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Original Research

Modelling of the flotation process by central composite design for obtaining superior grade feldspar

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A B S T R A C T

Feldspar is usually found in conjunction with titanium and iron minerals. Feldspar should be separated from other minerals by generally magnetic separation or/and flotation with high grade. However, due to the similarities in the properties, feldspars are difficult to separate from gangue minerals by flotation.

In this study, the degradability of iron was investigated by mica flotation, and Central Composite Design (CCD) was used to plan and analyze the results. During the study, the effect of pulp density, pH, collector amount and air flow rate on the iron grade and the recovery of the obtained feldspar concentrate were modelled. Then, estimation of the optimum conditions and verification tests were performed. The iron content was reduced to 0.14% by mica flotation. However, this reduction amount was found to be insufficient. Finally, iron content was reduced to 0.05%. Considering that the maximum iron content of the superior grade feldspar is 0.1%, it can be said that two stage flotation is successful without different processes such as magnetic separation.

Keywords: *Feldspar flotation, superior grade feldspar, central composite design, modelling of the mica flotation.*

Introduction

The feldspar which constitutes approximately 60-65% of the earth's crust has a wide range of use, mainly in the ceramic and glass industries. Turkey has 14% of the world feldspar reserves and as of 2016, the 5.5 million tons of feldspar export has been an important part of the country's economy with an income of 155.5 million dollars (Sahiner, 2017). The usability and market price of feldspar are strongly related to presence of the impurities such as rutile and ilmenite which contain the colorant Fe_2O_3 and TiO_2. In the world, the most common methods employed in the enrichment of feldspar are magnetic separation and flotation. In Turkey, feldspar is generally enriched by flotation method in industrial scale.

Celik et al. (Celik et al, 2001) tried to remove the colorant impurities by a combination of magnetic separation and flotation methods. In this study, magnetic separation was used both before and after flotation. It was found that magnetic separation after flotation produced superior grade feldspar concentrate. In the study of Seyrankaya in 2003 (Seyrankaya, 2003), obtaining albit concentrate which is suitable for ceramic and glass industries was investigated by using two-stage flotation method from Albit ore in Muğla-Milas region. In a study published in 2006 by Orhan and Bayraktar (Orhan and Bayraktar, 2006), for the flotation of mica and metal-oxides from Milas Cine feldspars, they examined the interaction of the amines that remained after the first stage mica flotation with the sodium oleate used in the second stage flotation. According to the results obtained, with dewatering and washing applied after the first stage, the yield was increased to 94.58% from 86.67% in case of no washing. Heyes et al. (Heyes et al, 2012) published a review about removing minerals such as quartz, mica, ilmenite, rutile and magnetite from feldspar by flotation. The feldspar flotation in this article was explained as three stages flotation; in first stage, micas were floated with amines, in the second stage, titanium and iron-oxide minerals were floated with anionic collectors, and in the last stage the feldspar that activated by fluorite ions were floated with amines and leaving quartz in tailing. In a study published in 2016, Larsen and Kleiv (Larsen and Kleiv, 2016) indicated that quartz could be separated from feldspar by flotation with highly selective and high yields with dilute HF. In another study of the same researchers (Larsen and Kleiv, 2017), they stated that quartz yield depends on the conditioning time with HF, the type of frother and the concentration

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of HF and frother. In the study published by Jia Tian et al. (Tian et al, 2017), it was determined that the mixture of dioxylammonium chloride as a cationic collector and sodium oleate as an anionic collector showed high selectivity in the separation of spodumene from feldspar. Surface tension measurements, adsorption measurements, zeta potential measurements and FTIR analysis were carried out to determine the adsorption mechanism. In 2018, the same research team (Wu et al, 2018) published a study about spodumene-feldspar flotation with the use of the cationic reactant with the anionic collector. Due to the attachment mechanism, feldspar flotation was reported to be improved as the particle size decreased by Longhua Xu et al. (Xu et al, 2017). Again, in 2018, the same researchers published another study comparing the different flow diagrams for the spodumene flotation performance and recycling of mica and feldspar from the lithium wastes (Tian et al, 2018).

In a study published by Gulgonul and Celik in 2018 (Gulgonul and Celik, 2018), the selective flotation of Na-K (albite-microcline) feldspar flotation in the presence of K^+ ions as the potential-determining ion was investigated. In Brazil as one of the most important granite slab producers, quartz and iron removal from granite wastes studies were done (Silva et al, 2019). In this study, iron was reduced from 3.2% to 0.48% by magnetic separation. In another study published in 2018 (Sulaymonova et al, 2018), magnetic separation as a physical method, chemical enrichment using $HCl+H_2SiF_6$ mixture reagents and flotation with a specially designed flotation cell were used for quartz separation. In a published review, it was stated that crystalline structure, monovalent salts, flotation reagents and particle size distribution are important factors in the process. Also, mixed collectors are given as a promising method for feldspar flotation (Zhang et al, 2018).

Statistical design methods which allow effective analysis of effective parameters with less experimentation have been used for many years. For example, in a study published by Aytar et al. (Aytar et al, 2014), the Taguchi methodology, a statistical design method, was used in the optimization of the process in the fungal desulphurization study. The usage of these methods especially in academic studies has been increased with the use of response surface methods (RSM) that define effective parameters better and enable the process to be mathematically modelled (Aksoy and Sagol, 2016; Oluklulu and Koca, 2018). As an example, the study of Koca et al. (Koca et al, 2017) can be given. In this study, Box Behnken Design (BBD) which is a RSM method was used to investigate microbial treatment and flotation of lignites. Another common RSM method in the literature is Central Composite Design (CCD). When these two response surface methods are compared, BBD method has a significant disadvantageous compared to CCD. The main disadvantage of BBD method is that it requires less experiments at low (-1) and high (+1) levels. Therefore, it generally decreases the success of the predictions of these regions during the optimization stage (Croarkin and Tobias, 2015). In the literature, CCD has been used in experimental studies on many different topics including modelling and optimization in the literature, such as the calibration criteria optimization of gas flowmeters (Guerra et al, 2018), optimization and the desirability function for sorption of methylene blue from aqueous solution (Sadhukhan et al, 2016) etc. As in all experimental studies, CCD has been utilized in mining and mineral processing as well, such as coal preparation (Aksoy and Sagol, 2016; Aslan, 2007), pyrolysis (Onay and Koca, 2019), gold-silver recovery in cyanidation process (Karimi et al, 2010), copper leaching from refractory flotation tailings (Bai et al, 2018), optimizing of sphalarite flotation (Mehrabani et al, 2010).

In this study, the cleanability of feldspar in Aydın Söke region by mica flotation was investigated. The studied sample contains 0.24% Fe. In the case of superior grade feldspar, it is required by the ceramic factories that the iron content should be below 0.1% Fe. The aim of this study was originally to reduce the iron content with mica flotation and statistical experimental design was used to plan flotation experiments and to analyze the results. However, according to results of mica flotation, the iron content was not found to be sufficient. Therefore, a series of iron oxide flotations was needed to reduce the iron content to less than 0.1% in order to obtain superior grade feldspar.

1. Materials and Method

1.1. Materials

Feldspar samples were taken from Aydin Soke region. The feldspar ore found in this region is also used extensively in the ceramic industry established in that region. The sale price of these Feldspars is inversely proportioned to the iron content of the ore. Therefore, it is aimed to reduce Fe content to less than 0.1% with the highest feldspar yield. Microscope studies showed that the mica and other impurities contained in the ore were in the liberated state at -0.1 mm. Chemical analyzes of the samples by XRF are given in Table 1. Due to the low rate of impurities in the ore, it was not possible to determine the minerals contained as impurity in the feeding sample. Therefore, for the determination of unwanted minerals, XRD and SEM-EDS analyzes were carried out on the floating products obtained from reverse flotations. One of the results of SEM-EDS is given in Figure 1 as an example. All analyses, XRF, XRD and SEM-EDS, were repeated three times and mean results were presented in the manuscript.

Table 1. *Chemical analysis of feldspar sample (XRF analysis).*

Content	%	Content	%	Content	%	Content	%
Al_2O_3	17.70	Cu	<0.002	Ni	0.002	TiO2	0.29
As	<0.002	Fe	0.24	P	0.148	V	<0.002
Ba	0.051	K_2O	3.36	Pb	0.016	Zn	0.002
CaO	0.58	MgO	0.16	S	0.003	Zr	0.020
Co	0.002	MnO	<0.01	SiO_2	70.00	LOI*	0.4
Cr	<0.001	Na_2O	7.43	Sr	0.013		

*Lost of ignition

According to XRD and SEM-EDS analysis, both sodium (albite) and calcium (anorthite) structure ($NaAlSi_3O_8$-$CaAl_2Si_2O_8$ (triclinic)) were determined in the ore. In addition, alkali feldspar (XAl(1-2)Si(3-2)O(8) X: Na, K or Ca) were observed, and quartz was also found as silicate mineral. In the analysis of the floated product of mica flotation, muscovite was determined as a mica mineral and also kaolin minerals were detected. In the floating product obtained from iron flotation, chlorite mineral was found as an iron-containing mineral. Again, the presence of apatite was found in the same product.

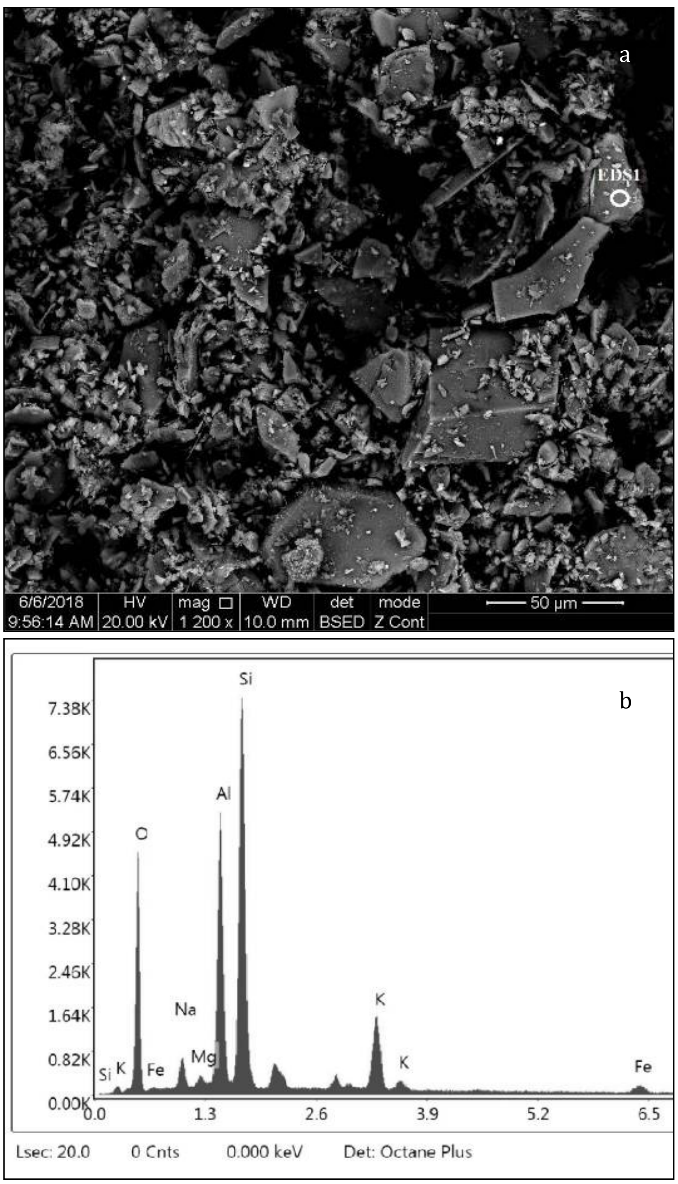

Figure 1. *(a) SEM-(b) EDS analysis example.*

1.2. Method

Mica flotation was originally selected to reduce the iron content and CCD method was chosen to plan flotation experiments and to analyze the results. However, according to initial results of mica flotation, the iron content was found to be insufficient. Therefore, a series of iron oxide flotations was needed to reduce the iron content to less than 0.1% in order to obtain superior grade feldspar. Therefore, the method used in this study can be called as two stage reverse flotation: mica flotation as stage I and iron-oxide flotation as stage II. Lab-scale Denver flotation machine was used for all flotation experiments. The test results were evaluated according to the iron content and weight yield of feldspar concentrates taken as sinking products by means of XRF analysis.

Before starting systematic studies, different collectors used in mica flotation in the literature were investigated with pre-trials for mica flotation and Procol CK 21 was found as the most effective collector among others. In stage I, Procol CK 21 which is an amine as a cationic collector and is produced by Ciba Specialty Chemicals was used as collector. The use of frother is not needed because of frothing property of chosen collector. The first conditioning was made in 55% solids ratio, the temperature was kept constant at 20^0C and the flotation time was 120 sec. In experiments; the effect of pulp density, pH, collector amount and air flow rate on the iron content and weight ratio of the concentrate (Recovery; %) were investigated. To determine the effect of each parameter, experiments were designed according to the Central Composite Design (CCD) which is one of the methods of response-surface methodology. These parameters were studied at 3 different levels and the α coefficient used to determine axial points in CCD was chosen as 2. In the software used, α value is suggested as 2 for 4 parameters. In various studies the α value is also suggested as 2 (Onay and Koca, 2019; Wang et al, 2016; Gungor et al, 2019) therefore the α coefficient was chosen as 2. The parameters and their levels are given in Table 2.

Table 2. *Selected parameters and their levels for Stage I.*

Parameters	Unit	Levels				
		-α	-1	0	1	+α
A (Pulp Density)	%	20	25	30	35	40
B (pH)		2.0	2.5	3.0	3.5	4.0
C (Collector Amount)	g/t	10	30	50	70	90
D (Air Flow Rate)	l/min	5.5	6.5	7.5	8.5	9.5

Number of experiments with α axial point at CCD was calculated as 29 by using following equation:

$$N = 2^k + 2\,k + n_c \tag{1}$$

Where N: number of experiments; k: number of factors, n_c: gives the number of midpoint experiments. After obtaining the test results, the results were subjected to variance analysis. Analysis of variance was performed according to 95% confidence interval. The equations proposed in the related module of the software used are based on the creation of reduced ANOVA tables and models. After the completion of the statistical analyzes, R^2 value indicating the compatibility of the obtained model equations with experimental data and PR^2 value indicating the prediction power of the model were examined. The optimum conditions optimizing iron content and the iron content and the yield of the concentrate taken under these optimum conditions were estimated. Experimental verification of the optimization study in the Stage I is also included. Design Expert software 10.01 was used for experimental design and the analysis of results.

Since Fe content remained at 0.14%, different collectors / collector mixtures recommended for iron-oxide flotation in the literature were also tested with preliminary studies. In the Stage II, iron oxide flotation was performed as reverse flotation to reduce iron content. 1:2:1 mixture of the A801+A825+A840 reagents which are petroleum sulphonates as an anionic collector produced by Cytec was selected as a collector. In the experiments, AF65 (produced by Clariant) was used as frother agent. At this stage, the amount of this collector mixture was studied and the initial conditioning was performed at 55% pulp density. Pulp density, pH and airflow were kept constant at 30%, 3 and 8 l/min, respectively. The amount of collector was studied for 200-300-400 g/t as total amount.

2. Results and Discussion

As mentioned earlier, products from experimental studies were subjected to XRF analysis. Experimental studies were evaluated for the iron content and yield of feldspar concentrate taken as sinking product. The results are given separately for each stage.

2.1. Stage I

The values of the response variables calculated according to the results of chemical analysis of the products taken in the Stage I are given in Table 3. In this Table, solid rate (%), pH, the collector amount (g/t) and the air flow rate (l/min) are shown as A, B, C, D, respectively, and parameter levels are given as coded values.

Table 3. *The results of Stage I.*

No	A	B	C	D	Fe (%)	Rec. (%)	No	A	B	C	D	Fe (%)	Rec. (%)
1	-1	-1	-1	-1	0.20	93.46	16	+1	+1	+1	+1	0.13	84.61
2	+1	-1	-1	-1	0.20	94.78	17	-α	0	0	0	0.19	93.82
3	-1	+1	-1	-1	0.22	96.07	18	+α	0	0	0	0.16	89.21
4	+1	+1	-1	-1	0.21	94.65	19	0	-α	0	0	0.15	90.61
5	-1	-1	+1	-1	0.16	88.89	20	0	+α	0	0	0.18	89.02
6	+1	-1	+1	-1	0.15	90.04	21	0	0	-α	0	0.23	96.55
7	-1	+1	+1	-1	0.16	88.52	22	0	0	+α	0	0.12	81.68
8	+1	+1	+1	-1	0.15	86.88	23	0	0	0	-α	0.17	89.50
9	-1	-1	-1	+1	0.21	95.44	24	0	0	0	+α	0.16	88.28
10	+1	-1	-1	+1	0.20	93.3	25	0	0	0	0	0.19	94.89
11	-1	+1	-1	+1	0.22	96.59	26	0	0	0	0	0.17	90.12
12	+1	+1	-1	+1	0.20	94.11	27	0	0	0	0	0.17	91.44
13	-1	-1	+1	+1	0.15	89.17	28	0	0	0	0	0.16	91.26
14	+1	-1	+1	+1	0.13	86.10	29	0	0	0	0	0.17	88.81
15	-1	+1	+1	+1	0.15	85.86							

The results were subjected to the analysis of variance for both selected response variables. Subsequently, the reduced ANOVA charts and final models were generated by subtracting the terms that were statistically insignificant in the 95% confidence interval. In this study, the results are given as reduced ANOVA charts. R^2 and PR^2 values are also given. Besides, the results obtained are graphically interpreted for all parameters, and the graphs of interaction terms, if any, are presented in both two and three dimensions. In all graphs, parameters other than the parameter whose effect is examined are kept at medium level.

2.1.1. Iron content of the concentrate of Stage I

The results of iron content obtained in the experiments were subjected to variance analysis. In the related module of the software, linear model is proposed. However, when a Quadratic model was created and analyzed, it was determined that there was a low interaction between the collector amount (C) and the airflow (D). Then, an ANOVA chart was created by adding the CD interaction term to the linear model. Summarized ANOVA chart is given in Table 4.

Recommended model is statistically significant, with p value less than 0.05 (less than 0.0001); and the error term is also greater than 0.05 (0.8898). The R^2 and PR^2 values of the model are 0.9362 and 0.9044, respectively. Figure 2 (a) is a graph showing the relationship between the experimental results and the estimated results. As it can be seen from the figure, the predicted iron content values from the model represent approximately 94% of the experimental results. This shows the power of the model. The main effect graphs of the parameters examined in the process are given in Figure 2 (b) for all parameters. The model equations for coded values and actual values are given in Equation 2 and Equation 3, respectively.

$$Fe = 0.17 - 6.66*10^{-3} A + 4.16 * 10^{-3} B - 0.029 C - 3.33 * 10^{-3} D - 3.75*10^{-3} CD \quad (2)$$

$$Fe = 0.22 - 1.33*10^{-3} A + 8.33 * 10^{-3} B - 5.21*10^{-5} C - 6.04 * 10^{-3} D - 1.88*10^{-4} CD \quad (3)$$

As it can be seen from the graph, the effect of A (Solid ratio), B (pH) and C (Collector amount) is significant at 95% confidence interval, while the effect of D (Air rate) is not statistically significant. When the P values in both the graph and ANOVA charts are examined, it is seen that the amount of collector is the most effective parameter. The graph shows that no parameter has a parabolic effect. In addition, when the ANOVA chart was examined, it was seen that the amount of collector interacted slightly with airflow (term CD). The interaction is graphically shown in Figure 3 in two dimensions (a) and in three dimensions (b).

As seen in Figure 3, the increase in the amount of collector causes a decrease in the iron content of the concentrate. However, this reduction occurs more slowly when the airflow decreases. This shows the interaction between these two parameters.

Table 4. *ANOVA chart and summary for the iron content of concentrate in Stage I.*

Source	P Values
Model	<0.0001
A	0.0005
B	0.0197
C	<0.0001
D	0.0569
CD	0.0784
Lack of fit	0.8898
R2	0.9362
PR2	0.9044

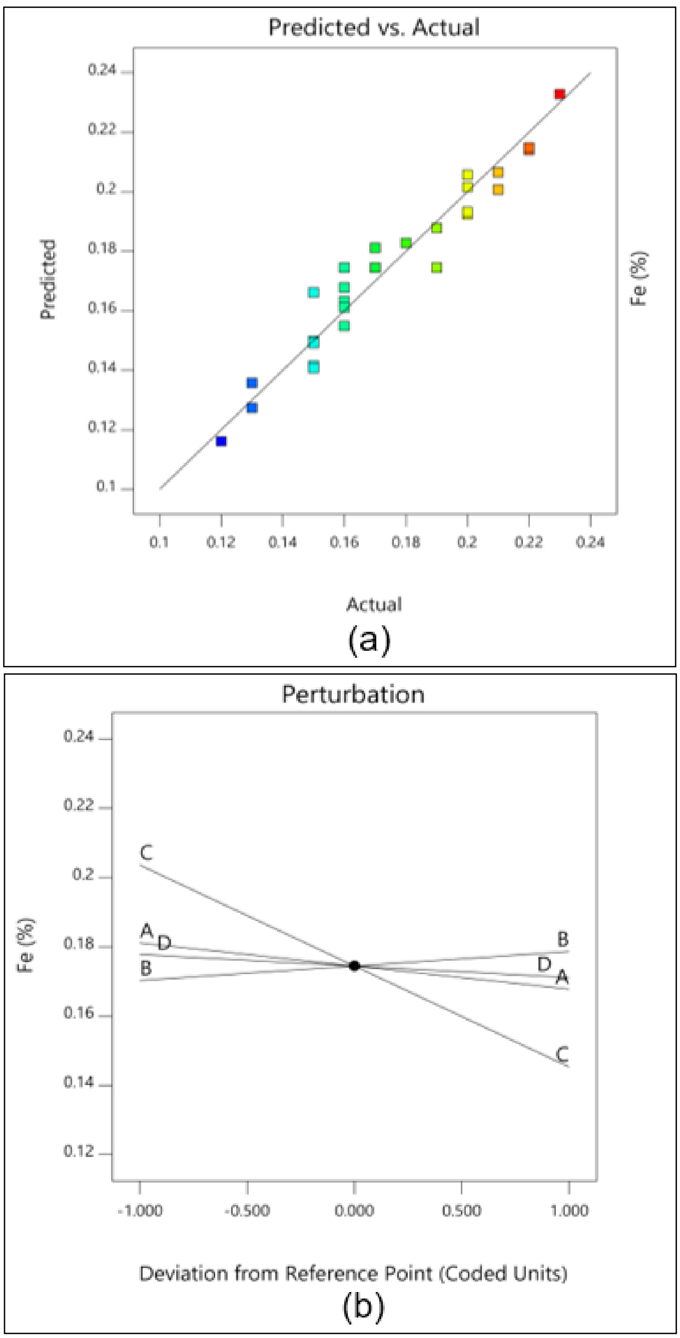

Figure 2. a) *Graph of experimental results versus predicted values for iron content of concentrate in Stage I;* ***b)*** *Main effect graphs of parameters on iron content of concentrate in Stage I.*

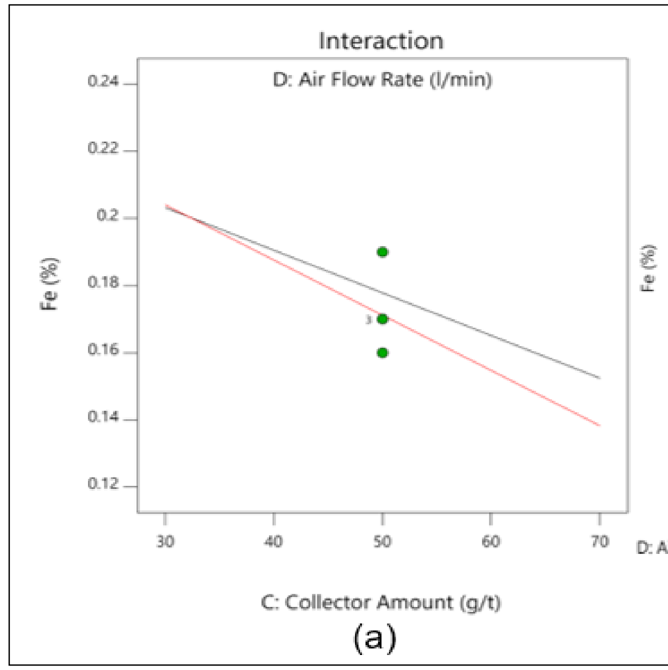

Figure 3. *Graph of the interaction between the collector amount and the airflow on the iron content of the concentrate in the Stage I; a) two-dimensional; b) three-dimensional*

2.1.2. The recovery of the concentrate of Stage I

The results of yield obtained in the experiments were also subjected to variance analysis. In the related module of the software, linear model is proposed. The ANOVA chart was created according to the linear model. Summarized ANOVA chart is given in Table 5.

Table 5. *ANOVA chart and summary for the yield of concentrate in Stage I.*

Source	P Values
Model	<0.0001
A	0.0304
B	0.3941
C	<0.0001
D	0.2077
Lack of fit	0.8983
R^2	0.8385
PR^2	0.7730

Recommended model is statistically significant with p value less than 0.05 (less than 0.0001); and the error term is also greater than 0.05 (0.8983). The R^2 and PR^2 values of the model are 0.8385 and 0.7730, respectively. Figure 4 (a) is a graph showing the relationship between the experimental results and the estimated results. As can be seen from the figure, the predicted yield values from the model represent approximately 84% of the experimental results. This shows the power of the model. The main effect graphs of the parameters examined in the process are given in Figure 4 (b) for all parameters.

The model equations for coded values and actual values are given in Equation 4 and Equation 5 respectively.

$$Recovery = 90.82 - 0.78\,A - 0.29\,B - 3.67\,C - 0.44\,D \quad (4)$$

$$Recovery = 109.74 - 0.16\,A - 0.59\,B - 0.18\,C - 0.44\,D \quad (5)$$

As it can be seen from the graph, the most effective parameter is the amount of collector (C). While the effect of solid ratio (A) was statistically significant at 95% confidence interval, the effect of pH (B) and airflow (D) parameters were not statistically significant. Similar to iron content analysis, no parabolic effect is observed in any parameter.

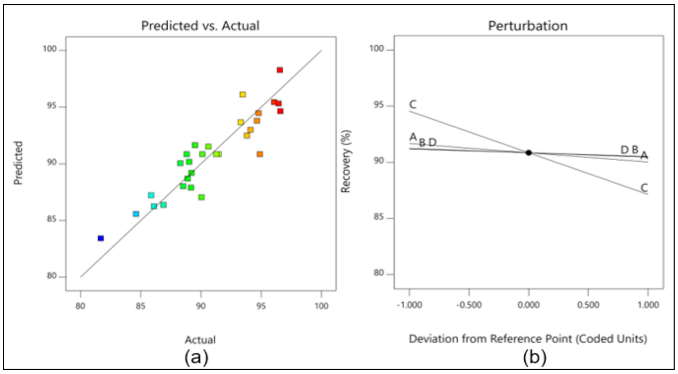

Figure4. *a) Graph of experimental results versus predicted values for yield of concentrate in Stage I, b) Main effect graphs of parameters on yield of concentrate in Stage I.*

2.1.3. The optimization of Stage I and verification results

With the help of the software, the estimation of the conditions that minimize the iron content of the sinking product (for coded values), the predicted values of the iron contents of the products taken under these conditions and the iron contents of the samples obtained from the experimental studies performed under the same conditions are given in Table 6.

The experimental data (0.14% Fe) corresponds to the estimated values in the 95% confidence interval (0.11% - 0.15% Fe). Therefore, it can be said that the obtained model is sufficient. In these conditions, the content of iron of floating product was found to be 0.97% Fe, and 88% of the feed was taken as feldspar concentrate. 51.42% of the iron in the feed was sunk. As a result, the values of all the response variables of the floating product and feldspar concentrate obtained from verification experiment in the optimum conditions matched up with the predicted value ranges calculated in the 95% confidence interval.

Table 6. *The results of verification tests of Stage I.*

Parameter	Value	Predicted value of Fe (%)			Experimental Value of Fe (%)
		Minimum	Average	Maximum	
A	+1	0.11	0.13	0.15	0.14
B	-1				
C	+1				
D	-1				

2.2. Stage II

In the Stage II, used sample was the sinking product of the first stage and with 0.14% Fe content. At this stage, the total amounts of collectors were determined as 200-300-400 g/t by preserving the 1:2:1 mixture ratio for A801+A825+A840. XRF results of these 3 experiments performed under these conditions are given in Table 7.

Table 7. *The effect of collector amount on iron content of feldspar concentrate in Stage II.*

Col. Amt. (g/t)	Recovery (%)	Iron Content (%)	Iron Dist. (%)
200	78.00	0.05	26.57
300	73.75	0.03	15.21
400	61.25	0.02	8.28

The increase in the amount of collector reduces the iron content of the concentrate, while the selectivity has also decreased. Therefore, the yield of feldspar taken as sinking product decreased rapidly. Considering that minus 0.1% Fe is the initial target, 200 g/t collector provides the target with 0.05 % Fe. As a result of this study, it can be stated that high quality feldspar which contains less than 0.1% Fe can be produced with the usage of a series collector, and the quality of feldspar can be increased significantly by increasing the amount of collector with a comprise on the feldspar yield.

On the other hand, the statistical design applied in the first stage and model equations of this stage were also acceptable according to the R2 and PR2 values of equations obtained. The model equation created especially for iron content is a model with a high predictive power with 0.9362 R^2 and 0.9044 PR^2 values. The result of the verification study carried out of this stage shows the power of model equations, too.

Conclusions

In this study, the possibility of reducing the iron ratio of feldspar taken from Aydın Soke region from 0.24% Fe to minus 0.1% Fe was investigated. The analysis shows that the ore consists mainly of plagioclase and alkaline feldspar and some quartz, and low amount of impurities such as muscovite, kaolin, iron containing chlorite and apatite minerals. In order to reduce the iron content, two stage reverse flotation was applied.

The mica flotation was applied in the stage I by using CCD methodology. XRF analysis was carried out on the products obtained from the experiments and the results were analyzed for the Fe content and the yield of concentrate.

The mathematical models of the process for these two response variables were examined. The correlation coefficient for the model of the iron content of the concentrate which is the first response variable was 93.62% and the PR^2 value of the model was 90.44%. These two values are over 90% and are very close to each other, and this situation shows that the model is very strong. According to the model and for the studied levels, the most effective parameter on the iron content was found to be the amount of collector. The models created for other response variable was also statistically significant. As a result, the iron content of feldspar was reduced from 0.24% to 0.14% by stage I.

In the stage II, the amount of the combination of A801+A825+A840 in the ratio of 1:2:1 determined as the most effective combination of collectors was studied. The used lowest amount of collector reduced the iron content in feldspar to 0.05% Fe, and this value coincided with the initial target as minus 0.1% Fe. This step was not optimized and it can be said that the iron content of the sinking product can be reduced to 0.02% Fe by increasing the amount of collector. However, increasing the amount of collector reduced the amount of sinking product by up to 60% by weight.

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