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Understanding the production and reduction of barium sulfate crystals through the use of additives and a controlled stirring rate

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ABSTRACT

Keywords: fecl2; srcl2, barium sulfate, response surface methodology.

Barium sulfate crystals are minerals responsible for scaling in piping systems. Controlling the growth of these crystals can use additives (FeCl2 and SrCl2) and varying agitation speeds. The research was to optimize the impact of additives and agitation speed on the results of crystal form using RSM through Minitab 19 with Box-Behnken Design. The optimum conditions using additive FeCl2 at a concentration of 25 ppm, a stirring speed of 120 rpm for 30 minutes, and a coefficient of determination (R2) of 90.995 provided 0.4485 gr of barium sulfate crystals. The optimum conditions, however, used SrCl2 additives at a concentration of 20.2049 ppm and a stirring speed of 459.394 rpm for 30 minutes, yielding 0.4345 g of barium sulfates with a coefficient of determination (R2) of 91.41%. The results of crystallizing barium sulfate without additives appear to be superior to those obtained with additives in terms of production. In contrast, additives of FeCl2 and SrCl2 can inhibit barium sulfate formation, resulting in a reduction in crystal mass.

Introduction

Crystal formation is a mineral scaling product crystallized in the industrial world, particularly in piping systems. Water used in underwater piping systems contains various types of content, including barium ions and sulfate ions. When the two ions combine, they form a barium sulfate compound. (Fatra & Suwignyo, 2020). The influence of the forming agent concentration in the flow system determines crystal formation. The higher ion concentrations lead to the faster the crystals grow. (Sodikin, 2016). The presence of these crystals can cause the scale to reduce the inner diameter of the pipe, resulting in fluid flow obstruction. (Fatra & Suwignyo, 2020).

Additionally, controlling crystal growth can be accomplished by adjusting additives and stirring speed. This additive can inhibit crystal growth. According to (Karaman, Mangestiyono, Muryanto, Jamari, & Bayuseno, 2019), chemical additives can help to prevent crystal growth. On the other hand, increasing the speed of stirring, according to Anggrainy (Anggrainy, Bagastyo, & Hermana, 2014), affects the rate of crystal formation reactions occurring in the system, increasing the size and mass of the particles.

Understanding crystal growth and subsequent controlling crystal product may use method response surface methodology to determine the optimum response with the additive concentration and the stirring speed factors. Response Surface Methodology (RSM) is a mathematical and statistical technique for modeling and analyzing problems with many variables to optimize responses. (Sartini, Fitriani, & Lubis, 2018). Central Composite Design (CCD) and Box-Behnken Design are experimental designs frequently used in research. These designing experiments are more efficient with fewer trials. (Nurmaya, Sunaryo, Algorithm, & Programming, 2013). Thus, this study investigated strontium chloride and ferric chloride additives to inhibit crystal growth and controlled stirring rates affecting the formation of barium sulfate crystals using RSM to determine the optimal crystal growth conditions.

Furthermore, barium sulfate is an inorganic compound with the chemical formula $BaSO_4$. An insoluble crystallized substance, barium sulfate is odorless and white. Barium sulfate is non-toxic and non-explosive (Subyakto, 2011). The barium sulfate compound can precipitate from solutions containing barium chloride and sodium sulfate. The reaction occurs when barium chloride is mixed with sodium sulfate, resulting in barium sulfate. (Dera, 2018), as follows:

$$Na_2SO_4 + BaCl_2{\cdot}2H_2O \rightarrow NaCl + BaSO_4 + H_2O \quad (1)$$

Naturally, barium sulfate (barite) can crystallize when sulfate ions in seawater interact with barium ions in the water. (Karaman, Jamari, Bayuseno, & Muryanto, 2017). The diffusion process of barite crystal growth occurs on the solid surface. Solute molecules or ions diffuse through the liquid phase to reach the growing crystal surface. (Pinalia, 2011). Supersaturation is one of the crystallization conditions. In particular, the use of additives affects the crystallization process. Additives inhibit the barite crystal growth by combining the structure on the crystal surface and disrupting the addition of growth units. (J. W. Mullin, 2001). Increasing the concentration of additives reduces the rate of settling. (Karaman et al., 2019). According to (Dera, 2018), the effect of acid additives lauric from 10 ppm to 20 ppm on the growth of barium sulfate crystals with a concentration of 3500 ppm was a decrease in the rate of crystal growth. Here additives of lauric acid can inhibit crystal growth resulting in a reduced crystal mass. Further factors that affect the process of crystal formation may include the stirring rate shortens the distance between particles, which results in more frequent contact and collisions. A higher stirring speed will also increase the amount of contact between reactants.

Further, (Karaman et al., 2019), investigated the effects of five green inhibitors on barite crystal growth in flow-induced vibration in a pipe under the influence of varying vibration frequencies, namely 0, 4, 8, and 10 Hz. After converting these frequencies to 600 rpm, barium crystal growth was significantly reduced, which can serve as a reference in the present study. The formation of barite crystals was studied using the batch crystallizer method at 600 rpm for a stirring time of 120 minutes with sampling every 15 minutes (Prayuga, Aruba, & Karaman, 2022). As a result, in this study, the time variable was used in the design box- Behnken design with an upper limit of 30 minutes and a lower limit of 120 minutes. On the other hand, excellent barite crystal formation results were from a crystallization process with citric acid additive operating at 50°C (Surya & Intifada, 2011). The higher the water temperature, the more likely crystals form (Samsudi Raharjo, 2020). This temperature effect is significant in water evaporation, causing the amount of water in the water to decrease. This mechanism reduces the formation of crystals. Correspondingly the Box-Behnken Design is an experimental design with the response surface method selected for this study. Design box-Behnken has advantages over CCD. The advantage is that this design is more efficient with fewer trial runs, especially for experiments with 3 or 4 factors (Nurmaya et al., 2013). This design function built a full quadratic model with only three levels for each predictor variable: the lower level (-1), the middle level (0), and the upper level (1) (Khamid, Herdiani, & Sirajang, 2017). Accordingly, use the box-Behnken design to determine variable runs used in research (Figure 1).

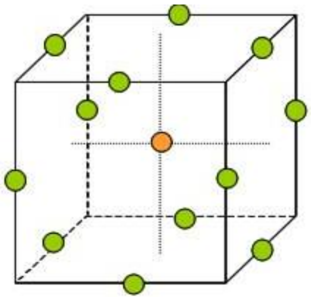

Figure 1
Box-Behnken Design

In addition to improving our understanding of how to control barite crystal growth, these findings will also be useful in the development of chemical additives for controlling crystal growth.

Method

Materials

Analytical grade powders $BaCl_2$, Na_2SO_4, $SrCl_2$, and $FeCl_2$ (Merck Germany) were selected and dissolved in the distilled water used throughout the study. Barium sulfate crystallization followed according to the reaction described below (Eq. 1). The concentration of barium sulfate (3500 ppm) was constant in the study with increasing temperature of the magnetic stirrer by 50 °C. The oven's drying temperature was 100 °C for 60 minutes. Without the addition of additives for independent variables (0 ppm). Each additive concentrations ($FeCl_2$ and $SrCl_2$) are 5, 15, and 25 ppm, respectively. Stirring speeds of 120, 360, and 600 rpm are possible for 30, 75, and 120 minutes. In each experiment, 31.165 gr $BaCl_2$ solid and 18.1 gr Na_2SO_4 were dissolved in 5 L water to achieve brine concentrations of 3500 ppm barium and sulfate. A crystal-forming solution was made by adding $SrCl_2$ and $FeCl_2$ additives.

Table 1
Determination of variables in research with Box-Behnken Design method (BBD)

Run	Code			Test		
	X_1	X_2	X_3	Additive Concentration (ppm)	Mixing Speed (rpm)	Time (minutes)
1	-1	1	0	5	600	75
2	-1	0	1	5	360	120
3	-1	0	-1	5	360	30
4	1	-1	0	25	120	75
5	0	-1	-1	15	120	30
6	0	0	0	15	360	75
7	-1	-1	0	5	120	75
8	0	0	0	15	360	75
9	0	-1	1	15	120	120
10	1	1	0	25	600	75
11	0	0	0	15	360	75
12	1	0	-1	25	360	30
13	0	1	1	15	600	120
14	0	1	-1	15	600	30
15	1	0	1	25	360	120

Experiments

In 100 ml burettes, place two different materials between barium chloride and sodium sulfate. Stirring temperature and speed were controlled by predefined variables. The filtrate was then screened and the precipitate was filtered before being dried in an oven for 60 minutes and weighed. Furthermore, when it reaches the executed time variable, the process is repeated with other additives with predetermined variables. The research data were collected and analyzed using an RSM approach.

Method

The mass of barium sulfate crystals was as experimental data. The barium sulfate mass results will then be analyzed with the Response Surface Methodology (RSM) in Minitab 19 software to determine optimal results with the Box Behnken Design (BBD). Table 1 shows the design results for the box-Behnken design.

Results and Discussion

Barium sulfate crystals without adding additives

The experimental data obtained in the experiments is the mass of barium sulfate crystals. The results of the barium sulfate mass will then be analyzed using the Response Surface Methodology (RSM) in Minitab 19 software with the Box-Behnken Design (BBD). The analysis was to determine the best results for the effect of additive concentration, stirring speed, and stirring time on the recovery of barium sulfate crystals to obtain the lowest crystal deposits.

Despite varying stirring rates and times, mass crystals weighing up to 0.54 gr were in the yield of barite crystal without chemical additives in Table 2.

Table 2
Crystal recovery without additives

Concentration (ppm)	Time (minutes)	Mixing Speed (Rpm)	Mass Crystal (gr)
0	30	120	0,5247
		360	0,5420
		600	0,5334
	75	120	0,5287
		360	0,5403
		600	0,5421
	120	120	0,5392
		360	0,5491
		600	0,5545

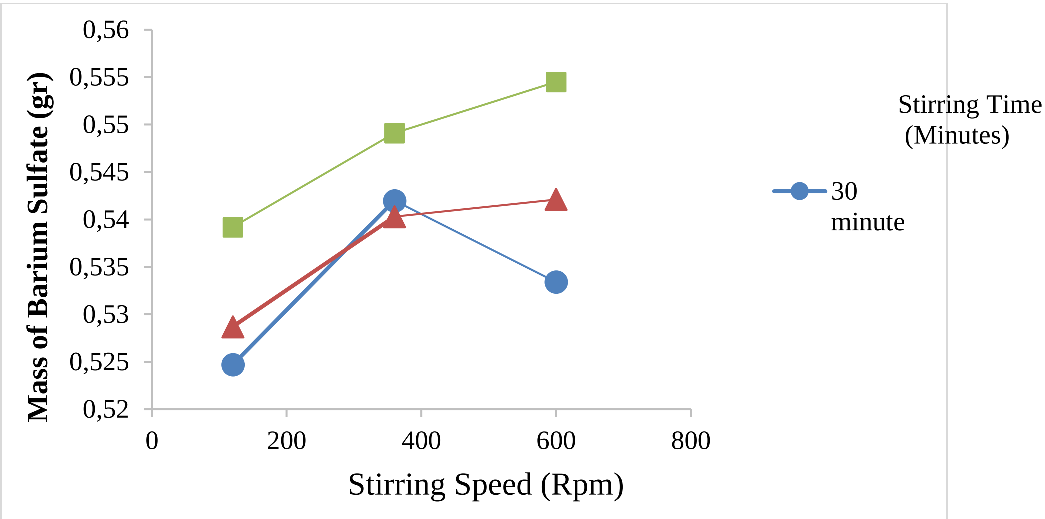

Figure 2
Relationship Between Stirring Time (Minutes) and Stirring Speed (Rpm) on the Mass of Barium Sulfate Crystals (gr)

According to Figure 2, the mass of barite crystals ($BaSO_4$) obtained increases as the stirring time and speed are increased. This research aligns with the study by (Anggrainy et al., 2014) The longer the stirring, the more nuclei form, and the sample solution becomes increasingly turbid. Increasing the stirring speed affects the speed of crystal formation reactions in the system, causing the particle size and mass to increase.

Effect of $FeCl_2$ additive concentration$_2$, stirring speed , and stirring time on barium sulfate crystal formation

Table 3
Crystal recovery at the concentration of the $FeCl_2$ additives, mixing speed, and mixing time

No	Factor			Response
	Additive Concentration (ppm)	Mixing Speed (rpm)	Time (minutes)	Mass Crystal (gr)
1	5	600	75	0.4692
2	5	360	120	0.4916
3	5	360	30	0.4786
4	25	120	75	0.4540
5	15	120	30	0.4618
6	15	360	75	0.4687
7	5	120	75	0.4893
8	15	360	75	0.4696
9	15	120	120	0.4742
10	25	600	75	0.4678
11	15	360	75	0.4596
12	25	360	30	0.4510
13	15	600	120	0.4689
14	15	600	30	0.4451
15	25	360	120	0.4563

The effect of $FeCl_2$ additive concentration, stirring speed, and time on the formation of barium sulfate crystals can use the ANOVA test, which can generate a mathematical model that relates the independent variables to the response variables, as shown in Table 4.

Table 4
Results of the analysis of variance (ANOVA) for the response of obtaining barium sulfate crystals with $FeCl_2$ additives

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	9	0.002191	0.000243	5.59	0.036
Linear	3	0.001711	0.000570	13.09	0.008
Ppm	1	0.001240	0.001240	28.46	0.003
Time	1	0.000371	0.000371	8.52	0.033
Ppm*Rpm	1	0.000287	0.000287	6.59	0.050
Lack-of-Fit	3	0.000157	0.000052	1.71	0.390

Minitab19 with Box Behnken Design is used to process the data in Table 5. (BBD). Where the choice of analysis is known, the interaction between factors with the resulting response. The probability value (p-value) of the degree of significance (0.05) and the lack of fit value when the p-value is greater than 0.05 indicate the suitability of the appropriate treatment model (Sari, Triastinurmiatiningsih, & Haryana, 2020). Based on the findings of the research, the P-value obtained is 0.05, and the P-value for lack of fit is 0.390. As a result, the obtained model is suitable for predicting the optimum conditions of the response to the acquisition of barium sulfate crystals.

The equation obtained from the selected model for the response of barium sulfate crystal acquisition is as follows:

$$y = 0{,}5034 - 0{,}00384x_1 + 0{,}000070x_2 + 0{,}000275x_3 + 0{,}000055x_1^2 - 0{,}000001x_3^2 + 0{,}000004x_1x_2 + 0{,}000004x_1x_3 \quad (2)$$

Where :

x_1x_1 = $FeCl_2$ additive $concentration_2$ (ppm)

x_2x_2 = stirring speed (rpm)

x_3x_3 = mixing time (minutes)

The coefficient of determination (R^2) denotes the variable magnitude of the response that can be explained by a model. (Aryantini, 2017). The regression model demonstrates that variables with positive constants in the model influence the obtained responses. (Ratnawati, Ekantari, Pradipta, & Paramita, 2013). The mathematical model in equation (2) yielded a coefficient value of 0.5017 for the $FeCl_2$ concentration. As a result, the concentration of the additive $FeCl_2$ has a significant influence on the test results. The mathematical model determined that the best value is 25 ppm; 120 rpm; and 30 minutes. According to (Sari et al., 2020), R_2 values greater than 70 % indicate that the observed and predictive values are quite precise in providing closeness to the results. The closer the R-value is to one (100 %), the better the model. (Rosalinda, Nurjanah, Saputra, & Bafdal, 2019). The coefficient of determination (R^2) calculated using minitab19 software is 90.95 %, with the remaining 9.05 % influenced by other factors that cannot be explained by the response. The following results were obtained by optimizing the area using a graphical contour plot :

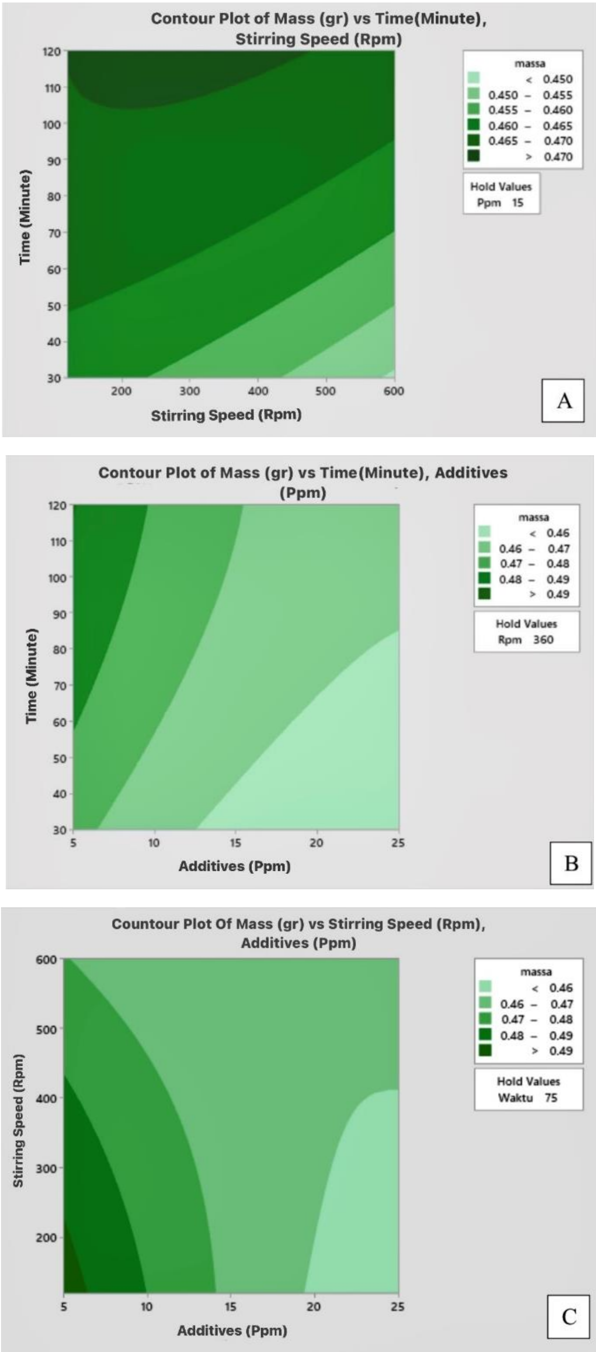

Figure 3
Contour optimization area of the recovery of barium sulfate crystals (a) variable stirring speed and stirring time (b) variable concentration of $FeCl_2$ additive and stirring time (c) the variable concentration of the $FeCl_2$ additive and stirring speed

The contour plot of barium sulfate crystal recovery with variable stirring speed and stirring time is shown in Figure 3 (a). The acquisition of barium sulfate crystals has decreased, as indicated by the bright green line with a stationary point of 0.450. According to (Fachry, Tumanggor, & Yuni, 2008), states that impurities can inhibit crystal growth because they are adsorbed on the crystal surface. As a result, Fe ions^{2+} adsorbed on the crystal surface can inhibit crystal growth and decrease crystal mass.

Figure 3 (b) depicts the contour plot of barium sulfate crystal recovery as a function of additive concentration and mixing time. As can be seen, the best results are described as the brightest color with a stationary point of 0.46. The longer the stirring time, the more crystals formed, according to (Anggrainy et al., 2014).

Figure 3 (c) depicts a contour plot graph of barium sulfate crystal recovery as a function of additive concentration and stirring speed. The optimal result of obtaining barium crystals is shown in a bright green color with a stationary point of 0.46. According to (Prayuga et al., 2022), the addition of additives can suppress or reduce the reaction rate, resulting in a decrease in the mass of crystals formed. As a result, the acquisition of barium sulfate crystals decreased as the concentration of $FeCl_2$ additives increased. The following results were obtained from the optimization area by surface plot graphs :

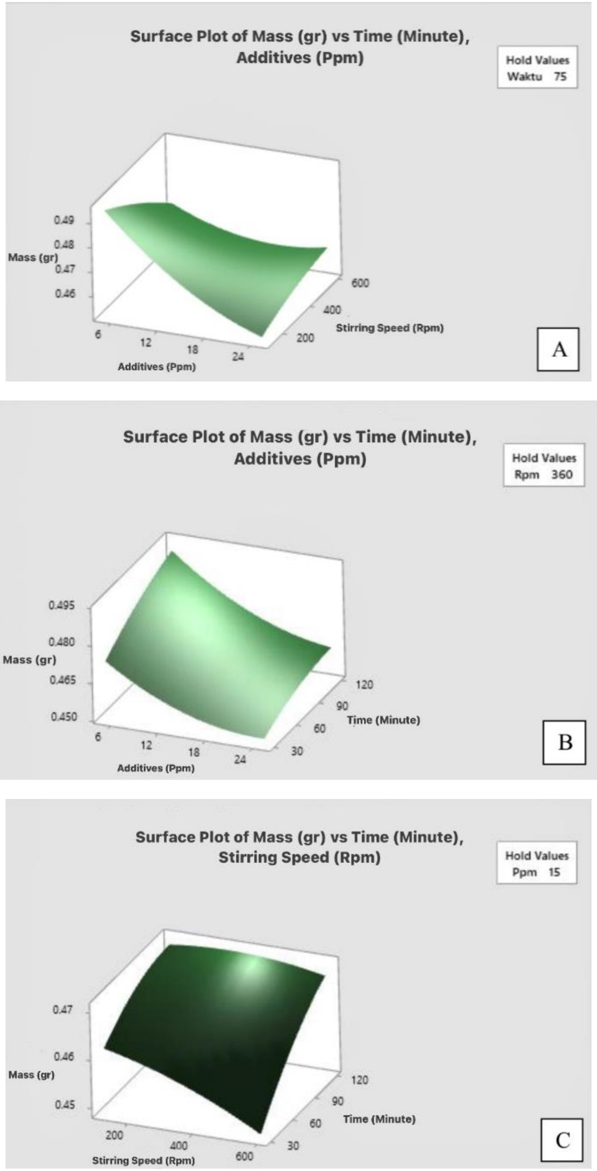

Figure 4

Surface plot of the optimization area of the recovery of barium sulfate crystals (a) variable concentration of $FeCl_2$ additives and stirring speed (b) variable concentration of $FeCl_2$ additives and stirring time (c) variables of stirring speed and stirring time

The determination of the optimization area can be depicted in 3D form in Figure 4. (a,b,c). Figures 4 (a), 4 (b), and 4 (c) show a curve with the smallest response. The color change on the curve represents the effect of each variable on the acquisition of barium sulfate crystals.

Effect of $SrCl_2$ Additive Concentration$_2$, Stirring Speed , and Stirring Time on Obtaining Barium Sulfate Crystals

Table 5
Crystal formation, $SrCl_2$ additive concentration, mixing speed, and mixing time

No	Factor			Response
	Additive Concentration (ppm)	**Mixing Speed (rpm)**	**Time (minutes)**	**Mass Crystal (gr)**
1	5	600	75	0.4653
2	5	360	120	0.4775
3	5	360	30	0.4632
4	25	120	75	0.4557
5	15	120	30	0.4389
6	15	360	75	0.4453
7	5	120	75	0.4728
8	15	360	75	0.4439
9	15	120	120	0.4501
10	25	600	75	0.4482
11	15	360	75	0.4524
12	25	360	30	0.4343
13	15	600	120	0.4573
14	15	600	30	0.4419
15	25	360	120	0.4503

The effect of $SrCl_2$ additive concentrations, stirring speed, and time on the formation of barium sulfate crystals can be evaluated using the ANOVA test, which can be used to generate a mathematical model that relates the independent variables to the response variables, as shown in Table 6.

Table 6
Results of the analysis of variance (ANOVA) for the response of obtaining barium sulfate crystals with $SrCl_2$ additives

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	9	0.001943	0.000216	5.91	0.032
Linear	3	0.001427	0.000476	13.02	0.008
Ppm	1	0.001019	0.001019	27.91	0.003

Time	1	0.000405	0.000405	11.08	0.021
Ppm*Ppm	1	0.000470	0.000470	12.88	0.016
Lack-of-Fit	3	0.000141	0.000047	2.26	0.321

Minitab19 with Box Behnken Design is used to process the data in Table 6. (BBD). Where the choice of analysis is known, the interaction between factors with the resulting response. The probability value (p-value) of the degree of significance (0.05) and the lack of fit value when the p-value is greater than 0.05 indicate the suitability of the appropriate treatment model. (Sari et al., 2020). Based on the findings of the research, the P-value obtained is 0.05, and the P-value for lack of fit is 0.321. As a result, the obtained model is suitable for predicting the optimum conditions of the response to the acquisition of barium sulfate crystals.

The equation obtained from the selected model for the response of barium sulfate crystal acquisition is as follows:

$$y = 0{,}5034 - 0{,}00384x_1 - 0{,}000070x_2 + 0{,}000275x_3 + 0{,}000055x_1^2 - 0{,}000001x_3^2 + 0{,}000004x_1x_2 + 0{,}000004x_1x_3 \quad (3)$$

Where :

x_1x_1 = $SrCl_2$ additive concentration$_2$ (ppm)

x_2x_2 = stirring speed (rpm)

x_3x_3 = mixing time (minutes)

The coefficient of determination (R_2) indicates the variable magnitude of the response that can be explained in a model. (Aryantini, 2017). The regression model shows that the responses obtained are influenced by variables that show positive constants in the model. (Ratnawati et al., 2013). The results of the mathematical model in equation (3) obtained the value of the $SrCl_2$ concentration coefficient of 0.4808. So it shows that the concentration of the additive $SrCl_2$ has a major influence on the test results. The mathematical model obtained the optimum value for x_1x_1 20.2049 ppm; x_2x_2 459.394 rpm and 30 minutes.

According to (Sari et al., 2020), states that the value of R_2 > 70 % indicates that the observed and predictive values are quite precise in providing closeness to the results. If the R_2 value the closer to number one (100 %), the better the model (Rosalinda et al., 2019). The results of calculations using Minitab 19 software that has been carried out show that the coefficient of determination (R^2) = 91.41 %, while the remaining 8.59 % is influenced by other factors that cannot be explained by the response.

Optimization area by graphical contour plot obtained the following results :

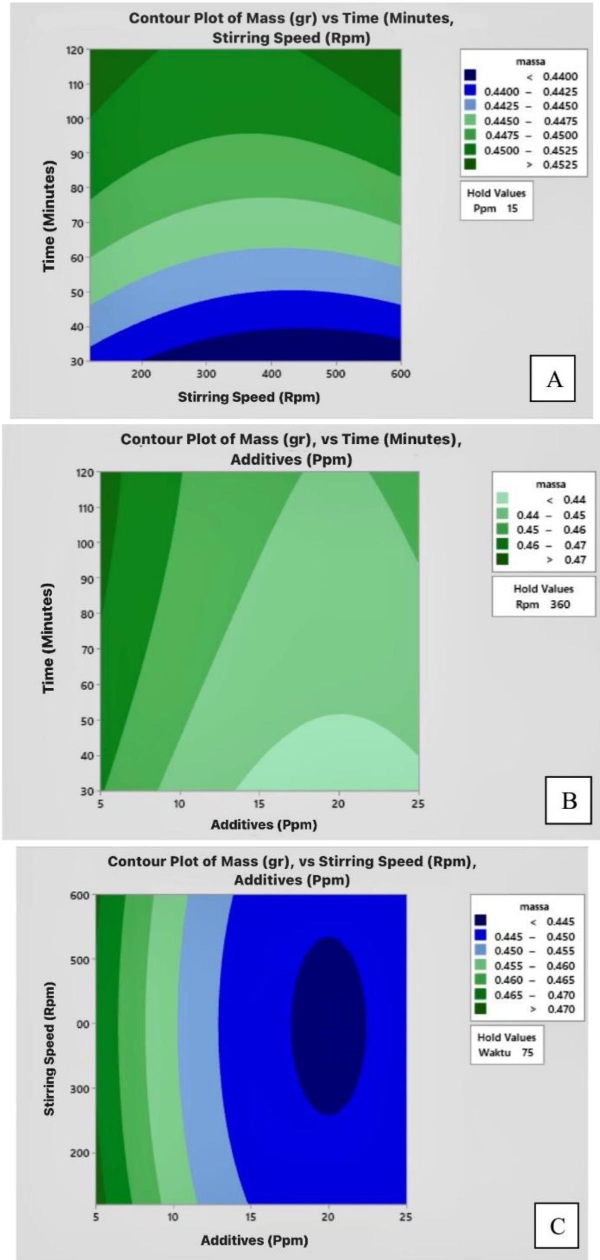

Figure 5
Contour optimization area of the recovery of barium sulfate crystals (a) variable mixing speed and stirring time (b) variable $SrCl_2$ additive concentration and stirring time (c) variable concentration of $SrCl_2$ additives and stirring speed

Figure 5 (a) shows the contour plot of the recovery of barium sulfate crystals with variable stirring speed and stirring time. It can be seen that the acquisition of barium sulfate crystals has decreased which is shown in dark blue with a stationary point of <0.44. According to (Fachry et al., 2008), states that impurities can inhibit crystal growth because impurities are adsorbed on the crystal surface. So Sr^{2+} adsorbed onto the crystal surface can inhibit crystal growth and the crystal mass tends to decrease.

Figure 5 (b) shows the contour plot of barium sulfate crystal recovery between the variable concentrations of the substance additive and mixing time. It can be seen that the optimal results obtained are described as the brightest color with a stationary point of <0.44. According to (Anggrainy et al., 2014), the longer the stirring time, the more crystals formed.

Figure 5 (c) shows a contour plot graph of the recovery of barium sulfate crystals between the variable concentrations of substances additive and stirring speed. It can be seen that the optimal result of obtaining barium crystals is depicted in dark blue with a stationary point of <0.445. According to (Prayuga et al., 2022), the addition of additives can suppress or reduce the reaction rate so that the mass of crystals formed decreases. So the recovery of barium sulfate crystals decreased with increasing concentration of $SrCl_2$ additives. The optimization area by graphical surface plot obtained the following results :

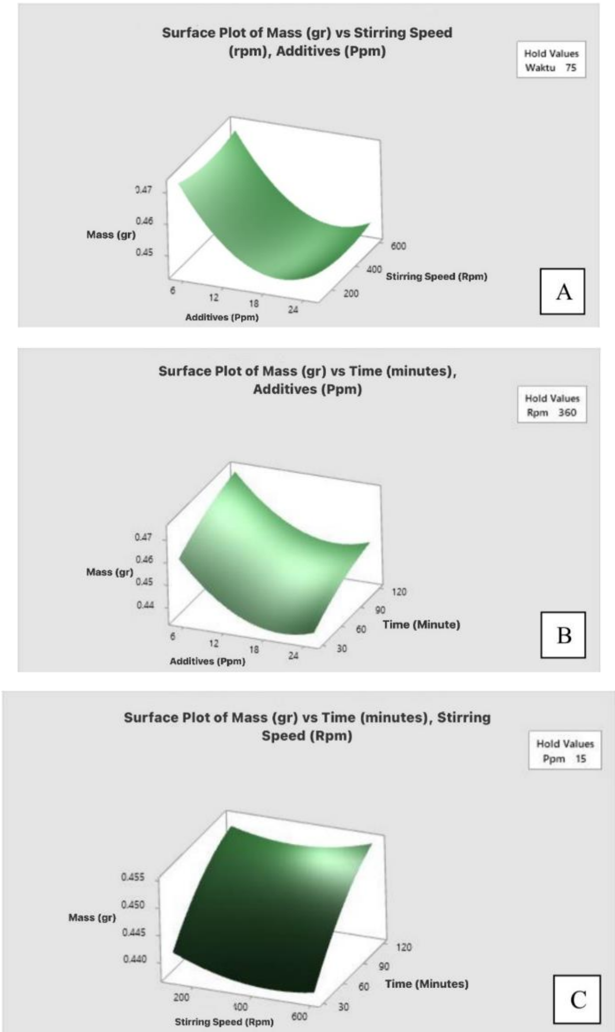

Figure 6

Surface plot of the optimization area of the recovery of barium sulfate crystals (a) variable concentration of $SrCl_2$ additives and stirring speed (b) variable concentration of $SrCl_2$ additives and stirring time (c) variables of stirring speed and stirring time

The determination of the optimization area can be described in 3D form as can be seen in Figure 6 (a,b,c). Figure 6 (a), 6 (b) and Figure 6 (c) produce a curve that forms the minimum response. The color change on the curve indicates the effect of adding each variable to the acquisition of barium sulfate crystals.

Use of $FeCl_2$ Additives and $SrCl_2$

According to the research, the best results were acquiring barium sulfate crystals with $FeCl_2$ additives on the variable 25 ppm and a crystal mass yield of 0.4485 gr. The acquisition of barium sulfate crystals with $SrCl_2$ additives yielded optimal results on the variable 20.2049 ppm with a crystal mass yield of 0.4345 gr. The density of the Fe compound is 7.9 gr/cm3, while the density Sr compound is 3.65 gr/cm3 (Farida, 2018). This finding demonstrates that the Fe compound has a higher molecular weight than the Sr compound. According to (Karaman et al., 2019), higher molecular weight inhibitors are difficult to incorporate into the lattice. As a result, additive compounds with lower densities can more easily enter the crystal lattice and inhibit crystal growth. The optimal difference in yield between the two additives is 0.014 gr. This result demonstrates that the additive $SrCl_2$ can reduce the recovery of barium sulfate crystals more than the $FeCl_2$ additives.

Conclusion

The optimum conditions for obtaining barium sulfate crystals using FeCl2 additives were a concentration of 25 ppm, a stirring speed of 120 rpm, a time of 30 minutes, namely 0.4485 gr, and a coefficient of determination (R2) of 90.95 %. Meanwhile, the optimum conditions were using the additive SrCl2 at a concentration of 20.2049 ppm, a stirring speed of 459.394 rpm, and a time of 30 minutes, which was 0.4345 gr and with a coefficient of determination (R2) of 91.41 %.

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