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## Modeling and Optimizing Aluminum Hydroxide Precipitation Process in Industrial Scale; case study: Iran Alumina Plant

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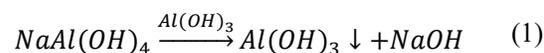
### Abstract

The precipitation of aluminum hydroxide from a supersaturated sodium aluminate solution is known as an essential production step in the Bayer process. In this work, the real precipitation process in the Iran Alumina Plant was modeled by the historical data with the help of Design Expert. According to the results obtained, the recovery is significantly improved with decrease in the super-saturation factor ( $\alpha$ ) of the solution. However, this modification was found to be the most difficult change due to the operational problems. The results obtained indicated the significant impact of the seed size on the product size. The negligible effects of the other parameters involved on controlling the amount of fine grains ( $< 44 \mu\text{m}$ ) and coarse grains ( $> 150 \mu\text{m}$ ) in the product showed the significance of reactivating the classification and agglomeration sections. Ultimately, it was found that the recovery process could be enhanced from 46.32% to 47.86% at a constant  $\alpha$  by increasing the seed concentration to 400 g/L, increasing the retention time by adding two precipitation tanks and reducing the temperature of the last precipitation tank by 2 °C (by reducing the temperature of the inlet suspension), while preserving the quality of the product.

## 1. Introduction

The Bayer process consists of dissolving bauxite in a sodium hydroxide solution, separating the insoluble red mud containing iron, silica, and other impurities, filtrating, and precipitating and calcining aluminum hydroxide in order to produce alumina. This method was invented 130 years ago, and is still the most commonly used technique to convert bauxite to alumina for the industrial aluminum and alumina production [1]. The recent variations in the Bayer method include the removal of silica impurities before dissolution and carbonate and oxalate after precipitation [2]. By definition, crystallization is the formation of solid particles in a homogeneous phase. This phenomenon is usually a slow process, in which the molecules are removed from the solution and a crystal structure is formed by the combination of molecules. Generally, precipitation comprises the relatively rapid formation of a soluble solid

phase from a soluble liquid phase [3]. The following reaction shows the seed precipitation stage [4]:



The three fundamental phenomena in the precipitation of aluminum hydroxide in the Bayer process include nucleation, growth, and agglomeration. Fresh fine grains are formed via nucleation. Crystal growth is the process in which the solute is deposited from a supersaturated solution on the crystal surfaces. The agglomeration process can be represented as a process in which the particles collide and adhere to each other to form larger particles. This process has a faster mechanism than the crystal growth through layer-by-layer surface integration. Moreover, it is greatly affected by external forces. The agglomeration process plays the most crucial role in increasing the

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particle size of the product [3]. Different studies have focused on the role of inorganic and organic impurities in the hydrate growth. According to the results of these studies, the majority of organic and inorganic impurities in the Bayer solution have resulted from bauxite [5]. Different molecular weight fractions of the Bayer-degraded humic substances have been found to have different detrimental effects on the gibbsite precipitation yields and crystal sizing, and surface area [6]. The classic theory of nucleation presented by Volmer (1939) assumes that the clusters in the solution are formed by an additional mechanism and stretched until a critical size is obtained. The critical radius represents the critical core size, and is specified by the maximum value on the Gibbs free energy curve. Re-dissolution is desirable in terms of energy at values less than the critical radius as can be seen from the  $\Delta G$  slope. At values larger than the critical radius, the crystal growth is more favorable due to a decrease in the free energy. The maximum  $\Delta G_{critical}$  at the critical radius,  $r_c$ , is obtained from the following equations [3]:

$$\frac{d(\Delta G)}{dr} = 0 \quad (2)$$

$$r_c = \frac{-2\gamma}{\Delta G_g} \quad (3)$$

The formation rate of nuclei by this mechanism is expressed by an Arrhenius-type function, where  $A$  is an experimental factor, being equal to 1030 nuclei/cm<sup>3</sup>s.

$$B_0 = Ae^{\left(\frac{-\Delta G_{critical}}{kT}\right)} \quad (4)$$

The super-saturated Bayer solutions are completely different from the common mineral salt solutions, and spontaneous nucleation hardly happens. In order to produce alumina and reduce the nucleation time, a large amount of seeds are supposed to be added so as to accelerate the precipitation of aluminum hydroxide [7]. The rate of crystal growth is dependent on the temperature, super-saturation (fast liquid cooling), and nature of fluid flow in the vicinity of the boundary layer. In the absence of thermodynamic barriers, the appropriate locations will be rapidly filled up, eventually leading to a complete crystal lattice. The crystal growth on smooth surfaces with a high surface energy is difficult, and this mechanism is dominant in the crystal lattice defects. Typically, the fluid flow does not directly affect the growth rate, and the crystal growth rate is not dependent on the nuclei [8]. Agglomeration might consist of different steps, namely crystal collision,

absorption, rupture, and cementation of crystals. The factors affecting the agglomeration step include the degree of super-saturation, temperature, retention time, concentration and solid particle size distribution (PSD), hydrodynamic conditions, impurities, and solid morphology [9]. Different strategies and methods have been proposed in order to enhance the rate of precipitation from the sodium aluminate solution. Optimizing the operational conditions, seed activation, and employing additives are the main methods proposed for hydrate production [10]. The researchers have investigated the factors controlling the precipitation of finer alumina trihydrate particles. According to their results, the hydrate particle size decrease with increase in the temperature. Finer hydrate particles have been produced by increasing the surface area, seed concentration, and soda concentration [11]. In another study, the effect of process parameters on the agglomeration of crystals has been evaluated. To this end, the temperature (55 °C to 70 °C), super-saturation factor,  $\beta$  (1.4-1.8), seed concentration (10 to 150 g/L), stirring speed (350 rpm to 515 rpm), and average seed size (2  $\mu$ m to 10  $\mu$ m) have been investigated. The results obtained have shown an increase in the agglomeration at high temperatures (70 °C) and high super-saturation (1.8); these two parameters have the highest impact on the agglomeration rate. The agglomeration rate increase with increase in the seed concentration up to 100 g/L, and then slowly decreases. At a seed concentration of 10 g/L, the agglomeration rate decreases with increase in the stirring speed from 330 rpm to 520 rpm. However, the maximum agglomeration rate occur at stirring speeds of 400-500 rpm and a seed concentration of 50 g/L. Furthermore, the agglomeration rate decreases with increase in the seed size [12]. In general, these results have also been confirmed in other studies [13-16].

The gibbsite precipitation in uniform and non-uniform mixing tanks has also been investigated. The results obtained show the significant impact of PSD on the agglomeration and nucleation rates [17]. The influence of temperature, seed concentration, and mixing rate has been reviewed on the agglomeration kinetics in the batch Bayer process. The results obtained have revealed that the agglomeration constant (agglomeration kernel) increase with an increase in the temperature and mixing rate. The maximum agglomeration rate was observed as a function of the seed concentration. Furthermore, the estimated value of the activation energy was approximate to growth [18]. Despite

the multiple studies for improving the growth of gibbsite particles, the main problems with the aluminum hydroxide precipitation stage, namely the low ratio of the product and long process time, are still visible in these studies. There has been a great interest in the kinetics of crystallization and agglomeration in a discontinuous cell, and especially in the modification of the key parameters including the concentration of caustic soda, super-saturation, seeds, and elimination of impurities [19-24]. Other studies on the enhancement of recovery or product quality can generally be divided into four groups: seed activation, addition of significant amounts of additives, addition of surface-active agents (surfactants), and use of ultrasonic waves or magnetic fields. Recently, there has been a special interest in the use of active seeds produced through different techniques in order to increase the precipitation rate. For example, the use of active seeds produced by mixing methods increased the precipitation rate by 3% to 4% [25]. In another study, by adding methanol to the sodium aluminate solution (1:1, vol/vol), 170 g/L  $\text{Na}_2\text{O}$ , and caustic to aluminate (1:1.55) at 60 °C for a retention time of 24 h, the recovery increased to 70%, which was 20% greater than the seeded precipitation of coarse-grained particles in the Bayer process in at least 50 h [4]. Other studies have also reported an increase in the precipitation rate from the sodium aluminate solution by adding ethylenediaminetetraacetic acid (EDTA) and its sodium salt ( $\text{Na}_4\text{EDTA}$ ) to the solution [10]. The effect of lithium ions on seed precipitation has also been investigated. The results obtained have indicated that the presence of lithium ions in the solution enhanced the percent of fine particles and also the rate of secondary nucleation, eventually leading to an increase in the precipitation efficiency. Nevertheless, the increase in the number of fine and shell-type grains adhered to the coarse grains of the product promotes a decrease in the strength of the product (alumina) [26]. Numerous studies have been conducted on the use of surfactants, for example, adding a non-ionic surfactant and an ether additive, respectively, to improve the precipitation rate by 6% and 4% [27, 28]. The researchers have reported a 3% increase in the precipitation rate by applying a magnetic field for 45 h [29]. In another study, a precipitation yield of 45% was obtained in 15-30 h using ultrasonic waves compared to 40-50 h of the conventional precipitation [30].

Despite the promising results of the above-mentioned studies regarding the increased

precipitation rate and improved quality of the product, most of them have been conducted on a laboratory scale, and employing their results on the industrial scale could increase problems or production costs. For example, given the presence of different impurities in industrial plant solutions, the use of surfactants may increase impurities. Moreover, the use of ultrasonic waves, magnetic fields, and addition of active seeds will exacerbate the undesirable conditions. On the other hand, the utilization of considerable amounts of additives also increases the production costs as well as the separation costs of additives. Changing the characteristics of the bauxite feed to the Bayer process causes changes in the properties of the sodium aluminate solution. Moreover, temperature variations in different seasons and temperature reduction during the process in the precipitation tanks make the scalability of the laboratory results to industrial conditions inaccessible and unreliable.

For the first time, in this work, the industrial conditions were realistically simulated. To this end, by examining the real data collected in 1 year (three shifts in one day) and using the historical data method in Design-Expert, the precipitation process in the Iran Alumina Plant was modeled. The precipitation process was modeled using the industrial data in order to investigate the effects of changes in the characteristics of the industrial sodium aluminate solution, characteristics of seed, and temperature variations in different seasons in the precipitation tanks on the process recovery and product quality. It is practically impossible to obtain these results in the laboratory tests. To this end, 11 parameters and 3 responses were evaluated. The parameters investigated in this work included the initial temperature of precipitation, final temperature of precipitation, retention time, seed concentration, percent of fine grains in the seed (finer than 44  $\mu\text{m}$ ), percent of coarse grains in the seed (coarser than 150  $\mu\text{m}$ ),  $\text{SiO}_2$  impurity in the seed,  $\text{Fe}_2\text{O}_3$  impurity in the seed, percent of  $\text{Al}_2\text{O}_3$  in the sodium aluminate solution (known as liquor), and percent of  $\text{Na}_2\text{O}_c$  in the liquor and the liquor super-saturation ( $\alpha$ ). Three responses including the recovery, percent of fine grains in the product (finer than 44  $\mu\text{m}$ ), and percent of coarse grains in the product (coarser than 150  $\mu\text{m}$ ) were also considered.

## 2. Materials and Methods

### 2.1. Precipitation process in industrial plant

The super-saturated sodium aluminate solution (called liquor) in the Iran Alumina Plant with a



exchangers). The effective retention time for agglomeration is 6-8 h, and the appropriate temperature equals 76 °C. The hydrate suspension is precipitated into the first tank after a complete agglomeration and after passing through secondary exchangers, where the coarse-grained seeds are added to the tank. At the same time, this part of the process was left out due to the process problems and the inability to regulate and control the situation. Sandy alumina is produced in the Iran Alumina Plant. According to the initial design regarding the product quality, the grain-yield of the produced hydrate was considered a maximum of 10% for the particles coarser than 105 μm and finer than 45 μm. These values were increased to a maximum of 10% for the particles coarser than 150 μm and those finer than 44 μm as the appropriate particle size distribution (PSD) of the product.

**2.2. Data preparation and modeling**

The flow rate of the sodium aluminate solution, slurry temperature and level in the precipitation tanks, and hydrate flow rate were monitored by a distributed control system (DCS). The sodium aluminate solution and product were sampled regularly (three shifts per day). The chemical properties of the liquor ( $Na_2O_c$  and  $Al_2O_3$ ) were measured by the titration methods. The particle size distribution of the product was analyzed by a particle size analyzer (PSA, Fritsch A22) with a precision of 0.001 μm. The impurities in the hydrate were measured by an X-ray fluorescence spectrophotometer (XRF, Siemens-SRS3000, Germany) with a precision of 100 ppm. Alpha ( $\alpha$ ) in this manuscript refers to the super-saturation factor as obtained from the following equation:

$$Alpha = \alpha = 1.645 \times \frac{Na_2O_c}{Al_2O_3} \tag{5}$$

where  $Na_2O_c$  and  $Al_2O_3$ , respectively, show the concentrations of caustic soda (active caustic) and aluminum oxide in the liquid phase, both in terms of g/L.

Also the recovery of the precipitation process is defined as the difference between the ratio of the amount of dissolved  $\alpha$  after and before the precipitation process to the amount of the dissolved  $\alpha$  after precipitation, which is calculated from the following equation:

$$Recovery = \frac{\alpha_2 - \alpha_1}{\alpha_2} \tag{6}$$

where  $\alpha_1$  and  $\alpha_2$ , respectively, represent the super-saturation factor of the sodium aluminate solution before and after the precipitation process.

After collecting and normalizing, the following conditions were considered in order to verify the subject and calculate the required parameters:

- The useful volume of the precipitation tanks was considered to be equal to 2300 m<sup>3</sup>.
- According to the calculated mean retention time, the liquor profile in the last two days was considered as the liquor entering the precipitation unit.
- The specifications (particle size and impurities) of the produced hydrate in the last two days were considered as the precipitation seed.

The average values of the real data (parameters and responses) collected in a year are reported in Table 1.

**Table 1. Average parameters and actual responses of the precipitation process in the year.**

Seed (g/L)	Time (h)	Seed size		Impurity of seed		Temperature of liquor		Liquor			R (%)	Product size (microns)	
		-44 μ (%)	+150 μ (%)	SiO <sub>2</sub> (%)	Fe <sub>2</sub> O <sub>3</sub> (%)	T in (°C)	T out (°C)	Na <sub>2</sub> O <sub>c</sub> (g/L)	Al <sub>2</sub> O <sub>3</sub> (g/L)	Alpha		-44 μ (%)	+150 μ (%)
324.47	53.07	11.29	18.41	0.009	0.010	56.92	51.22	128.44	128.41	1.646	46.32	11.39	18.16

The response surface method (RSM) and *Historical Data* with low (the minimum value of the parameter in an interval) and high (the maximum value of the parameter in an interval) levels were used for optimizing the precipitation process. These values were used as the inputs to the software (Table 2). As it can be seen in Table 2, the parameters of the industrial precipitation process

are highly fluctuating due to the use of various bauxite resources (concentration variations and process impurities), changes in the caustic concentration (due to evaporation and the desalting unit, PU18), changes in the operational flow rate due to repair or industrial problems, and changes in the regional climatic conditions throughout the year.

**Table 2. Low (minimum parameter value) and high (maximum parameter value) levels in the precipitation process (according to the industrial data).**

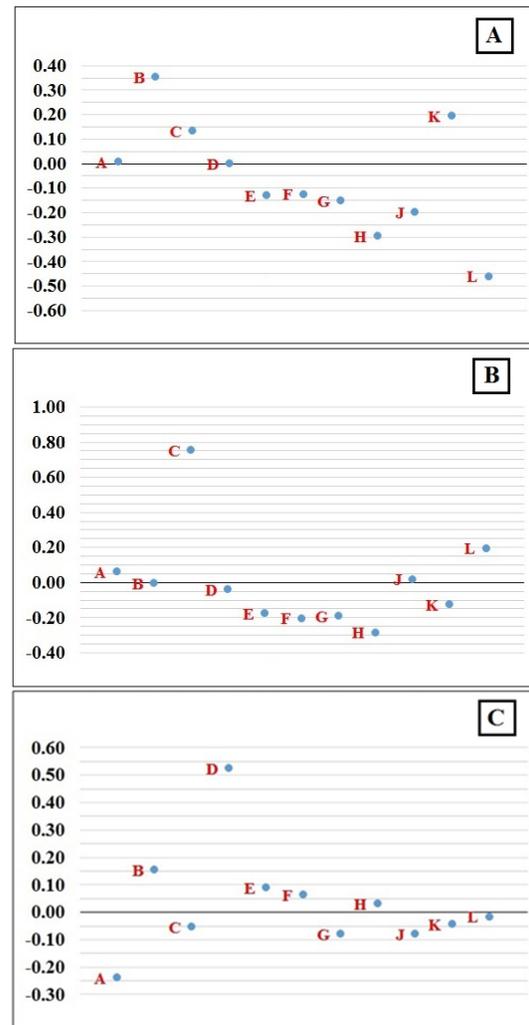
Factor	Name	Unit	Low actual	High actual
A	Seed	g/L	232.67	411.84
B	Time	h	41.68	92.29
C	Seed size, -44 microns	%	6.01	17.92
D	Seed size, +150 microns	%	10.97	29.69
E	Impurity of seed-SiO <sub>2</sub>	%	0.007	0.017
F	Impurity of seed-Fe <sub>2</sub> O <sub>3</sub>	%	0.007	0.021
G	Temperature in	°C	48.90	64.70
H	Temperature out	°C	42.00	58.00
J	Liquor-Na <sub>2</sub> O <sub>e</sub>	g/L	107.30	137.00
K	Liquor-Al <sub>2</sub> O <sub>3</sub>	g/L	109.10	142.00
L	Liquor-Alpha	-	1.53	1.83

### 3. Results and Discussion

#### 3.1. Primary modeling

The correlation coefficients of the parameters were first examined as compared to different responses. In the case of recovery response, the liquor  $\alpha$  and the retention time were significantly correlated, respectively, with correlation coefficients of -0.46 and 0.35. The percent of particles finer than 44  $\mu\text{m}$  and coarser than 150  $\mu\text{m}$  in the seed, respectively, with the correlation coefficients of 0.75 and 0.52 were significantly correlated with the percent of the particles finer than 44  $\mu\text{m}$  and coarser than 150  $\mu\text{m}$  in the product. Figure 2 shows the correlation coefficients of the parameters and responses.

After fitting different models to the data and removing the data outside the range (43 rows of 1095 data rows were removed due to the industrialization of data and high volatility), the results obtained from modeling different responses in the precipitation process and effective parameters were analyzed. In the case of the recovery of the precipitation process, the linear model was selected as the best fit through power transformation. According to the selected model, the analysis of variance (ANOVA) is provided in Table 3. Considering a P-value less than 0.05, the selected model and parameters are statistically significant at a very high confidence level. Moreover, the validation data presented in Table 4 indicates the very good fitness of the model to the data.



**Figure 2. Correlation coefficients of parameters (A) on the efficiency, (B) Percent of fine grains (< 44  $\mu\text{m}$ ), and (C) percent of coarse grains (> 150  $\mu\text{m}$ ).**

**Table 3. ANOVA of the model obtained for the precipitation recovery.**

Source	Sum of squares	df	Mean squares	F-value	p-value Prob > F	
Model	2.186E + 010	11	1.987E+009	161.64	< 0.0001	Significant
A-Seed	2.283E + 008	1	2.283E+008	18.57	< 0.0001	
B-Time	1.796E + 009	1	1.796E+009	146.12	< 0.0001	
E-Seed, SiO <sub>2</sub>	1.234E + 009	1	1.234E+009	100.35	< 0.0001	
G-Temperature in	3.457E + 008	1	3.457E+008	28.13	< 0.0001	
H-Temperature out	1.075E + 009	1	1.075E+009	87.44	< 0.0001	
K-Liquor, Al <sub>2</sub> O <sub>3</sub>	1.074E + 008	1	1.074E + 008	8.73	0.0032	
L-Liquor, alpha	8.127E + 009	1	8.127E + 009	661.14	< 0.0001	
BK	1.264E + 008	1	1.264E + 008	10.28	0.0014	
EG	1.186E + 008	1	1.186E + 008	9.65	0.0019	
EL	6.358E + 007	1	6.358E + 007	5.17	0.0232	
GL	1.199E + 008	1	1.199E + 008	9.76	0.0018	
Residual	1.278E + 010	1040	1.229E + 007			

**Table 4. Validation of the model obtained for the recovery response in the precipitation process.**

		Desired limit
Adj R-Squared	0.6310	
Pred R-Squared	0.6271	
(Adj R-Squared)-(Pred R-Squared)	0.0039	< 0.2
Adeq precision	108.391	> 4

The final equation for the actual factors is presented as follows:

$$(R)^{2.86} = +7.34774E + 005 + 21.79299 \times \text{Seed} - 1364.77151 \times \text{Time} + 9.81616E+006 \times \text{Seed, SiO}_2 - 6509.66959 \times \text{Temperature in} - 538.04711 \times \text{Temperature out} - 1059.86222 \times \text{Liquor, Al}_2\text{O}_3 - 3.33464E+005 \times \text{Liquor, alpha} + 12.88256 \times \text{Time} \times \text{Liquor, Al}_2\text{O}_3 - 95397.14640 \times \text{Seed, SiO}_2 \times \text{Temperature in} - 3.12186E+006 \times \text{Seed, SiO}_2 \times \text{Liquor, alpha} + 4170.36922 \times \text{Temperature in} \times \text{Liquor, alpha}$$

Considering the two responses related to the product quality, namely the percent of fine (finer than 44 μm) and coarse grains in the product (coarser than 150 μm), diagrams B and C in Figure 2 and preliminary modeling in Design Expert indicate the significant impact of the seed size (parameters C and D) on the particle size distribution of the product. Due to the significant impact of these parameters, the effect of other parameters seem to be practically insignificant. This result also shows the importance of classifying seeds during the process into fine and coarse grains, and activate the agglomeration stage (according to the preliminary design of the plant). ANOVA of the model obtained for estimating the percent of fine and coarse grains in the product is, respectively, shown in Tables 5 and 6.

**Table 5. ANOVA of the model obtained for estimating the percent of fine grains in the product in the precipitation process.**

Source	Sum of squares	df	Mean squares	F-value	p-value Prob > F	
Model	5.78	7	0.83	282.32	< 0.0001	Significant
B-Time	0.060	1	0.060	20.62	< 0.0001	
C-Seed size, - 44 microns	3.33	1	3.33	1139.63	< 0.0001	
D-Seed size, +150 microns	0.23	1	0.23	77.40	< 0.0001	
G-Temperature in	0.029	1	0.029	9.87	0.0017	
H-Temperature out	0.19	1	0.19	65.01	< 0.0001	
CD	0.015	1	0.015	5.21	0.0226	
C <sup>2</sup>	0.20	1	0.20	69.95	< 0.0001	
Residual	3.05	1044	2.923E - 003			
Cor total	8.83	1051	0.83			

**Table 6. ANOVA of the model obtained for estimating the percent of coarse grains in the product in the precipitation process.**

Source	Sum of squares	df	Mean squares	F-value	p-value prob > F	
Model	5084.14	8	635.52	144.88	< 0.0001	Significant
A-Seed	83.26	1	83.26	18.98	< 0.0001	
B-Time	35.72	1	35.72	8.14	0.0044	
C-Seed size - 44 microns	51.49	1	51.49	11.74	0.0006	
D-Seed size +150 microns	3611.41	1	3611.41	823.30	< 0.0001	
H-Temperature out	115.51	1	115.51	26.33	< 0.0001	
AH	45.25	1	45.25	10.32	0.0014	
BH	40.78	1	40.78	9.30	0.0024	
D <sup>2</sup>	260.42	1	260.42	59.37	< 0.0001	
Residual	4575.11	1043	4.39			
Cor total	9659.25	1051				

After fitting different models to the obtained data, the quadratic model was finally selected as the best fit through logarithmic transformation in order to estimate the percent of the fine grains in the product. The P-values (< 0.05) in Table 5 clearly show that the selected model and parameters are significant at a very high confidence level. Furthermore, the validation data in Table 7

indicates a very good fit of the model to the data. The quadratic model was selected as the best fit for the percent of coarse grains in the product. According to the P-values (< 0.05) in Table 6, the selected model and parameters are significant at a very high confidence level. Furthermore, the validation data presented in Table 8 indicates a very suitable fit of the model to the data.

**Table 7. Validation model obtained for the percent of fine grains in the product.**

	Desired limit
Adj R-Squared	0.6543
Pred R-Squared	0.6520
(Adj R-Squared)-(Pred R-Squared)	< 0.2
Adeq precision	> 4

**Table 8. Validation model obtained for the percent of coarse grains in the product.**

	Desired limit
Adj R-Squared	0.5263
Pred R-Squared	0.5227
(Adj R-Squared)-(Pred R-Squared)	< 0.2
Adeq precision	> 4

Finally, the equation for estimating the responses with real factors for the two quality responses is presented as follows:

- For the percent of fine grains in the product (as a response):

$$\text{Log}_{10}(\text{Product size} - 44 \text{ microns}) = + 1.03905 - 1.23192\text{E-}003 \times \text{Time} + 0.066285 \times \text{Seed size} - 44 \text{ microns} - 6.13368\text{E-}004 \times \text{Seed size} + 150 \text{ microns} - 2.79962\text{E-}003 \times \text{Temperature in} - 6.71216\text{E-}003 \times \text{Temperature out} + 5.04726\text{E-}004 \times \text{Seed size} - 44 \text{ microns} \times \text{Seed Size} + 150 \text{ microns} - 2.00822\text{E-}003 \times \text{Seed size} - 44 \text{ microns}^2$$

- For the percent of coarse grains in the product (as a response):

$$\text{Product size} + 150 \text{ microns} = + 100.11183 - 0.19716 \times \text{Seed} - 0.49958 \times \text{Time} + 0.098812 \times \text{Seed size} - 44 \text{ microns} - 0.75080 \times \text{Seed size} + 150 \text{ microns} -$$

$$1.56866 \times \text{Temperature out} + 3.63240\text{E-}003 \times \text{Seed} \times \text{Temperature out} + 0.010595 \times \text{Time} \times \text{Temperature out} + 0.036632 \times \text{Seed size} + 150 \text{ microns}^2$$

### 3.2. Effects of parameters

#### 3.2.1. Response 1: Recovery

According to the F-values in Table 3, the supersaturation factor of the liquor entering the precipitation stage is the most significant factor affecting the recovery response of the precipitation process. The percent of SiO<sub>2</sub> impurity in the seed, final temperature of precipitation, initial temperature of precipitation, and seed concentration are the other important factors involved in the precipitation process. It is noteworthy that the percent of Al<sub>2</sub>O<sub>3</sub> in the liquor is also one of the influential factors with a less

effectiveness. The proposed model shows four interactions between the parameters. Figure 3 shows the effects of the factors on the response, and Figure 4 shows the simultaneous effect of the parameters.

The positive and negative effects of different parameters on the recovery are presented in Figure 3. Considering the slope of the curves and F-values in the ANOVA table, the super-saturation factor and time have the most significant impact on the recovery. As the super-saturation factor decreases from 1.83 to 1.53, the recovery increases from 39.35% to 51% (Graph G), and when  $\alpha$  decreases from 1.68 to 1.58 (in the middle values of the other parameters), the recovery will increase by 3.5%. Moreover, the recovery only increased by 0.76% when the retention time of the precipitation process increased by 10 h (Graph B). Given that the crystallization rate is proportional to the surface area of seeds per unit volume, the recovery of the precipitation process increased by about 0.54% by increasing the seed concentration from 322.26 g/L to 411 g/L (Graph A). The process recovery increased by reducing the initial and final temperatures of the precipitation tank, especially by reducing the temperature of the last precipitation tank in cold seasons due to an increase in the amount of nucleation and a decrease in the  $\text{Al}_2\text{O}_3$  solubility, and the subsequent increase in the super-saturation factor (Graphs D and E). The process recovery seems to be decreased by increasing the percent of  $\text{SiO}_2$  impurity in the seed (soluble impurities) and the  $\text{Al}_2\text{O}_3$  concentration in the liquid phase at a constant  $\alpha$  as seen in Graphs C and F (in fact, the liquid phase thickens). Considering the effects of the existing parameters and operational conditions, the recovery of the precipitation process can be increased by reducing the super-saturation factor of the input liquor, increasing the retention time (with increasing the number of precipitation tanks), reducing the temperature, and increasing the seed concentration.

Considering the interaction effects of the parameters in Figure 4, the recovery significantly decreases with increasing the  $\text{Al}_2\text{O}_3$  concentration in the liquid phase at low retention times (Graph A). However, the recovery will probably increase at high retention times due to the sufficient time for nucleation and growth and discharge of the liquid phase. The percent of  $\text{SiO}_2$  impurity at high temperatures at the beginning of the precipitation process and high super-saturation factors have a more negative effect on the efficiency (Graph B and C). Regarding the effect of temperature at the beginning of the precipitation, there is no

significant change in the efficiency at high super-saturation factors but the reduction in the initial temperature of the precipitation tank has a greater effect at lower super-saturation factors (Graph D).

### 3.2.2. Response 2: Percent of fine grains in product (finer than 44 $\mu\text{m}$ )

According to the F-values in Table 5, the percent of fine grains in the seed has the greatest effect on this response. After this parameter, the percent of coarse grains in the seed, final temperature of precipitation, and time and the initial temperature of precipitation have, respectively, the greatest effects on the precipitation process. Notably, there is a large difference between the F-values of the percent of fine grains in the seed as compared to the other parameters. It can be argued that the effect of other parameters, except the seed size, is insignificant on the fine grains in the product. The proposed model shows an interaction between the fine and coarse grains in the seed. Figures 5 and 6, respectively, show the effects of factors and the interaction effects of the parameters.

The graphs in Figure 5 show the positive and negative effects of different parameters on the percent of fine grains in the product. The percent of fine grains in the seed has the greatest impact on the percent of fine grains in the product (Graph B). By increasing the retention time, the percent of fine grains is reduced in the product due to sufficient time for the particle growth (Graph A). Increasing the percent of coarse grains in the seed also increases the percent of fine grains in the product probably due to breaking coarse grains by hydrodynamic forces (Graph C). Decreasing the temperature at the beginning and end of the precipitation process caused an increase in the nucleation rate and the percent of fine grains in the product (Graph D and E). Considering the slopes of curves, the temperature reduction at the end of precipitation has a more significant impact on the response. The low slope of the graphs for the various parameters, except the seed size, indicates the insignificant effect of these parameters on this response. This confirms the need to activate the agglomeration section in the process.

Due to the interaction effect of the percent of fine and coarse grains in the seed on the percent of fine grains in the product (Figure 6), when the percent of fine grains in the seeds is low, the percent of coarse grains in the seeds has an insignificant effect on the percent of fine grains in the product. However, the effect of the percent of coarse grains in the seed increased with increasing the percentage of fine grains in the seed.

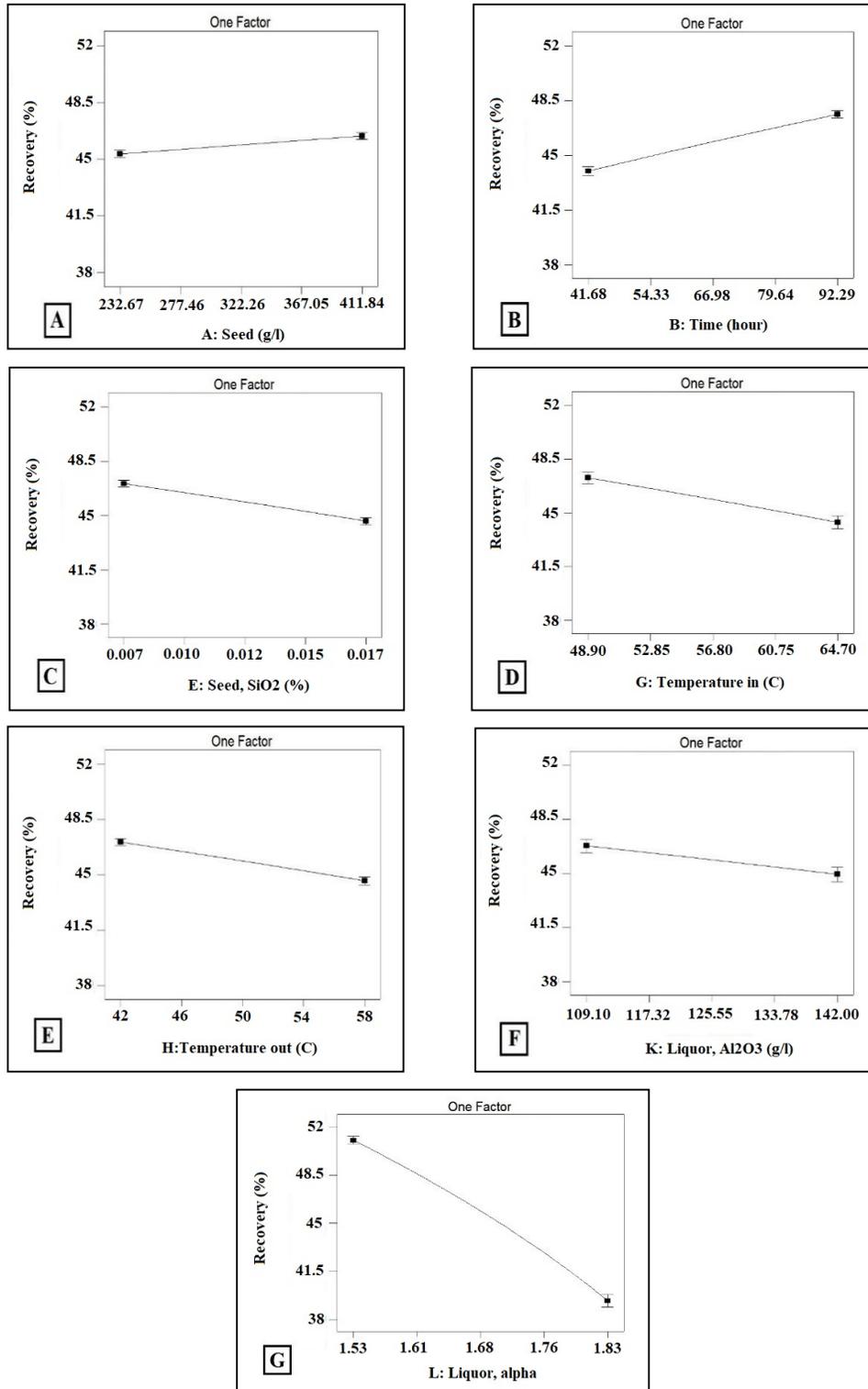


Figure 3. Effects of different factors on the recovery of the precipitation process (other parameters are in their intermediate values in each graph).

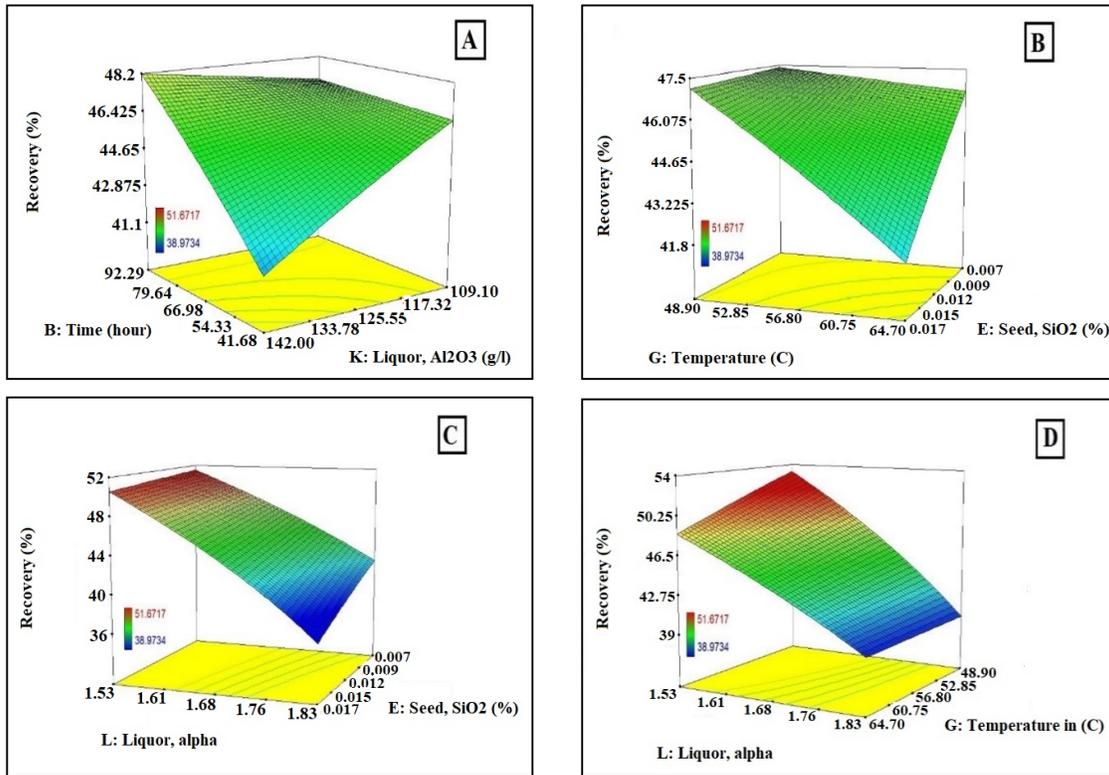


Figure 4. Interaction effects of the parameters on the recovery of the precipitation process (other parameters are in their intermediate values in each graph).

**3.2.3. Response 3: Percent of coarse grains in product (coarser than 150 μm)**

According to the F-values in Table 6, the percent of coarse grains in the seed has a significantly greater impact than the other parameters on the percent of coarse grains in the product. After this parameter, the final temperature of the precipitation process, seed concentration, particle size, and retention time are, respectively, effective in making the product coarser in the precipitation process. The proposed model also shows two interactions between the parameters. Figure 7 shows the effects of various factors on this response, and Figure 8 displays the simultaneous effects of the parameters.

The positive and negative effects of different parameters on the percent of coarse grains in the product are shown in Figure 7. By increasing the seed concentration, the percent of coarse grains decreases in the product (Graph A). This is likely due to the increased particle collisions in the suspension. Increasing the percent of coarse grains in the seeds has the most significant effect on the percent of coarse grains in the product (Graph D). By increasing the retention time and the percent of fine grains in the seed, the percent of coarse grain

grains in the product is increased partially (Graph B and C). The increasing temperature at the end of the precipitation process also causes an increase in the percent of coarse grains in the product due to the reduced nucleation (Graph E). As mentioned in the previous section, the seed size has the greatest effect on the percent of coarser grains in the product; it can be said that due to the lack of classification and agglomeration in this plant, there is no control over the fine-grained and coarse-grained particles in the product.

According to the interaction of the parameters in Figure 8, by increasing the seed concentration at high final temperatures, the percent of coarse grains in the product increases. In contrast, an increase in the seed concentration at low final temperatures reduces the percent of coarse grains in the product (A). Regarding the retention time of the precipitation process, when the temperature at the end of the precipitate is at its upper limit, increasing the retention time increases the percent of coarse grains (probably due to the increased growth of hydrate particles). However, increasing the retention time at low final temperatures causes a slight decrease in the percent of coarse grains in the product.

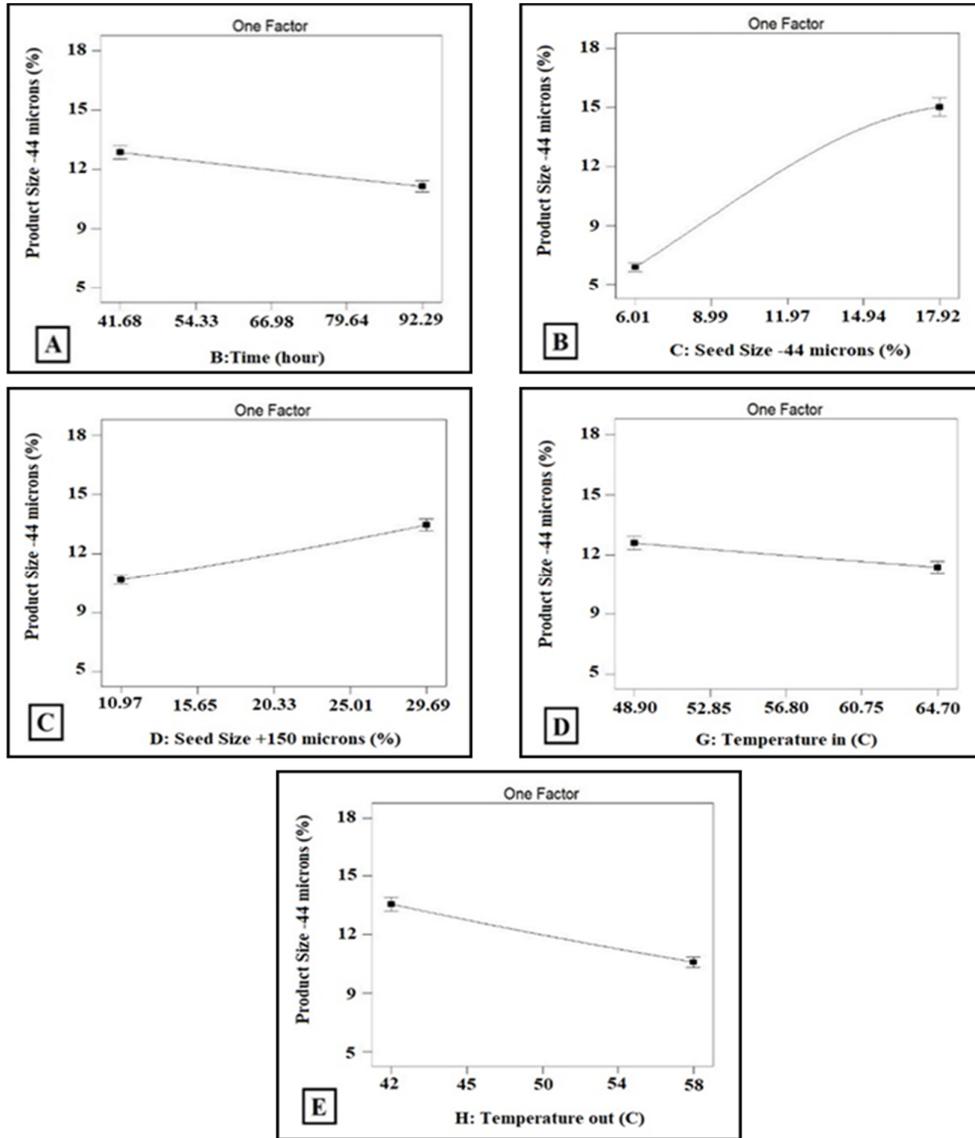


Figure 5. Effects of different factors on the percent of fine grains in the product (other parameters are in their intermediate values in each graph).

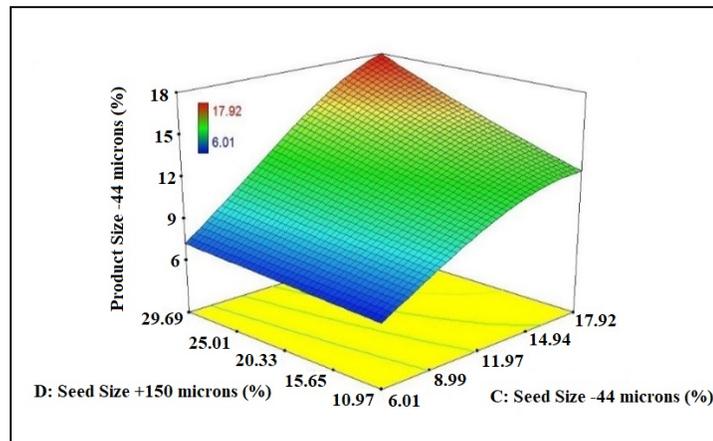


Figure 6. Interaction effect of the parameters on the percent of fine grains (other parameters are in their intermediate values).

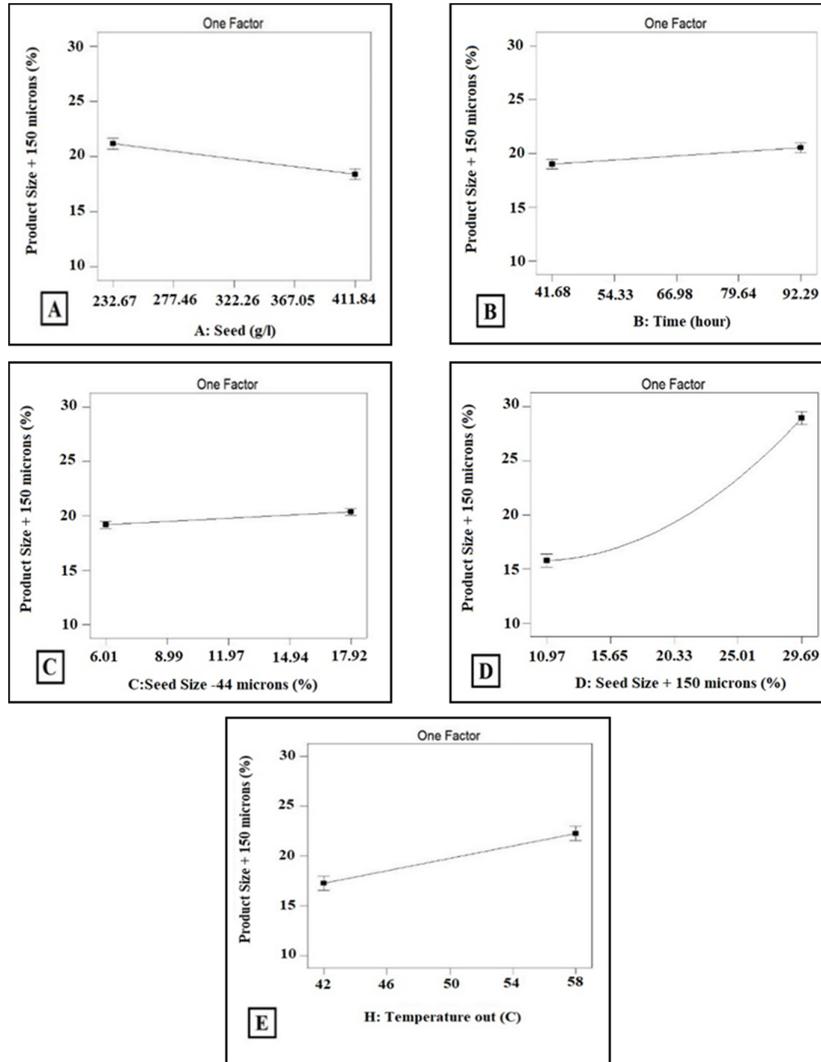


Figure 7. Effects of different factors on the percent of coarse grains in the product (other parameters are in their intermediate values in each graph).

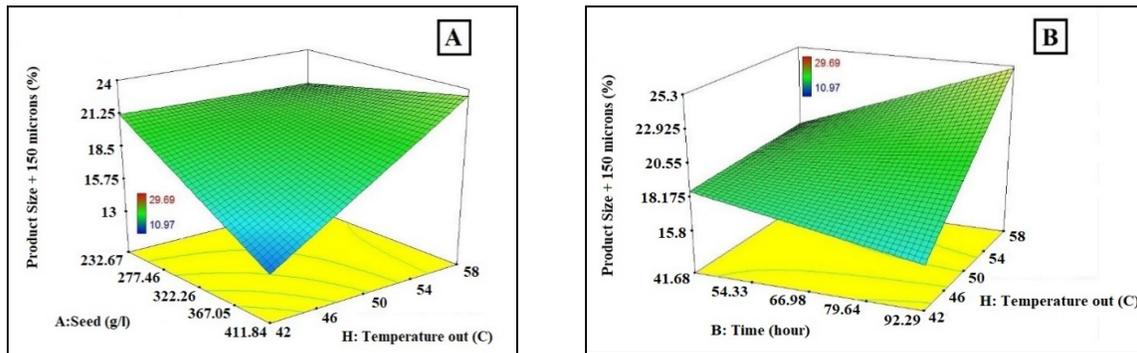


Figure 8. Interaction effects of parameters on the percent of grains in the product in the precipitation process (other parameters are in their intermediate values).

### 3.3. Optimization

The operating conditions in the aluminum hydrate production plants vary across the world due to differences in the bauxite quality, caustic

soda concentration, the Bayer liquor concentration, and the operational conditions. Most studies on the laboratory scale have investigated increasing the efficiency of the precipitation process according to

the type of alumina (sandy or floury) by changing the operating conditions. In this research work, the three seed concentrations of 500, 600, and 700 g/L, three temperatures of 55, 60, and 65 °C, three soda concentrations of 140, 145, and 150 g/L, and three super-saturation factors of 1.56, 1.65, and 1.73 were considered. Finally, the seed concentration of 600 g/L, a temperature of 55 °C, a low caustic soda concentration of 140 g/L, and  $\alpha = 1.56$  were selected as the best conditions [31].

Considering the effects of various parameters in the previous section, it can be stated that the results of this work obtained from modeling the actual and operational data of the Iran Alumina Plant are consistent with those obtained in various laboratory studies. For example, the literature shows an increase in the process recovery at a high super-saturation (low  $\alpha$ ), a low temperature, a high retention time, and seed concentration [7, 16, 17, 31]. Furthermore, various studies indicate the importance of agglomeration and classification in the production of hydrate and product quality control [12, 14, and 18]. Finally, due to the elimination of the agglomeration section and the classification of hydrate in the Iran Alumina Plant and possibly the impossibility of setting them up in a short timeframe, the following items were considered in order to increase the recovery and improve the quality of the product taken into account the existing operational conditions:

- Reducing the final temperature of the precipitation process by 2 °C (by reducing the temperature of the liquor at the beginning of precipitation).
- Increasing the seed concentration up to 400 g/L.

- Increasing the retention time by adding one or two precipitation tanks (3.38 h according to the average suspension flow rate).

- Decreasing  $\alpha$  by changing the  $\text{Na}_2\text{O}_c$  concentration in the liquor.

- Due to the impossibility of seed classification, the seed particle size was considered in its mean and constant values.

The above items were evaluated separately (Runs 2 to 6), binary (Runs 7 to 11), triple (Runs 12 and 13), and concurrently (Runs 14 and 15). The proposals are presented in Table 9 in order to enhance the recovery (as the priority of the production process) and also to reduce the percent of fine and coarse grains in the product. According to Run 1 in Table 9 and compared to the data in Table 1, the models are able to well-predict the actual response of the process. As it can be seen, decreasing  $\alpha$  will significantly increase the recovery due to the great impact of this parameter. On the other hand, the operational conditions and problems led to the reduction of  $\alpha$  as the most difficult operational alternative. Consequently, the changes in other parameters were examined. It appears that the recovery of the precipitation process can be increased by up to 1.5% without changing  $\alpha$  by the simultaneous change of the other parameters. Moreover, these changes will reduce the percent of coarse grains in the product but it is necessary to control the size of the input seeds (by classification and agglomeration) to more precisely control the product size. As it can be seen from the data in Table 9, the changes in the product size are not large without changing the seed size.

**Table 9. Validation and optimization of the model obtained for the precipitation process.**

Run	Seed (g/l)	Time (hour)	T in (°C)	T out (°C)	Liquor			R (%)	Predicted Response	
					$\text{Na}_2\text{O}_c$ (g/l)	$\text{Al}_2\text{O}_3$ (g/l)	Alpha		Product size -44 $\mu$ (%)	Product size +150 $\mu$ (%)
1	324.47	53.07	56.92	51.22	128.44	128.41	1.646	46.32	11.39	18.16
2	324.47	53.07	56.92	51.22	119.59	128.41	1.532	49.99	11.39	18.16
3	324.47	53.07	54.92	49.22	128.44	128.41	1.646	46.89	11.90	17.81
4	324.47	56.45	56.92	51.22	128.44	128.41	1.646	46.59	11.28	18.30
5	324.47	59.84	56.92	51.22	128.44	128.41	1.646	46.86	11.17	18.45
6	400.00	53.07	56.92	51.22	128.44	128.41	1.646	46.78	11.39	17.32
7	324.47	56.45	54.92	49.22	128.44	128.41	1.646	47.16	11.78	17.89
8	324.47	59.84	54.92	49.22	128.44	128.41	1.646	47.42	11.67	17.96
9	400.00	53.07	54.92	49.22	128.44	128.41	1.646	47.34	11.90	16.42
10	400.00	56.45	56.92	51.22	128.44	128.41	1.646	47.04	11.28	17.46
11	400.00	59.84	56.92	51.22	128.44	128.41	1.646	47.31	11.17	17.61
12	400.00	56.45	54.92	49.22	128.44	128.41	1.646	47.60	11.78	16.50
13	400.00	59.84	54.92	49.22	128.44	128.41	1.646	47.86	11.67	16.57
14	400.00	56.45	54.92	49.22	122.94	128.41	1.575	49.98	11.78	16.50
15	400.00	59.84	54.92	49.22	123.33	128.41	1.58	50.06	11.67	16.57

#### 4. Conclusions

In this work, we aimed to model and optimize the precipitation process using the real process data in one operational year of the Iran Alumina Plant in order to increase the recovery and quality of the product. To this end, 11 parameters including the initial and final temperatures of the precipitation process, retention time, seed concentration, percent of fine grains in the seed (finer than 44  $\mu\text{m}$ ), percent of coarse grains in the seed (coarser than 150  $\mu\text{m}$ ), percent of  $\text{SiO}_2$  impurity in the seed, percent of the  $\text{Fe}_2\text{O}_3$  impurity in the seed, percent of  $\text{Al}_2\text{O}_3$  in the liquor, percent of  $\text{Na}_2\text{O}_c$  in the liquor and  $\alpha$  and also three recovery responses, percent of fine grains in the product (finer than 44  $\mu\text{m}$ ), and percent of coarse grains in the product (coarser than 150  $\mu\text{m}$ ) were considered in order to increase the recovery and quality of the product. After modeling and investigating the effects of the above parameters,  $\alpha$  was found to be the most significant parameter affecting the recovery so that the recovery of the precipitation process significantly increased by reducing  $\alpha$  (increasing super-saturation). However, due to the operational problems, the reduction in the bauxite modulus entering the plant, and other limitations, changing this parameter was the most difficult task operationally. Finally, regarding the available facilities and operational conditions, increasing the process retention time by adding one or two precipitation tanks, increasing the seed concentration in the precipitation process up to 400 g/L, and reducing the temperature of the last precipitation tank by 2 °C were evaluated by reducing the temperature of the input suspension. According to the results obtained, by modifying these three parameters, the process recovery could be increased by 1.5%. In order to increase the recovery up to 50%, it is necessary to reduce the alpha value of the liquor entering the precipitation process to 1.58 (by adding two tanks, increasing the seed concentration, and decreasing the temperature). Furthermore, the model and diagrams for the effects of different parameters on the percent of fine and coarse grains showed the significant impact of the seed size on the product size. The low slope of the diagrams for other parameters confirmed the insignificant impact of these parameters, and in general, the lack of full control over the particle size distribution of the product. Finally, it seems that the use of classification and agglomeration (according to the preliminary design of the process) plays a significant role in the quality control of the product.

#### Acknowledgments

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#### Abbreviations

Ethylenediaminetetraacetic acid	EDTA
Process unit	PU
Caustic sodium oxide concentration in the liquor	$\text{Na}_2\text{O}_c$
Degrees of freedom	df
Sodium aluminate solution temperature at the first precipitation tank	$T_{in}$
Sodium aluminate solution temperature at the last precipitation tank	$T_{out}$
Recovery	R
Distributed control system	DCS

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## مدلسازی و بهینه‌سازی فرآیند ترسیب در مقیاس صنعتی، مطالعه موردی: کارخانه آلومینای ایران

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### چکیده:

در فرآیند بایر، ترسیب هیدروکسید آلومینیوم از محلول فوق اشباع آلومینات سدیم به عنوان یک گام اساسی شناخته می‌شود. در این پژوهش، فرآیند واقعی ترسیب در کارخانه آلومینای ایران با استفاده از روش Historical Data با کمک نرم‌افزار Design Expert مدلسازی شد. با توجه به نتایج به دست آمده مشخص شد که بازیابی با کاهش در مقدار فاکتور فوق اشباعیت ( $\alpha$ ) محلول به صورت قابل توجهی افزایش می‌یابد. این در حالی است که اصلاح این پارامتر به دلیل مشکلات عملیاتی به عنوان دشوارترین تغییر شناخته می‌شود. نتایج به دست آمده حکایت از اثرگذاری بسیار زیاد اندازه جوانه بر اندازه محصول دارد. اثرگذاری کم پارامترهای دیگر در کنترل مقدار بخش ریزدانه ( $< 44 \mu\text{m}$ ) و درشت دانه ( $> 150 \mu\text{m}$ ) در محصول، اهمیت فعالسازی مجدد بخش طبقه‌بندی و آگلومراسیون را نشان می‌دهد. در نهایت مشخص شد که بدون تغییر  $\alpha$ ، با افزایش مقدار جوانه تا ۴۰۰ گرم بر لیتر، افزایش زمان ماند با اضافه کردن دو تانک ترسیب و کاهش دمای تانک آخر ترسیب به اندازه ۲ درجه سانتیگراد (با کاهش دمای محلول ورودی) می‌توان با حفظ کیفیت محصول، بازیابی را از ۴۶،۳۲ به ۴۷،۸۶ درصد افزایش داد.

**کلمات کلیدی:** ترسیب هیدروکسید آلومینیوم، هسته‌زایی، رشد، آگلومراسیون، فوق اشباعیت.