewater containing high phosphorus and nitrogen will be a good source of struvite [2]. Likewise, new wastewater among the available technologies, namely urine transfer toilet seems to be the most effective [3] and phosphorus compounds can be extracted from urine as struvite [4]. Rodrigues et al evaluated the interaction of pH and Mg: PO_4 ratio using response surfaces as a major factor for struvite precipitation and are recommended at pH 8.5 with Mg: PO_4 ratio 1.2: 1 and30 minute contact time [5]. From previous studies on the effect of adding organic and inorganic additives to crystallization of struvite it was found that for citric acid [6], metal ions Cu^{2+}, Pb^{2+}, Zn^{2+} [7], and maleic acid [8] the higher the concentration the lower the rate of constant and the less crystals obtained. Furthermore, Perwitasari et al presents the interaction of Temperature and maleic acid concentration using response surfaces for struvite precipitation where an optimum mass scale response of 10.43 mg is obtained [9]. Tartaric acid was an environmentally friendly carboxylic acid and meets the main requirements which are effective as additives other than citric acid and maleic acid which can inhibit the growth of struvite crystals [10]. [6], [8].Zhang et al reported that temperature and pH affect the size of struvite crystals and are not much focused on phosphat recovery [11].In this work, optimization of the process uses the central composite design (CCD) for caculating therespon surface methodology (RSM) of crystalline mass to find the optimal value in liquid waste, involving temperature, stirring speed, tartaric acid concentration and maximizing struvite precipitation. A second order polynomial models was determined for the mass scale results as a function of these variables and provides the best combination of process parameters for crystallization of struvite in liquid waste as fertilizer. Material characterization, including SEM for morphological analysis and XRPD for phase composition was applied in the study.

Materials and methods

2.1 Chemicals

Materials needed in the preparation of the supersaturated solutions were $MgCl_2.6H_2O$ and $NH_4H_2PO_4$ with analytical grade chemicals (Merck^{TM}). The Tartaric Acid ($C_4H_6O_6$) with analityeal grade chemicals (Merck^{TM}).was also used as additives. Here 250 ml 0,10 M ($MgCl_2.6H_2O$ and $NH_4H_2PO_4$) were prepared accordingly. Moreover, the solution with pH 9 was prepared by diluting 1 N KOH solution. In this work, the effect of temperature 30, 35, and 40°C,tartaric acid concentration 1; 10, and 20 ppm, and stirring speed 200, 250 and 300 rpm was evaluated. The obtained slurries were then separated through a paper filter, whereas the obtained precipitaties were dried at room temperature and subsequently were weighed using sartorius weigh-scale.

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2.2SEM-EDXand XRPD method

The particles size and morphology were analysed by scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDX). Measurements on the SEM images were made with J-image software. For EDX analysis, the samples were coated in a thin-film of gold.

Phase identification of scale mineral was conducted by XRPD (X-ray powder diffraction) analysis. In this method, the peak positions and peak heights were verified agains the entries in the ICDD-PDF.The identified mineral phases were subsequently adjusted by Rietveld method using Fullprof-2k, software, program version 3.30 [12].

2,3 Design of Experiment

In this present study, the optimizationvariables of stirring speed, temperature and tartaric acid concentration to yield the optimum mass scale was perrformed by SRM within the CCD (Table 1).SRM calculating was conducted by the statistical v.6 software packages (StatSoft, Tulsa, OK, USA). Using this method, the proper response value and mathematical model fitted to the measured data was acquired from the experiments, and the independent variables of optimal conditions.

Table 1. Independent variables and their level

	Range and Level		
Independent variables	LowLevel	(-1) Center(0)	HighLevel (+1)
Temperature (^{0}C)	30	35	40
Tartaric acid concentration (ppm)	1	10	20
Stirring speed (rpm)	200	250	300

Results and Discussion

Precipitate characteristics

SEM images show that crystals morphology to be irregular prismatic crystal, the EDX spectrum consisted of the highest peaks of K, Mg, P and O corresponding to chemical elements of struvite and struvite (K). Furter, the corresponding solid crystals were subjected to XRPD.Each peak profile hadbeen justifed by the Rietveld revinement and matched by struvite and struvite (K).

3.1 Predicted response model

Variabels for response optimization was modeled using SRM (table 1), where there are 3 factorial design 2 [3] in CCD providing nc = 8; ns = 6; no = 2 and run = 16. Moreover preliminary studies were caried out to determine the required range of temperature (X_1, 30 - 40 °C), tartaric acid concentration (X_2, 1 - 20 ppm) and (X_3 stirring speed 200 - 300 rpm). The Level for SRM consisted of low level (-1) (30, 1, 200), high level (+1)(40, 20, 300) and center point (0) (35, 10, 250). The mass of struvite obtained from all the experiments is presented in table 2. The yield response of mass precipitate (mg) is presented in table 2.The second-order polynomial equation with the mathematical model for the regression analysis of the experimental data, the optimum resulted follows the equation:

$$(y) = 23.07008-0.42634X_1+0.00509X_1^2+0.05543X_2+0.00339X_2^2-0.04895X_3+0.00007X_3^2-0.0021X_1X_2+0.00054X_1X_3-0.00035X_2X_3$$

Where X_1, X_2, X_3 are the coded variablesfor temperature, tartaric acid concentration and stirring speed and Y is the yield of mass scale.

Table 2. Experimental design with Independent variables

Run	Temperature (°C)	Tartaric acid (ppm)	Stirring speed (rpm)	Responses mass scale (mg)
	Independent variables			
1	30.000	1.000	200.000	10.851
2	30.000	1.000	300.000	11.427
3	30.000	20.000	200.000	10.584
4	30.000	20.000	300.000	10.507
5	40.0001.000		200.000	11.240
6	40.0001.000		300.000	12.384
7	40.00020.000		200.000	10.595
8	40.00020.000		300.000	11.035
9	26.591	10.000	250.000	10.873
10	43.408	10.000	250.000	11.548
11	35.0001.977		250.000	12.956
12	35.00025.977		250.000	10.437
13	35.00010.000		165.910	11.218
14	35.00010.000		334.089	11.541
15	35.00010.000		250.000	10.620
16	35.00010.000		250.000	10.620

Furthermore, the analysis of varian for the statistical testing of the model is shown in table 3. The effect of the significance of a factor can be seen from F-value and p-value.The fitting by quadratic regression model shows the value of the coefficient (R^2) of 0,974, there is no significant lack of fit ($p > 0.05$). The result of the calculated model 95% and only 2.6 % of the total variation did not fit the model.F-value was calculated from the ratio between MSF (mean squares of the factor) of the MSE (mean squares of error). A factor has a significant effect when theF-value is greater than F-table. It is shown in table3 that the value of the F-table is less than F-value.
The experimental results of all parameters were also presented in chart Pareto (figure 3). The chart Pareto show that of tartaric acid concentration most significant, which is then followed by temperature and stirring speed in controlling struvite production.

Table 3. Analysis of variance yield response

Source	Sum of square (SS)	Degree of freedom (DF)	Mean square (MS)	F-value	F-table	R^2
SS regression	6.830530	9	6.830530	46.4715	4.1	0.974
SS error	0.881895	6	0.146982			
SS total	7.712425	15				

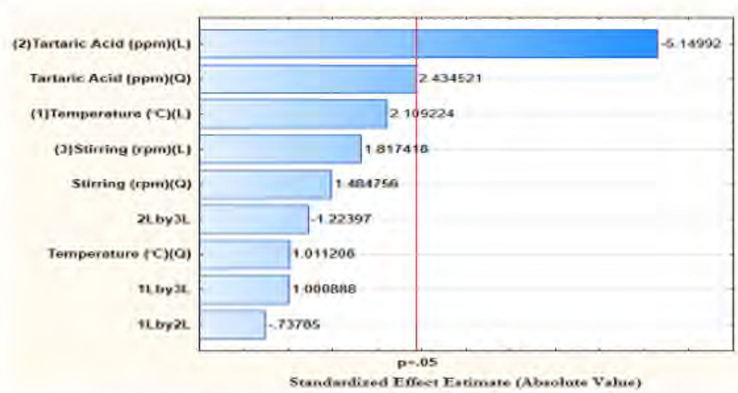

Figure 1. The Yieldresponse of struvite production by Pareto chart

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3.3 The optimum mass scale yield use response surfaces

The interaction of independent and dependent variables in response of mass scale yield was presented 3D and 2Dcontour plots (Fig 2 to 4), determine of struvite production (mass in mg). The significant interactions between thevariables are shown in an elliptical contour plot. Fig 2 illustrates the interaction between tartaric acid concentration and temperature for the yield of struvite. The increase oftemperature in the high amount of mass scale, and otherwise the increasing tartaric acid concentration to reduce mass scale yield. While Figure 3 shows the relationship between stirring speed and temperature where the higher the stirring speed and temperature, the mass scale will raise between 10-11 mg.For the interaction between stirring speed and tartaric acid concentration is shown in Figure 4 where with increasing stirring speed the mass scale will increase otherwisewith increasing tartaric acid concentration the mass scale will decrease.

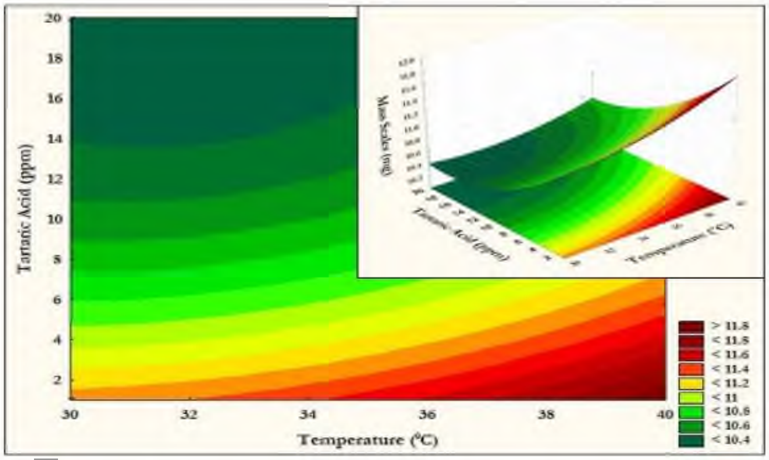

Figure 2. Contour plots of Temperature and Tartaric Acid Concentration to mass scale yield

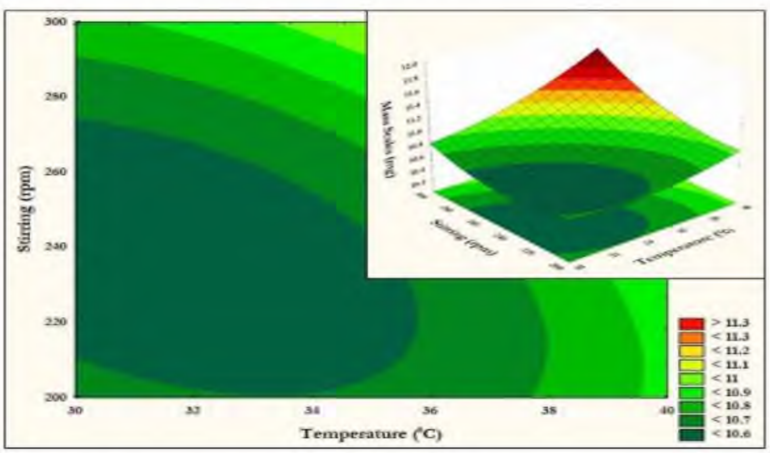

Figure 3. Contour plots of Temperature and Stirring Speed to mass scale yield

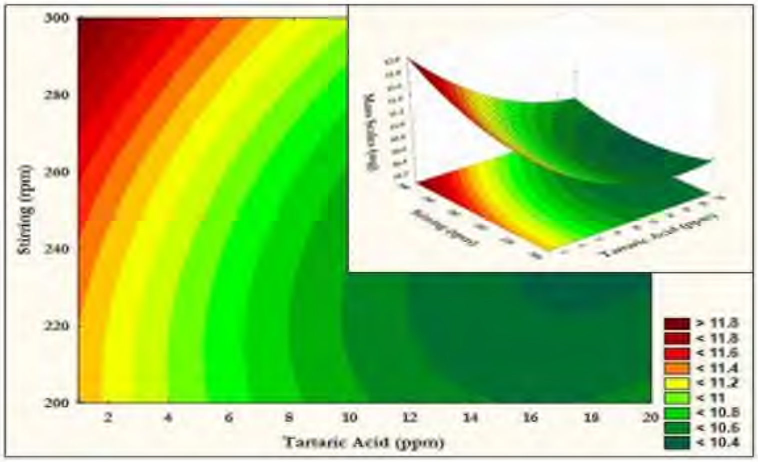

Figure 4. Contour plots of Tartaric Acid Concentration and Stirring Speed to mass scale yield

Figure 2-4 shows that an increase in stirringspeed and temperatureresults an increase in mass scale (mg). Increasing of the concentration tartaric acid significantly decreases mass scale.By entering the optimum variablevalue into the polinomial equation, the optimum mass scales can be seen in table 4.

Table 4. Optimum mass scale Analysis

Factor	Optimum values	Optimum precipitate mass (mg)
Temperature (^{0}C)	36	
Tartaric acid concentration (ppm)	18	10.483
Stirring speed (rpm)	262	

Verification is done by comparing the results of Response Surfase Metodhology optimization with the results of laboratory experiments.The comparison was to get the percentage of diversity for mass scaleyeald is 3.506% so the accuracy value is 96.494%. The percentage of diversity value resultsof the optimum variable as presented in table 5.

Table 5. Optimum variables and the response and the experiment result

Optimum Variables	Result of response	Result of lab experiments	% Diversity
Temperature (^{0}C) Tartaric acid concentration (ppm) Stirring speed (rpm)	10.483	10.864	3.506 %

Diversity (%) = [(Result of lab experiments - result of response)/result of lab experiments]×100%

Conclusions

The method uses response surface methodology (RSM) for optimization of struvite products shows thatof temperature of 36^{o}C, stirring speed of 260 rpm and tartaric acid concentration of 18 ppm, the struvite crystal mass was 10.483 mg. While the one that significantly influences the production of struvite is the Tartaric acid concentration.This gave the best combination of process parametersfor struvite crystallization in liquid waste as fertilizer materials.

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