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Determination of Optimum Conditions with Regression Analysis within the Scope of 6 Sigma for Eliminating Caking Problem in Nitrogen Fertilizers

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Abstract

In the nitrogen fertilizer industry, the nitrogen content of chemical fertilizers is an important parameter that determines its physical strength, storage capacity and storage life. The production variables that determine this physical parameter, which is characterized as degradation and caking, can be expressed as the characteristics that determine the course of the chemical process. Although there are improvements in reaction conditions to solve the caking problem, the cost problem of the manufacturer limits the processing conditions. For this reason, in order to minimize the cost problem in any nitrogen fertilizer production process, the analysis of working conditions and the development of quality management systems accordingly constitute the focus of the manufacturer. In this study, within the scope of lean production, regression analysis of the 6-sigma quality method system was performed, the process parameters during the production were analyzed, the optimum conditions were determined and the effect on the production cost was investigated by comparing with the current production conditions.

Keywords: 6 sigma, regression analysis, caking, degradation, bulk height, nitrogen concentration, crushing strength, coating amount

1. Introduction

The need for nitrogen fertilizers in agricultural areas has made its production on an industrial scale mandatory (Gezerman, 2020). Although Ammonium nitrate is the most widely used fertilizer worldwide, various processes such as gradual evaporation have been developed to produce ammonium nitrate on an industrial scale (Gezerman, 2020). In all production methods of ammonium nitrate, chemicals are added to limit its moisture sorption properties (Gezerman, 2021). The chemical used to limit the hygroscopic properties of ammonium nitrate is not a preferred method by manufacturers due to the cost problem (Tyc et al., 2021; Tyc et al., 2020).

In order to deal with the caking phenomenon in a systematic way, the process parameters must be analyzed. Various methods have been developed in the computational and statistical chemistry literature for this analysis process (Grimme et al., 2018; Brendel et al., 2011). The methods used in the whole statistical process resulted in an error calculation with their closeness to the real values encountered in the process conditions (Thiel et al., 2018; Leszczynski et al., 2014). For this reason, the form of the analysis gains importance in the methods developed to minimize the error calculation for the solution of the encountered problem (Downs et al., 2003; Dykstra et al., 2011).

To improve the caking properties of ammonium nitrate, it is essential to analyze the process parameters (Štrbová et al., 2020; Elzaki et al., 2020). The production process of ammonium nitrate consists of the reaction of anhydrous ammonia and nitric acid, the evaporation way of the water in the content of ammonium nitrate, which has a concentration of around 70-80%, and the addition of anti-caking chemicals in order to limit the moisture absorption capacity by granulating it after it reaches 99% concentration (Hong et al., 2020; Chen et al., 2019). The bulk conditions of ammonium nitrate subjected to the storage process after such a production process can be considered as the most important parameter affecting the physical characterization of ammonium nitrate (Afrassiabian et al., 2019; Tyc et al., 2021).

Optimizing the production conditions by analyzing the process parameters and thus solving the caking problem seems to be a preferable method for the manufacturer, rather than the cost burden of the anti-caking chemicals added to the ammonium nitrate production process after granulation. In this study, while characterizing the caking problem in the ammonium nitrate production process with the regression analysis evaluated within the scope of the 6 sigma quality method system, a certain numerical range was determined for each parameter and an alternative solution was found for the producers to minimize the caking problem of ammonium nitrate by providing optimization for the production conditions. It is aimed to propose a solution.

2. Method

2.1 Process and Chemical Raw Material Analysis

In this study, anhydrous ammonia, nitric acid, anti-caking chemicals were used as raw materials for the ammonium nitrate production process. Anhydrous ammonia (99.9% w/w) and nitric acid (55% w/w) were obtained from Toros Agri-Industry. Amine-based coating oils were used as anti-caking material.

2.2 Regression Analysis

Regression analysis was performed using the minitab program within the scope of 6 sigma quality management and analysis system.

3. Results

In order to understand the effect of the parameters affecting the caking phenomenon in the production process of ammonium nitrate, it is essential to analyze the whole process. In Table 1, the contribution of all process parameters to the process for each stage in the production process is expressed numerically. A score of 10 was given for the value that affected the process the most and 1 point was given for the value that affected the process the least.

In order to perform the rating process, the effect of process parameters on caking should be determined and the effect of multiple variables on this rating should be analyzed in the regression analysis. For this purpose, in a detailed process analysis made on the system, as seen in Table 1, a quantitative evaluation of all parameters affecting the final product in the process was made. The highest rating corresponding to the quantitative values determined within these parameters can be considered as the parameters included in the regression analysis. As the regression analysis parameters, product and storage humidity, coating oil ratio and nitrogen content were determined as the most important parameters of the regression analysis. In addition, the effect of bulk height on the particle physical strength and the effect on the caking process determine the limits of the regression analysis when evaluated in terms of stock life.

Table 1. Process parameters and grading of the effect of these parameters on caking

No	Process step	Parameter	Effect on caking
1	storage	Storage temperature	10
2	storage	Storage humidity	10
3	storage	bulk height	10
4	cooling	Air Temperature	9
5	cooling	air humidity	9
6	coating	Coating feature	9
7	storage	Stocking time	9
8	bagging	Package type/ property	9
9	shipment	Particle size (bulk / packed)	8
10	shipment	Climate conditions	8
11	prilling	Particle size	8
12	prilling	bucket cycle	8
13	prilling	product moisture	8
14	cooling	Ammonium nitrate temperature after cooling	8
15	coating	Coating oil spray system Nozzle cleaning	8

16	mixing tank	Ammonium nitrate temperature in the mixing tank	7
17	mixing tank	post-reaction storage tank fertilizer inlet temperature	7
18	mixing tank	The amount of $Ca(NO_3)_2$ formed	7
19	mixing tank	Chemical composition of the fertilizer	7
20	Mixing tank heating	Ammonium nitrate temperature at mixing tank heating inlet	7
21	prilling	Product temperature at the base of the Prilling Tower	7
22	cooling	Soğutma giriş AN sıcaklığı	7
23	coating	coating oil temperature	7
24	II. stage evaporation	Saturator vapor pressure	7
25	II. stage evaporation	Steam flow rate	6
26	prilling	bucket mechanism	6
27	storage	Handling temperature	6
28	ammonium nitrate production reactor	Ammonium nitrate temperature at the reactor outlet	5
29	ammonium nitrate production reactor	pH of the reaction medium	6
30	I. stage evaporation	Ammonium nitrate concentration at the end of stage I.	5
31	II. stage evaporation	I. stage system pressure	5
32	II. stage evaporation	Ammonium nitrate concentration at the end of the II. stage evaporation	5
33	II. stage evaporation	II.stage system pressure	5
34	mixing tank	physical appearance	5
35	prilling	bucket number	5
36	prilling	bucket cleaning	5
37	coating	coating oil pressure	5
38	coating	coating oil Nozzle type	5
39	storage	outdoor/indoor environment	5
40	shipment	Delivery vehicle (road, seaway, railway)	5
40	Uptake of nitric acid	Concentration of nitric acid	4
41	Reactor (production of ammonium nitrate)	reactor pressure	4
42	Reactor (production of ammonium nitrate)	reactor temperature	4
43	Reactor (production of ammonium nitrate)	Liquid ammonium nitrate level in the reactor	4
44	Reactor (production of ammonium nitrate)	Ammonium Nitrate temperature at the reactor outlet	4
45	1st stage evaporation	1st stage ammonium nitrate temperature	4
46	1st stage evaporation	Ammonium nitrate feed flow at the end of the first stage evaporation	4
47	II. stage evaporation	II. stage feed tank temperature	4
48	II. stage evaporation	II. stage Ammonium Nitrate feed flow	4
49	prilling	Scraper speed in bucket	4

50	cooling	Cooling system cleaning/maintenance	4
51	mixing tank	Moisture content of fed raw materials (%)	7
52	Reactor (Ammonium nitrate production)	Raw material inside the reactor Distributor maintenance	3
53	coating	coating oil Drum speed	3
54	coating	coating oil Drum cleaning	3
55	Evaporation of ammonia	Ammonia temperature	2
56	Evaporation of ammonia	Pressure of ammonia	2
57	Evaporation of ammonia	Impurities in ammonia	2
58	Evaporation of ammonia	water content of ammonia	2
59	Evaporation of ammonia	Liquid ammonia level	2
60	Uptake of nitric acid	nitric acid temperature	2
61	Uptake of nitric acid	nitric acid pressure	2
62	I. stage evaporation	Ammonium nitrate tank level in stage I	2
63	Evaporation of ammonia	The temperature of the cooling water	1
64	Evaporation of ammonia	The pressure of the cooling water	1
65	Evaporation of ammonia	Ammonia flow rate taken into the reaction medium	1
66	Uptake of nitric acid	nitric acid flow	1

3.1 Regression Analysis

It is an analysis method used to measure the relationship between two or more numerical variables. If the analysis is done using a single variable, it is called univariate regression, if more than one variable is used, it is called multivariate regression analysis. With the regression analysis, information can be obtained about the existence of the relationship between the variables and, if there is, the strength of this relationship.

In regression, one of the variables must be dependent and the other independent variable. The logic here is that the variable on the left of the equation is affected by the variables on the right. The variables on the right are not affected by other variables. Being unaffected here, in a mathematical sense, means that when these variables are put into a linear equation, they do.

In order to analyze the caking problem of ammonium nitrate using the regression method, it is necessary to determine the parameters that directly affect the caking parameters. For this, first of all, the amount of coating oil added to the process medium after ammonium nitrate is granulated, the bulk height and fracture strength, which determines the physical strength of ammonium nitrate, and the moisture absorption properties of ammonium nitrate are included in the regression analysis.

During these processes, along with the formation reaction of ammonium nitrate, various methods are developed as the storage method and production process of hygroscopic ammonium nitrate, and it provides significant progress in solving the problem of caking of nitrogen fertilizers during storage in industrial production processes.

The determination of the optimal level with the experimental design for the regression analysis to eliminate the undesired caking (caking) problem of the Ammonium Nitrate fertilizer was carried out by considering the following mechanisms.

- Increasing the cooling efficiency in the drying system after the in-unit granulation process
- Air-conditioning the warehouse in terms of temperature and humidity conditions
- A constantly moving triper mechanism during warehouse product transfer
- An additional cooling system after coating the granulated product
- Alternatives of the DAP solution added to the reaction medium for product stabilization can contribute to the system.
- Decreasing grain size can have a significant impact on reducing degradation problem.

- Because controllable particle size can reduce degradation problem.

In the Boxplot diagram made as a part of the regression analysis, it is seen that the coating oil applied to the particle surface during production for the purpose of caking characterization and the bulk height are the two parameters that most affect the caking parameter (Tables 2-5).

Table 2. Change in caking amount at minimum bulk height (6 m)

Bulk Height (m)	Coating Amount (p.p.m.)	Nitrogen Concentration (%)	Crushing Strength (kg/pril)	Caking Rate (%)
6	488	25,5	2,66	0,65
6	487	25,9	2,69	0,75
6	497	26,3	2,67	0,85
6	491	25,7	2,58	0,77
6	504	26,4	2,53	0,66
6	482	25,8	2,68	0,81

It is seen that at a fixed bulk height of 6 m, the amount of caking does not reach the maximum rate of 1% for caking (Table 2).

Table 3. Change in caking amount at maximum bulk height (10 m)

Bulk Height (m)	Coating Amount (p.p.m.)	Nitrogen Concentration (%)	Crushing Strength (kg/pril)	Caking Rate (%)
10	554	26,5	2,58	1,4
10	557	25,8	2,71	1,5
10	589	25,9	2,69	1,3
10	596	25,6	2,63	1,1
10	568	26,1	2,85	1
10	566	26,3	2,64	1,2

In another data analysis conducted to understand the effect of bulk height on caking, it is seen that the caking rate exceeds 1% for a bulk height of 10 m (Table 3).

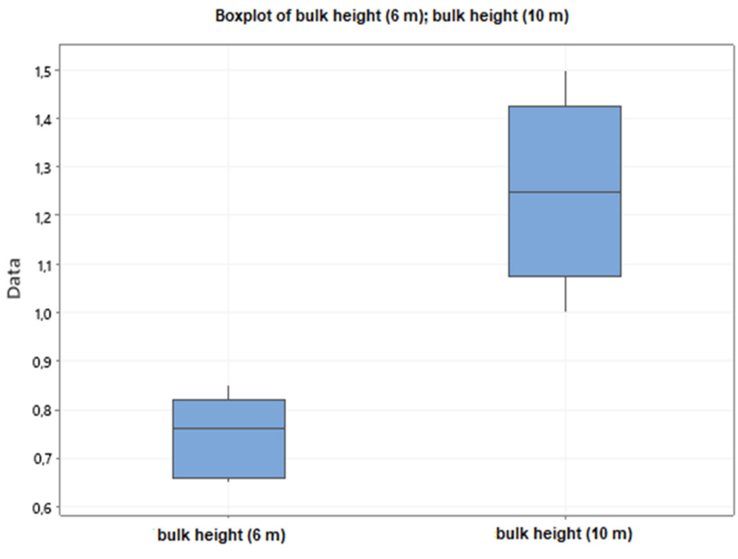

Figure 1. Comparison of 6 m and 10 m bulk heights with Box plot analysis

In a Box plot analysis performed as a part of the regression analysis, the effect of bulk height on the caking rate, as seen in Figure 1, increases with the increase in the height of the stockpiled bulk. Because, as can be seen from the experimental data, there is an increase in the degradation tendency with the increase of the pressure on the ammonium nitrate particles with the increase in the bulk height. According to the box plot analysis, the tendency to caking at bulk height spreads over a wider area.

Table 4. Change in caking amount at maximum coating oil (450 p.p.m) concentration

Coating amount (p.p.m)	Nitrogen Concentration	Crushing strength (kg/prill)	Bulk Height(m)	Caking (%)
450	25,9	2,71	6,1	1,2
450	25,8	2,82	6,3	0,89
450	25,9	2,69	5,8	0,99
450	25,7	2,59	6	0,95
450	26	2,54	5,9	0,96
450	25,8	2,68	6	1,3

In the data analysis made with reference to the enterprise data in the caking rate characterization, it is seen that the amount of coating oil used has a significant effect on the caking rate (Table 4, 5). 450 – 550 p.p.m ranges have been determined as coating oil concentrations recommended by the manufacturer for application under operating conditions. For the 450 p.p.m value, which is the minimum coating oil rate determined for the study, the caking rate, which is the maximum target, was determined as 1% (Table 3). Based on this, it was determined that as the coating oil ratio increased, the caking ratio was lower (Table 5).

Table 5. Change in caking amount at maximum coating oil (550 p.p.m) concentration

Coating amount (p.p.m)	Nitrogen Concentration	Crushing strength (kg/prill)	Bulk Height(m)	Caking (%)
550	25,8	2,57	5,8	0,9
550	25,9	2,56	6	0,85
550	25,6	2,63	5,7	0,77
550	25,7	2,75	5,9	0,96
550	26,1	2,66	6,1	0,88
550	25,3	2,68	6	0,69

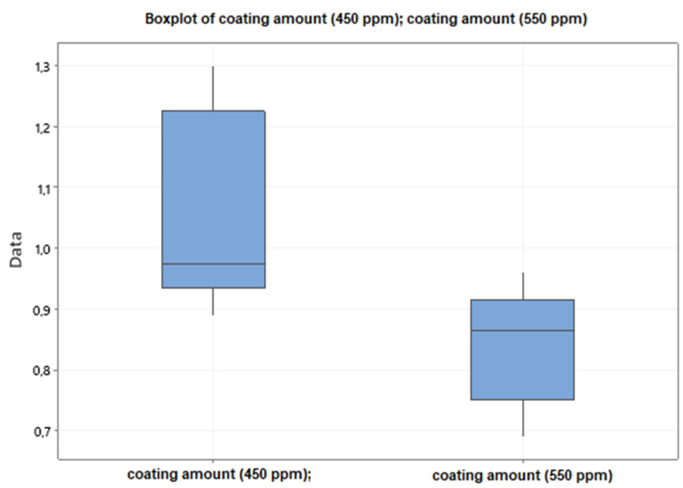

Figure 2. Comparison of coating oil amounts of 450 p.p.m and 550 p.p.m with Boxplot analysis

In another Boxplot analysis, in order to observe the effect of coating oil on caking tendency for ammonium nitrate particles, different coating oil ratios were studied and it was observed that the effect on caking tendency at relatively low concentrations of coating oil was observed in a wider range. It was observed that as the amount of coating oil increased, the caking rate decreased and the caking tendency of the same particles was in a lower range (Figure 2).

3.2 Control of Variables and Their Effects on Regression Analysis

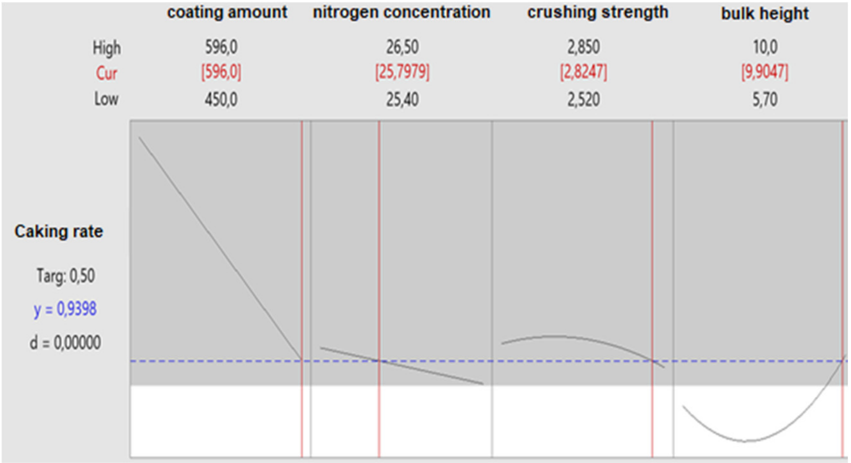

Figure 3. Examination of the effect of parameter values in operating conditions on the caking ratio by regression analysis

In a regression analysis made with reference to various operating parameters, it was determined that the caking rate was measured as 0.9398% at a nitrogen content of 25.79%, with a bulk height of 9.9047 m, a coating thickness of 596 p.p.m, and ammonium nitrate particles with a physical strength of 2.8247 kg/pril (Figure 3). In such a regression analysis, the nitrogen concentration of ammonium nitrate particles varied between 26.50% and 25.40%. In this regression analysis, the system parameters at a caking rate of 0.9398% were 450 - 596 p.p.m for the coating thickness particles, the nitrogen concentration changed between 25.40 - 26.50%, the physical strength (crushing strength 2.52 - 2.85 kg/pril, the bulk height 5.7-10 m).

In the current situation, approximately 600 p.p.m of coating thickness at a bulk height of 10 m and ammonium nitrate fertilizer production with a crushing strength of 2.82 kg/pril are produced. In the current situation, a caking value of close to 1% is obtained in the stocked product.

3.3 Improvements that Can Be Made in Current Conditions

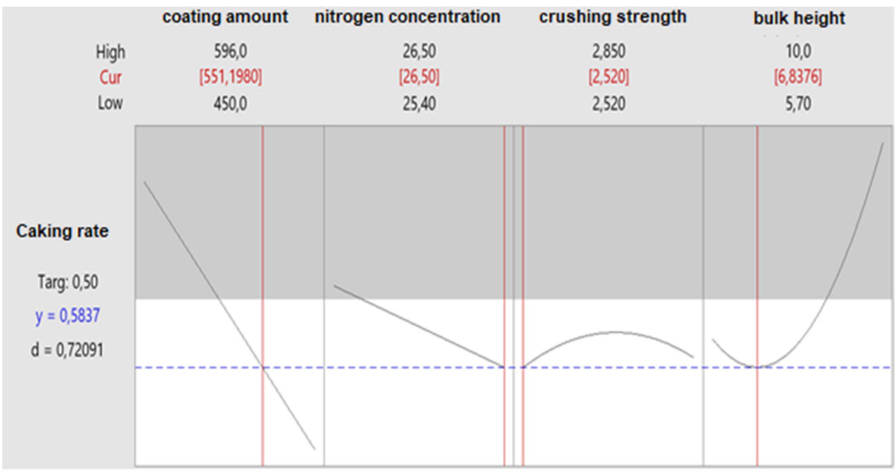

Figure 4. Investigation of the effect of operating parameter values on the caking rate under certain conditions by regression analysis (coating oil: 551 ppm, Nitrogen concentration: 26.5%, crushing strength: 2.52 bulk height 6.83)

According to the regression analysis, in order to reach the targeted caking value (0.5%) under current conditions, a bulk height of 7 m is obtained under storage conditions, and a caking rate of 0.58% is obtained when 550 p.p.m of coating oil is covered.

In the same regression analysis used to monitor the caking rate of ammonium nitrate particles with different operating parameters, this time, when the amount of coating oil is decreased and the nitrogen concentration is increased, the crushing strength decreases, while the caking value corresponding to these variables is 0.5837% when the bulk height is decreased (Figure 4).

3.4 Regression Analysis - Situation Assessment

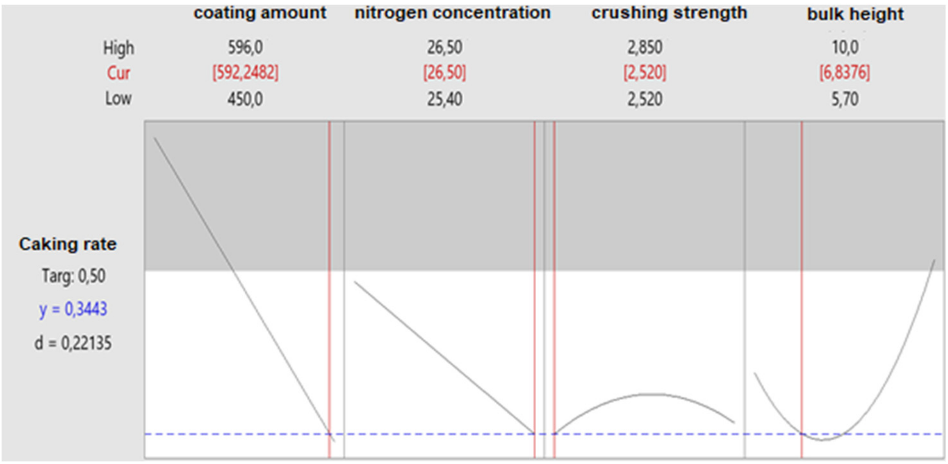

Figure 5. Investigation of the effect of increased coating oil ratio on the caking ratio by regression analysis (coating amount: 592 ppm, Nitrogen: 26.5%, crushing strength: 2.52 bulk height 6.83)

The minimum caking ratio that can be achieved by regression analysis, with the operating parameters, the coating oil ratio is 592.24 p.p.m, and the ammonium nitrate particles have a physical strength of 2.52 kg-pyril, a bulk height of 6.83 m, and a nitrogen content of 26.50%, a minimum of 0.3443%. caking rate can be determined (Figure 5). It is one of the important results of the regression analysis that this caking rate can be achieved by using the recommended amount of coating oil used by the manufacturer as the bulk height decreases.

In another regression analysis, the conditions were evaluated in order to reduce the caking rate to the lowest possible rate under the current conditions, and as a result, approximately 600 p.p.m of coating amount, 26.5% nitrogen concentration and 2.52 kg/prill crushing strength were obtained and a bulk height of approximately 7 meters was obtained. with the lowest caking rate can be obtained.

3.5 Current Situation-Regression Analysis

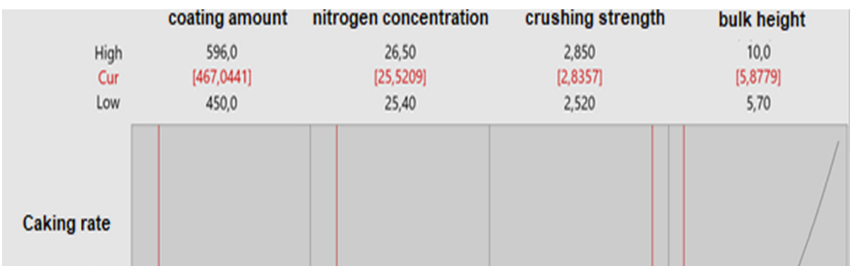

Figure 6. Investigation of the effect of reduced bulk height on the caking ratio by regression analysis (coating amount: 467 ppm, Nitrogen: 25.52%, crushing strength: 2.83, bulk height 5.87 m)

The decrease in the amount of coating oil as an anti-caking chemical draws attention as another parameter that triggers caking in this regression analysis (Figure 6). For the regression analysis, in which different variations are tried in order to improve the physical properties of the fertilizer obtained as a result of the process conditions in the current situation, the caking rate was determined as 0.74% by the regression analysis under the conditions where the amount of coating oil used is the least under current conditions and the bulk height is limited to 6 meters.

3.6 Expectation-Regression Analysis

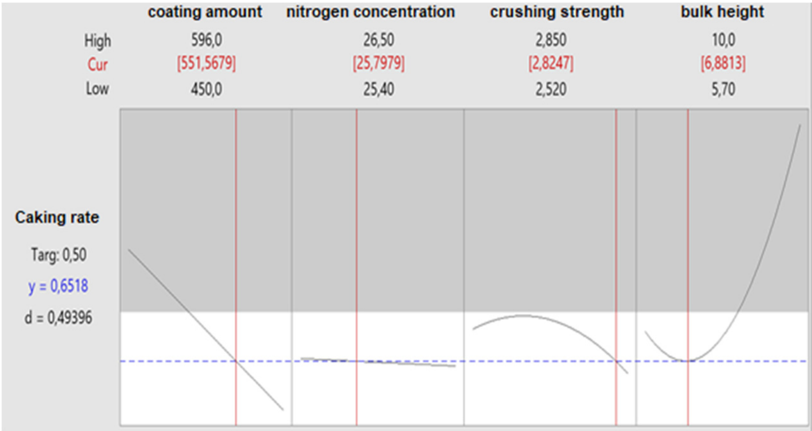

Figure 7. Investigation of the effect of reduced nitrogen concentration and increased fracture strength on the caking rate by regression analysis (coating oil: 551 ppm, Nitrogen: 25.79%, Crushing strength: 2.82, bulk height: 6.88 m)

By reducing the coating oil feed from 600 p.p.m to 550 p.p.m, 0.65% caking can be achieved at 7 m bulk height (Figure 7). In a regression analysis carried out to improve caking rates by taking the existing operating conditions as a reference, 551 p.p.m coating oil rate and 25.79% nitrogen content for a 0.65% caking rate within acceptable limits, ammonium nitrate particles with a crushing strength of 2.82 kg/prill were determined as 6.88. It has been determined that a caking rate of 0.65% can be achieved with a bulk height of m.

In this study, an optimum was determined by examining the effects of crushing strength, coating oil ratio, nitrogen content and bulk height on the caking rate by a Box plot and regression analysis made with 6 sigma methodology, and by examining the caking rate in the current operating conditions, and this optimum was determined. Under these conditions, the caking parameter was determined and the relevant parameters were examined in detail. Accordingly, as a result of the regression analysis, it was determined that the coating oil and bulk height parameters were the parameters that most affected the caking rate.

4. Conclusion

As can be seen in the Regression analysis carried out in order to systematically understand the caking problem of ammonium nitrate and to minimize this problem, the following determinations were made in the modeling of the experimental setups in which each experimental parameter was kept constant under certain conditions in a 6-variable experiment optimization under fertilizer production conditions.

- In storage conditions after nitrogen fertilizer production; It can be seen from the optimization conditions that the moisture content in the fertilizer during production has the most important effect on degradation.
- As another parameter, it has been observed that limiting the bulk height to 6 meters is effective in terms of moisture content, with the observation that it has a direct effect on the crushing strength.
- In storage conditions after nitrogen fertilizer production; It has been seen that the effect of bulk height in the fertilizer during production has the most important effect on the caking problem. In addition, the effects of crushing strength, coating oil and nitrogen contents on the caking rate were also investigated in the regression analysis.
- It has been observed that limiting the bulk height has a positive effect on caking, with the observation that it has a direct effect on crushing strength as another parameter.
- In case of reducing the amount of in-process chemicals such as coating oil used for system improvements, there will be financial gain, as seen in the regression analysis.
- The caking rate will decrease to 0.65% if the nitrogen value is not decreased below 25.80%, the coating oil feed is not below 550 p.p.m during production, and the bulk height in the warehouse does not exceed 7 meters.
- It is recommended to gradually increase the bulk height while bulking during warehouse use, and not exceed 7 meters at the final height

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