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Mathematical model formation of the process of deriving boric acid from ulexite mineral by full factorial design method

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ABSTRACT

Ulexite is a sodium calcium boron hydrate used in producing compounds, its chemical formula is Na2O2CaO5B2O3.16H2O, and it superabounds in Turkey. One of the significant boron compounds derived from boron minerals is boric acid. The aim of this study is producing boric acid in the wake of interaction of ulexite with hydrochloric acid solution, and offering an alternative process to producing boric acid by forming the mathematical model of this processing. Full factorial design method has been used in the study. Some of the parameters used in the process have been made steady, and the others have been accepted as variable factor in formation of mathematical model. Invariant parameters are granule size, reaction time and stirring speed. Hydrochloric acid concentration has been accepted as temperature and solid to liquid ratio variable factors for the formation of mathematical model. Primarily, the mathematical model of the process and three-parameter full factorial desing have been evaluated in the measureless coordinate system, then the values in the model has been assessed by Student criteria, and the validity of the originated model has been controlled by Fisher criteria. Henceforwards the current model of the process has been formed in the real coordinate system.

Key words: ulexite, boric acid, full factorial design, mathematical model.

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1. INTRODUCTION

Boron is a crucial element used in many fields, especially strategic ones. However, boron is not directly used in numerous fields in element, it is mostly used in its compounds. Boron subsists in nature in the form of metal borates, mostly in the form of sodium, calcium, sodium-calcium and magnesium borates (Kum et. al., 1994). Those subsisting in nature prevalently are colemanite ($2CaO\ 3B_2O_3.5H_2O$), ulexite ($Na_2O2CaO5B_2O_3.16H_2O$) and tincal ($Na_2O2B_2O_3.10H_2O$). The products like boric acid, hydrated borax, anhydrite borax and sodium per borates are derived from these substances (Boncukcuoğu et. al., 2003, Durak and Genel, 2012). A lot of studies has been held on ulexite that is a sodium-calcium borate. In the studies, boric acid output has been researched by analysing the dissolution mechanism of ulexite in different solvents (İmamutdinova V. M., 1967). Dissolution kinetics has been analysed by using distinct parameters in the studies held. Dissolution kinetics of ulexite has been viewed in the solution saturated with CO_2 (Kocakerim et. al., 1993) and in the solution saturated with SO_2 (Alkan and Kocakerim, 1987). Further there are studies on deriving boric acid and borax from ulexite (Yapıcı et. al., 1994). The mechanism of deriving borax from ulexite has been scrutinized (Demircioglu and Gulensoy, 1977). Tunc et. al., (1999) have analysed dissolution mechanism of ulexite in sulphuric acid. İmamutdinova and Adrashitova (1970) have analysed the dissolubility of ulexite in acetic acid solutions, and found out that maximum solution ratio is in low acid concentration, and besides they have found that the solution ratio falls as acid concentration increases. Kunkul et. al., (1997) have researched the dissolubility of ulexite in ammonia solution saturated with CO_2 ,and they have found out that dissolution ratio soars with the increasement of the concentration the dissolution ratio. The studies conducted are related to solution mechanism.

Boric acid is a crucial raw material for industry and trade. The abatement of difficulties that are encountered in the production of boric acid will reduce the cost. With this study, it seems that the production of boric acid by the activation of ulexite with HCI, and the production of calcium chloride as byproduct is possible. Calcium chloride is utilised in numerous fields from agriculture to chemical industry (Fusheng et. al., 2011, Alexander et. al., 2006). Most of the studies related to ulexite are pertinent to the kinetics and mechanism of dissolution. In this conducted study, the mathematical modelling of the derivation process of production oriented boric acid has been performed. The product consisted by this modelling method may occur depending upon the reacting products. Full factorial design method has been used in numerous studies. A. R. Cestari et. al., (2008) have utilised full factorial design method in resolving anionic red dye in aqueous solutions. Özturk and Kavak (2004) have applied full factorial design method in boron removal from aqueous solutions by adsorption. Bashirov et al.,(2012) have applied full factorial desing method in production of hydrogen gas at sodiumborohydride with cobalt catalyst. A. P. Rodriquez et. al., (2008) have employed full factorial design method in crystalizing kluyveromyces lactis β-galactosidase enzyme. Kose (2008) has observed the clearance of colorant from agricultural residue with anion exchanger by means of full factorial design. Özturk and Kavak (2008) have used cerium oxide in aqueous solutions of boron waste, and full factorial design method in the adsorption of boron waste. Y. Seki et. al., (2006) have utilised full factorial design method in the boron removal from aqueous solutions with Al_2O_3 based adsorbants. Salafranga et. al., (1999) have researched the optimization of the supercritical liquid extraction of Iraganox 1076, Irgafos 168 and Chimassob 81 substances with full factorial design method. R. I. Trezona et. al., (2000) have viewed the erosionresistance of automotive polishers with full factorial design method.

It is possible to remove the deficiencies that have appered during getting the correlation in the full factorial design method and classic regression analysis used in this study, and among the coefficients of regression equations used in this study. In the study conducted, the experimental design has been determined by considering the determination of the matter and experimental results. The study consists of several phases. The results gotten after each phase help the determination of next phases of the experiment. Hence the experiment is provided to be controlled optimally. The whole differential parameters can be also changed by experimental design method, and the relations between variables can be assessed. Differently from other research methods, searching the relations between variables in this method raises the productivity of experiments substantially.

2. EXPERIMENTAL

2.1. Materials and Methods

In full factorial design method, the whole combinations of variables at maximum and minimum levels are taken into consideration. The application of full factorial design method is found with the formula of the number of the experiments to be tested for **n** slew levels of **k** slew variables.

$$N= \mathbf{n}^{\mathbf{k}} \quad (1)$$

In the study conducted, there are 3 variable values that is **k**=3. If these 3 factors change at two levels consisting of maximum and minimum, the number of the experiments to be tested should be $N=2^3 = 8$.

Supposing that there are three variables consisting of Z_1, Z_2 ve Z_3 in the study conducted. The range of these factors are as folows:

$$\begin{cases} Z_1^{min} \le Z_1 \le Z_1^{max} \\ Z_2^{min} \le Z_2 \le Z_2^{max} \\ Z_3^{min} \le Z_3 \le Z_3^{max} \end{cases} \quad (2)$$

As is seen, each Z_k changes between its own maximum and minimum values like Z_k^{min} and Z_k^{max} : $Z_k[Z_k^{min}, Z_k^{max}]$, k=1,2,3. as indicated before, 8 experiments should be held in order to take mathematical model as the number of factors are 3.

Initially the following calculations are done :

$$\left.\begin{aligned} Z_k^0 &= \frac{Z_k^{max} + Z_k^{min}}{2}, \text{k=1,2,3} \\ \Delta Z_i &= \frac{Z_k^{max} - Z_k^{min}}{2}, \text{k = 1, 2, 3.} \end{aligned}\right\} \quad (3)$$

Here the points of Z_1^0 , Z_2^0 ve Z_3^0 are called experimental design centre. ΔZ_k - is the changeability value of relevant variable. The values of the 8 experiment held in measureless and natural coordinate system have been shown in Table 1. Minimum values of Z_i have been displayed with -1 , and its maximum values with +1. ın compliance with this design, 8 experiments are tested and Y_{exp} basic material values derived from each experiment are added to Table 1. Henceforwards, mathematical model is formed

$Y_x = a_0 + a_1X_1 + a_2X_2 + a_3X_3 + a_{12}X_1X_2 + a_{13}X_1X_3 + a_{23}X_2X_3 + a_{123}X_1X_2X_3$ (4)

in dimensionless coordinate system. Coefficient values of model a_0, a_1, a_2, a_3, a_{12}, a_{13}, a_{23} ve a_{123}

$$a_j = \frac{1}{N}\sum_{i=1}^{N} X_{ji} Y_i \quad (5)$$

is computed with the formula above.

Table 1. The plan of full factorial design for three factors.

Dimensionless coordinate system				Natural coordinate system			
Experiment No	X_1	X_2	X_3	Z_1	Z_2	Z_3	Y^{tec}
1.	-1	-1	-1	Z_1^{min}	Z_2^{min}	Z_3^{min}	Y_1
2.	1	-1	-1	Z_1^{max}	Z_2^{min}	Z_3^{min}	Y_2
3.	-1	1	-1	Z_1^{min}	Z_2^{max}	Z_3^{min}	Y_3
4.	1	1	-1	Z_1^{max}	Z_2^{max}	Z_3^{min}	Y_4
5.	-1	-1	1	Z_1^{min}	Z_2^{min}	Z_3^{max}	Y_5
6.	1	-1	1	Z_1^{max}	Z_2^{min}	Z_3^{max}	Y_6
7.	-1	1	1	Z_1^{min}	Z_2^{max}	Z_3^{max}	Y_7
8.	1	1	1	Z_1^{max}	Z_2^{max}	Z_3^{max}	Y_8

Then in the experiment station, that is at the points of Z_1^0 , Z_2^0 ve Z_3^0 , three collateral experiments are tested, and the values of Y_1^0 , Y_2^0 və Y_3^0 out parameters are computed in accordance with them. After that, the mean value of these out parameters are found:

$$\overline{Y}^0 = \frac{\sum_u^3 Y_u^0}{3} \quad (6)$$

There after

$$S_1^2 = \frac{\sum_u^3 (Y_u^0 - \overline{Y})^2}{3} \quad \text{and} \quad (7)$$

$$S_{a_m} = \frac{S_1^2}{\sqrt{8}} \quad (8)$$

‘s values are computed. By considering the calculated (6), (7) and (8) values, and using Student criteria.

$$t_m = \frac{|a_m|}{S_{a_m}} \quad (9)$$

with the formula, the values found by the formula in (5) of the mathematical model in (4) are seen from private Student criteria table by using P=0.05 condition that they are f=2, $t_p(f)$=4.3 for these values. The values are utilised in the model in (4) by using the formula in (9) if each t_m value is larger than 4.3. The rest, that is the values which are lower than 4.3 are not used. Henceforwards, variences may be in the mathematical models in (4).

Thereafter, the validity of the new model is checked by Fisher criteria :

$$F = \frac{S_2^2}{S_1^2} \quad (10)$$

Here

$$S_2^2 = \frac{\sum_{i=1}^{8} (Y_i - \overline{Y}_i)^2}{N} \quad (11)$$

The mathematical model in (4) taken in dimensionless coordinate system is capable of supplying the actual process exactly. To form the valid mathematical model of natural process, the following change of variable formula in model (4) is used :

$$X_j = \frac{Z_j - Z_j^0}{\Delta Z_j} \text{ ,j=1,2,3.} \quad (12)$$

Mathematical model is taken as the follows in natural coordinate system after certain mathematical conversions :

$Y_z = b_0 + b_1Z_1 + b_2Z_2 + b_3Z_3 + b_{12}Z_1Z_2 + b_{13}Z_1Z_3 + b_{23}Z_2Z_3 + b_{123}Z_1Z_2Z_3$ (13)

These b_0, b_1, b_2, b_3, b_{12}, b_{13}, b_{23} ve b_{123} are new values taken as a result of conversion of the formula and values in (12).

Dissolution operation takes place according to the following equation :

$Na_2O\ 2\ CaO\ 5B_2O_3.16H_2O + 6HCl \longrightarrow 2NaCl_{(aq)} + 2CaCl_{(aq)} + 10H_3BO_{3\ (aq)} + 4H_2O$

The ulexite used in the study conducted has been supplied from the region of Balıkesir-Bigadiç in Turkey. Boron ore has been segregated into the requested fractions by being sifted through in ASTM class after it is cleaned from the apparent contamination. The chemical analysis of ulexite ore has been given in Table 2. The hydrocloric acid used in this study is 37 % by weight, and it has been supplied from merck. In Tablo 3, the parameters used in the experiment has been shown.

Dissolution experiments have been held in 250 mL cylindrical glass batch reactor by usingmechanical mixer. Temperature has been regulated by using a 0.1 ^oC sensitivity thermostate. A condenser has been used to prevent the solvent loss originating from evaporation. Initially, 100 mL hydrocloric acid has been added to the reactor, and the requested temperature has been watched over by running the mechanical mixer. Then dissolution operation has been embarked on by adding ulexite. When the time is over, the experiment has been halted, and the substance taken from the reactor has been filtered. Henceforwards, B_2O_3, D-mannitol and volumetrik in the substance have been analysed (Scott, 1963). The transformation fraction of the solute B_2O_3 has been retained by being divided into the B_2O_3 ratio in original ore.

2.2. Dissolution Studies

Table 2. The chemical analysis of the ulexite used in the study.

Component	% Composition
CaO	13.68
B_2O_3	42.24
H_2O	34.18
Na_2O	6.12
MgO	3.15
Others	0.63

Table 3. The parameters used in the experiment.

Variable	Range
Concentration (mol/L)	0.5 – 2.0
Solid/liquid ratio (g/mL)	0.02 – 0.1
Tempereture (K)	293 – 333
Particle size (mm) fixed	1.205
Stirring speed (rpm) fixed	400
Reaction time (min) fixed	5

3. RESULTS AND DICUSSION

The mathematical model of

$$Na_2O\cdot 2CaO\cdot 5B_2O_3\cdot 16H_2O+6H_3O^{+} \longrightarrow 2Na^{++}_{(aq)} + 2Ca^{+2}_{(aq)} + 10H_3BO_3 + 10\ H_2O \quad (14)$$

process has been formed by using full factorial design method with the study conducted.

The mathematical model to be formed of the process will be as follows

$Y_z = b_0 + b_1Z_1 + b_2Z_2 + b_3Z_3 + b_{12}Z_1Z_2 + b_{13}Z_1Z_3 + b_{23}Z_2Z_3 + b_{123}Z_1Z_2Z_3$.

Here:

Z_1 – Hydrocloric acid concentration is denominated with mol/L ;

Z_2 – temperature is denominated with ^{0}K ;

Z_3 – solid/liquid ratio is denominated with g/mL .

reaction time has been stabilized as 5 minutes in all the experiments.

The range of Z_1, Z_2 and Z_3 parameters are as follows :

$$\left.\begin{array}{l} 0.5 \le Z_1 \le 2 \\ 20 \le Z_2 \le 60 \\ 0.02 \le Z_3 \le 0.1 \end{array}\right\} \quad (15)$$

In accordance with the values given in Table 1, full factorial design long shot and the used values of parameters have been displayed in Table 4.

$$a_j = \frac{1}{N}\sum_{i=1}^{N} X_{ji}Y_i$$

using the formula above, the values of mathematical model to be formed in dimensionless coordinate system have been found as they are shown in (16) system.

$$\left.\begin{array}{ll} a_0 = 3.063915 & a_{12} = 0.362485 \\ a_1 = 0.16404 & a_{13} = 0.206658 \\ a_2 = 0.561375 & a_{23} = 0.490343 \\ a_3 = 0.714313 & a_{123} = 0.376693 \end{array}\right\} \quad (16)$$

Thus, the following mathematical model has been formed in dimensionless corrdinate system:

$$Y_x = 3.063915 + 0.16404\ X_1 + 0.561375\ X_2 + 1.714313X_3 + 0.362485X_1X_2 + + 0.206658X_1X_3 + 0.490343X_2X_3 + 0.376693X_1X_2X_3 \quad (17)$$

To assess the values of (17) model with Student criteria, 3 collateral experiments have been tested at the points of (Z_1^0 , Z_2^0 , Z_3^0) which is the centre of full factorial design plan. The centre of our experimental design is the point consisting of (1.25; 40; 0.06) coordinates. At this central point, the results of 3 collateral experiments held have been found as follows :

$$Y_1^0 = 4{,}0949;\ Y_2^0 = 4{,}1181;\ Y_3^0 = 4{,}1412 \quad (18)$$

Hence

$$\overline{Y}^{o} = \frac{Y_1^0 + Y_2^0 + Y_3^0}{3} = 4.118067 \quad (19)$$

$S_1^2 = \dfrac{\sum_{u}^{3}(Y_u^0 - \overline{Y})^2}{3}$ by using the formula, the result is found as $S_1^2 = 0.00053515$.

$S_{a_m} = \dfrac{S_1^2}{\sqrt{8}}$ from the formula, the result is found as

$$S_{a_m} = \frac{S_1^2}{\sqrt{8}} = \frac{0.00053515}{2.828} = 0.000189 .$$

As the found S_{a_m} is a rather low number, all the values of the t_m numbers found by (9) formula are larger than $t_p(f)$=4,3 number. And this shows that all the found coefficient values a_0, a_1, a_2, a_3, a_{12}, a_{13}, a_{23} and a_{123} are useable. Thus,

$$Y_x = 3.063915 + 0.16404\ X_1 + 0.561375\ X_2 + 1.714313X_3 + 0.362485X_1X_2 + + 0.206658X_1X_3 + 0.490343X_2X_3 + 0.376693\ X_1X_2X_3 \quad (17)$$

All the coefficient values of the model have been accepted as useable. For instance, the value of a_0 coefficient can be calculated.

$$t_0 = \frac{|a_0|}{S_{a_m}} = \frac{3.063915}{0.000189} = 15959.81 \rangle\rangle 4.3$$

(useable).

Whether the other coefficient values are useable or not has been determined by this method. Henceforwards, the validity of (17) mathematical model has been checked by Fisher criteria, and by (10) and (11) formulas.

The mathematical model of the process converter in natural coordinate system, it has been benefited from (12) formula as follows :

$$\left.\begin{array}{l} X_1 = \dfrac{Z_1 - Z_1^0}{\Delta Z_1} = \dfrac{Z_1 - 1.25}{0.75} = \dfrac{4Z_1 - 5}{3} \\ X_2 = \dfrac{Z_2 - Z_2^0}{\Delta Z_2} = \dfrac{Z_2 - 40}{20} \\ X_3 = \dfrac{Z_3 - Z_3^0}{\Delta Z_3} = \dfrac{Z_3 - 0.06}{0.04} = \dfrac{50Z_3 - 3}{2} \end{array}\right\} \quad (20)$$

After the variable value in (20) formula, the following mathematical model has been formed in natural coordinate system by using (17) model and making certain mathematical simplifications :

$$Y_z = 0.408787833 + 0.345559333Z_1 + 0.008172567Z_2 + 41.12100833Z_3 - 0.013503633Z_1Z_2 - 18.22426667Z_1Z_3 - 0.171848333Z_2Z_3 + 0.627821667Z_1Z_2Z_3 \quad (21)$$

(21) the calculated Y_{Zi}^{model} values by using (21) model have been added to the last column of Tablo 4.

The mathematical model values and relevance of the tested experiment results which have been formed in dimensionless coordinate system (17), and in natural coordinate system (21) has been given in Figure 1.

As can be seen in the Figure 1, the values of the derived basic materials in both dimensionless coordinate system and in natural coordinate system are rather close to each other. Therefore, the process can be researched through in larger parameters by using the formed mathematical model.

Table 4. Full factorial design plan and resentful values of parameters.

Parameters in dimensionless coordinate system											Natural coordinate system			
Experiment number	X_0	X_1	X_2	X_3	X_1X_2	X_1X_3	X_2X_3	$X_1X_2X_3$	Y^{exp}	Y_x^{model}	Z_1	Z_2	Z_3	Y_z^{model}
1	1	-1	-1	-1	1	1	1	-1	1.307	1.30698	0.5	20	0.02	1.306985
2	1	1	-1	-1	-1	-1	1	1	1.251	1.25016	2	20	0.02	1.25018002
3	1	-1	1	-1	-1	1	-1	1	1.478	1.47746	0.5	60	0.02	1.47746503
4	1	1	1	-1	1	-1	-1	-1	1.364	1.36381	2	60	0.02	1.36382806
5	1	-1	-1	1	1	-1	-1	1	4.095	4.09499	0.5	20	0.1	4.09499501
6	1	1	-1	1	-1	1	-1	-1	3.358	3.35803	2	20	0.1	3.35805002
7	1	-1	1	1	-1	-1	1	-1	4.720	4.72007	0.5	60	0.1	4.72007503
8	1	1	1	1	1	1	1	1	6.940	6.939822	2	60	0.1	6.93984206

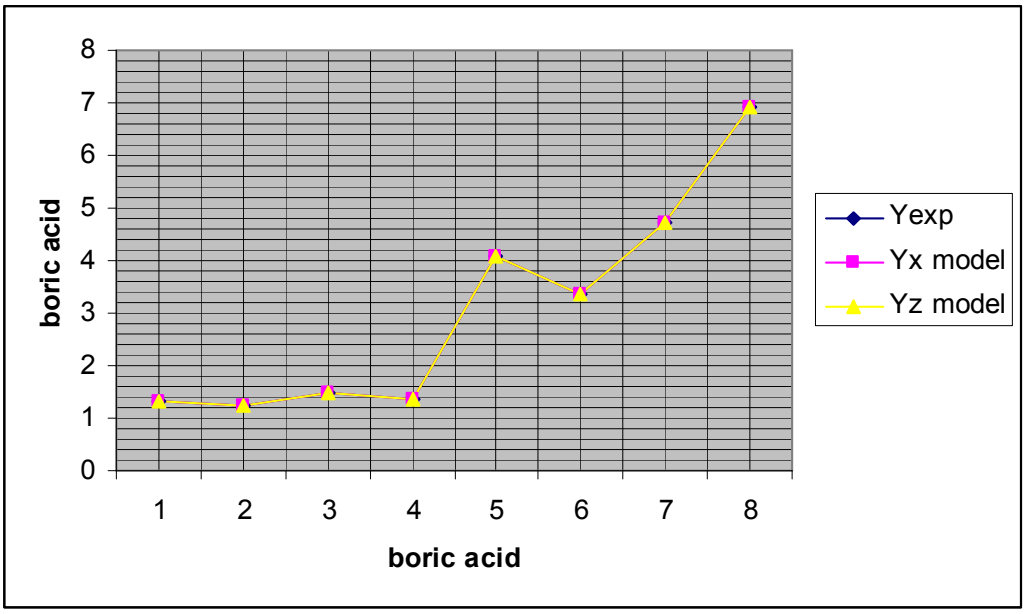

Figure 1. Graphical display of the validity of the process: Y_{exp} – values received experientially; $Y_{x\ model}$- calculated values in dimensionless coordinate system by mathematical model; $Y_{z\ model}$- calculated values in natural coordinate system by mathematical model.

CONCLUSIONS

Most of the field of sciences processes is important to identify the optimum conditions. This situation is related to both cost and time. Optimum conditions of a system using the optimum experimental parameters conditions. The formation of the mathematical model of the process by retaining the optimum accrual conditions of the chemical processes that occur depending upon a lot of variables enables this process to be checked automatically. In this study, reaction time as 5 minutes, stirring speed as 400 revolution/minutes, and particle size as 1.205mm has been stabilized during accruing of the chemical process.

With full factorial desing method, in dimensionless coordinate system

$Y_x = 3.063915 + 0.16404\ X_1 + 0.561375\ X_2 + 1.714313X_3 + 0.362485X_1X_2 + 0.206658X_1X_3 + 0.490343X_2X_3 + 0.376693\ X_1X_2X_3$ model has been formed.

The model formed in natural coordinate system ;

occurs as

$Y_z = 0.408787833 + 0.345559333Z_1 + 0.008172567Z_2 + 41.12100833Z_3 - 0.013503633Z_1Z_2 - 18.22426667Z_1Z_3 - 0.171848333Z_2Z_3 + 0.627821667Z_1Z_2Z_3$.

The acquired product amount in consequence of both models show relevance to experimental results. This demonstrates the validity of the formed mathematical model. The process can be brought under control as expected by using this mathematical model.

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