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The Modeling and Optimization of Titanium Dioxide Extraction, Case study: The Slag Sample of Blast Furnace

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ABSTRACT

In this research work, the application of the Response Surface Methodology (RSM) and the Central Composite Design (CCD) techniques for modeling and optimization of some of the operating variables on the titanium dioxide extraction were studied. This study was performed, using sodium hydroxide roasting and sulfuric acid leaching. Four main parameters, i.e., leaching temperature, time, liquid to solid ratio, and the concentration of acid, were changed during the experiments. The two parameters of the stirring rate (250 rpm), and the feed size (d_{80}= 106 micrometers) were considered to be constant. Based on the findings, several empirical equations were modeled for the titanium dioxide extraction with the above mentioned parameters. The empirical equations were then individually optimized by employing the quadratic programming to maximize the extraction within the experimental range. In conclusion, the optimum conditions were accordingly obtained at 85°C, 235 minutes, liquid to solid ratio of 15, and the acid concentration of 2.4 M, in which the maximum TiO_2 extraction of 81.32% was achieved.

Keywords: *Experimental design, Central composite design, Alkali roasting method, Metal Extraction*

1. Introduction

The steel industries, in addition to producing their main product, manufacture significant amounts of byproducts including spent catalysts, fly ashes, sludge, ore slag and ashes [1, 2]. Due to the storage and environmental problems of these products, companies have been recently forced to reuse the waste products[2]. Several projects have been implemented in this regard all over the world. Today, the iron slag is used in production of cement, construction of railways and railroads, manufacturing of glasses and artificial stones, considering as secondary source of vanadium and etc [2, 3].Titanium is quite abundant in the earth's crust (0.63%) [4], and due to the unique characteristics of titanium dioxide, it could be used in many important industries including the absorbent production, pigment, porcelain, paper, rubber, and plastics [5-7].

The sulfate and chloride methods are the most common methods of titanium dioxide production. Due to some problems associated with these methods, other technique shave been recently developed [5, 7-12]. A new method was suggested by some researchers to enhance titanium containing minerals or titanium slag. The method is involved with dissolution by alkali Na or K, and then washing with water. The product is then dissolved by acid and after several operations; the titanium dioxide pigment is finally produced [10, 13-16].

During the roasting process, various compounds in the slag are formed according to the reactions of (1) to (7) [10, 13]. Right after roasting, the silicon and aluminum as impurities are converted to water soluble compounds. Na_2TiO_3 and Na_2TiSiO_5 compounds are accordingly formed, and then subjected to further H_2SO_4 for acid dissolution [10, 17-23].

$$TiO_2 + NaOH \rightarrow Na_2TiO_3 \quad (1)$$

$$2TiO_2 + 2FeO + NaOH \rightarrow 2NaFeTiO_4 \quad (2)$$

$$TiO_2 + SiO_2 + NaOH \rightarrow Na_2TiSiO_5 \quad (3)$$

$$Al_2O_3 + NaOH \rightarrow 2NaAlO_2 \quad (4)$$

$$V_2O_4 + NaOH \rightarrow 2NaVO_3 \quad (5)$$

$$Cr_2O_3 + NaOH \rightarrow Na_2Cr_2O_4 \quad (6)$$

$$SiO_2 + NaOH \rightarrow Na_2SiO_3 \quad (7)$$

The titanium slag is roasted with $NaCO_3$, and is then subjected to sulfuric acid dissolution. After hydrolyses, the produced sulfate solution in presence of oxalic acid as a reducing agent and hydrolyzed precipitate is filtered, washed, and then calcined. As a result, a very highly pure TiO_2 (99.8%) is produced [10]. For the recovery of TiO_2, Al_2O_3 and pig iron of red mud, the pyro-hydrometallurgy process is employed, and 84.7% of TiO_2 was recovered [17]. It has been reported [19]that in order to upgrade the poor titanium slag, it was roasted by Na_2CO_3 at 500°C. The product was then washed to remove the water-soluble impurities and it was dissolved by HCl and the H_2TiO_3 to finally produce the TiO_2.

The usual way to determine the affecting parameters for TiO_2 leaching processes is to change one parameter and keep the others constant. The main disadvantage of this method is the lack of examination of interactive effects among the variables parameters [24, 25]. Thus, in the last few decades, the use of experimental design method has been expanded. In this technique, in addition to the determination of the interaction effects of parameters, a significant decrease in the number of the experiments is being achieved. Accordingly, the results are obtained in the shortest time and the whole operation will be cost-saving [24-26].

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The response surface method (RSM) is a combination of mathematical and statistical techniques that is useful for developing, improving and optimization of the processes. The central composite design (CCD) method has been known as an effective design method due to its ability to provide the accurate estimations from different forms of interactions that occurring among influencing parameters of a system [27-30].

Due to the large quantities of iron ore slag in Esfahan steel-making Company and also with regards to the increasing trend of TiO_2 production, the extraction of titanium dioxide from the iron ore slag has been studied. The purpose of this research work is to show the application of an experimental design technique called "the response surface method, using central composite design (CCD)"in modeling, optimization and evaluation of the effects and interactions of the operational parameters. This method was conducted on the process of recovering titanium dioxide from the Blast Furnace Slag sample, using roasting by the sodium hydroxide.

2. Materials

The iron slag sample was prepared from the mixer unit of Esfahan steel-making Company, located in Esfahan province, Iran.

3. The analytical procedures

The X-ray fluorescence (XRF) method was used to determine the chemical compositions of the iron ore slag. The mineralogy of sample was determined using a computer controlled D8-Advance, Bruker AXS, X-ray diffractometer machine (XRD), equipped with Cu K_α radiation (λ=1.540598Å) with a 2theta-theta goniometer geometry. An SEM Xl30, Philips scanning electron microscope equipped with WDX-3PC analysis unit was used to study the morphology and structure of sample. The concentrations of iron were determined by the dichromate titration method. The chromatography method was employed to determine the titanium dioxide content in the studied sample and the leaching solution. The titanium absorbance values were measured at 430 nm, using a Unicam UV2-100 spectrophotometer by means of tiron as the complex agent. Some pre-treatment operations including crushing, milling, and gravity separation were precisely conducted on the sample to make it ready for sodium hydroxide roasting.

The XRF results of prepared sample are shown in Table 1. The X-ray diffraction (XRD) analysis of the prepared sample (Figure 1) indicated that the main titanium containing phase was sodium titanium silicate (Na_2TiSiO_5). The XRD spectrum of slag samples are normally broad and difficult to interpret [30]. Due to the compact nature of studied slag sample, an anamorphous phase, probably silicate compound was obviously made it difficult to recognize the rest of crystalline phases from the XRD diffractogram. The SEM images of the raw and leached samples are shown in Figure 2 (a), and Figure 2 (b), respectively. The EDX analysis indicated that calcium, silicon, iron, and titanium had a uniform distributed in the sample.

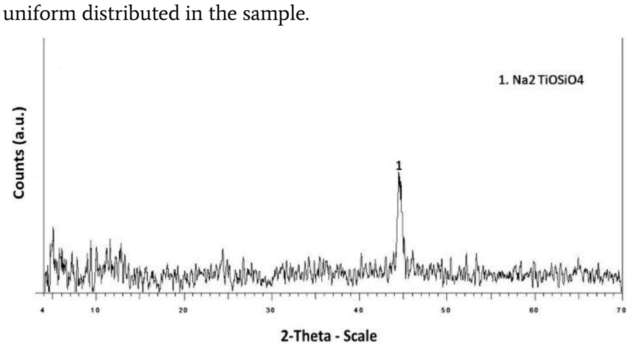

Figure 1. The XRD pattern of roasted iron ore slag.

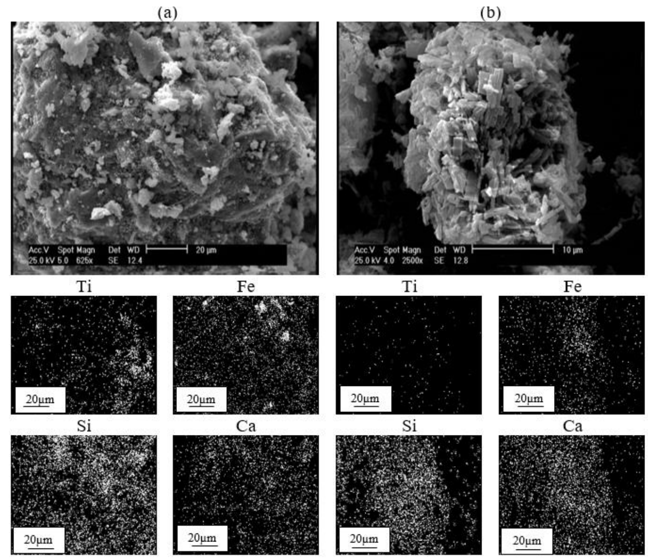

Figure 2. The SEM images and the EDX analysis of roasted iron ore slag before leaching (a), and (b) after leaching.

Table1. The chemical composition of the roasting iron ore slag (The results were obtained from the XRF test).

	SiO_2	CaO	Fe_2O_3	MnO	Al_2O_3	TiO_2	MgO	Na_2O
%	38.3	20.1	15.3	7.7	7.2	5.0	3.5	2.73

4. Design of Experiments (DOE)

The RSM method based on CCD was applied to investigate the effect of four variables on the titanium dioxide extraction from roasting slag with sodium hydroxide by means of Design Expert 10 (DX10) software. This statistical method evaluates the relationship between the response and independent variables for matching the model with empirical data[29].

The RSM was used to evaluate the effect of independent variables on the response performance (i.e., TiO_2 recovery), as well as the best response rate. The independent variables of this study were four loading ratios of leaching temperature, time, liquid to solid ratio, and the concentration of acid. The maximum value for each variable was chosen to be "+1", and the minimum level was set to be "-1", consequently, the mid value was considered as "0" (Table 2). The total number of experiments was chosen to be 30 points, which included 16 factorial points, 8 axial points (2 × 4) and 6 central points (Table 3).

Table 2. The independent variables and their levels for the central composite design (CCD).

		Coded variable level				
		Lowest	Low	center	high	highest
Variable	Symbol	-α	-1	0	+1	+α
Temperature (°C)	X_1	30	30	57.5	85	85
Time (min)	X_2	60	60	150	240	240
Liquid to Solid ratio (g/g)	X_3	5	5	10	15	15
Concentration (M)	X_4	1	1	2	3	3

In this series of experiments, the primary feed was obtained from roasting the iron ore slag with sodium hydroxide. The tests were performed inside a glass reactor on a magnetized hot plate. The two parameters of the stirring rate (250rpm) and the feed size (d_{80}= 106 micrometers) were considered to be constant. All pores and outlets of the reactor were carefully chosen to be watertight to prevent the release of gases and also to reduce the evaporation during the process, especially at high temperatures.

5. Results and discussion

According to the design matrix, 30 tests were conducted by running the sequence defined by DX10 software. The conditions of TiO_2 leaching experiments, their results and predicted response are shown in Table 3.

The obtained experimental results of TiO_2 extraction were modeled using a full quadratic equation by applying the multiple regression analysis. The TiO_2 extraction (Y) in the model (equation 8)was expressed as a function of leaching temperature (x_1), time (x_2), liquid to solid ratio (x_3), and the concentration of acid (x_4).

$$\ln(Y) = 3.74 + 0.14x_1 + 0.061x_2 + 0.35x_3 + 0.16x_4 + 0.054x_1x_3 + 0.072x_1x_4 - 0.21{x_3}^2 - 0.26{x_4}^2 \quad (8)$$

Table 3. The actual and predicted levels of variables with experimental result.

Run	Variable				Actual Value (%)	Predicted Value (%)
	Temperature (°C) (X_1)	Time (min) (X_2)	Liquid to Solid ratio (g/g) (X_3)	Concentration (M) (X_4)		
1	85	240	15	1	38.6	38.22
2	30	60	15	3	29.64	31.95
3	57.5	150	10	2	43.26	42.22
4	30	240	5	1	14.91	16.61
5	85	240	5	3	27.62	27.34
6	57.5	150	5	2	27.09	24.13
7	57.5	150	10	3	40.73	38.48
8	30	240	15	1	31.72	30.00
9	57.5	150	10	2	43.94	42.22
10	85	60	15	1	33.51	33.80
11	30	240	15	3	33.88	36.13
12	57.5	150	10	1	27.71	27.69
13	85	150	10	2	43.5	42.22
14	57.5	150	15	2	45.81	48.56
15	57.5	150	10	2	44.38	42.22
16	57.5	150	10	2	42.9	42.22
17	85	240	15	3	64.56	61.32
18	85	60	5	1	15.2	15.07
19	30	150	10	2	36.57	36.75
20	57.5	240	10	2	40.35	44.90
21	30	60	5	1	13.82	14.69
22	30	60	15	1	28.81	26.53
23	30	60	5	3	18.47	17.69
24	57.5	150	10	2	43.5	42.22
25	57.5	60	10	2	38.73	39.70
26	30	240	5	3	20.76	20.00
27	85	60	5	3	22.63	24.18
28	85	60	15	3	54.58	54.23
29	57.5	150	10	2	44.2	42.22
30	85	240	5	1	17.38	17.04

The F-test was employed to check the statistical significance of the proposed model. The analysis of variance (ANOVA) for the response surface quadratic model is shown in Table 4. The F value for this model was 117.79, and the P value was less than 0.001. The result indicated that the statistically significant results were achieved.

The liquid to solid ratio had the greatest effect on the titanium dioxide extraction, according to the Equation model (Eq. 8), and the ANOVA results. The acid concentration and leaching temperature were the next effective parameters, respectively. The effect of other parameters was lower than the expressed parameters (C> D> A> AD). The coefficient of determination (R^2 = 0.9829) and variance (C.V = 2.02) indicated that the proposed model was suitable and statistically significant for determining the optimal conditions. The satisfactory value of R^2 indicated that the quadratic equation was capable of representing the system under the given experimental domain. This confirmed from the plots of the predicted values versus observed values for TiO_2 extraction presented in Figure 3. The remainders had a normal distribution, because of the point's behavior to follow a straight line.

Table 4. The analysis of variance (ANOVA) of the response surface model to predict the TiO_2extraction.

ANOVA for the Response Surface Reduced Quadratic Model					
Source	Sum of Squares	Df	Mean Square	F Value	p-value Prob > F
Model	4.59	8	0.57	117.79	< 0.0001
A-T	0.35	1	0.35	71.03	< 0.0001
B-t	0.068	1	0.068	13.97	0.0012
C-L/S	2.20	1	2.20	452.22	< 0.0001
D-Co	0.49	1	0.49	100.23	< 0.0001
AC	0.047	1	0.047	9.62	0.0054
AD	0.082	1	0.082	16.92	0.0005
C^2	0.15	1	0.15	31.15	< 0.0001
D^2	0.23	1	0.23	46.84	< 0.0001
Residual	0.10	21	4.869E-003		
Lack of Fit	0.10	16	6.337E-003	36.73	0.0004
Pure Error	8.625E-004	5	1.725E-004		
Cor Total	4.69	29			

The signal-to-noise ratio (Adeq-Precision) was 37.389. Due to the fact that the amount of accuracy was more than 4, the proposed model can be used to predict the accurate design. The difference between Pred R-Squared and Adj R-Squared was less than 0.02. This criterion was another reason to prove the suitability of the proposed model. Although Lack of Fit is significant, but according to the above description, the model is acceptable and this can be ignored. The statistical parameters for the validation of the proposed model were presented in Table 5.

Table 5. The statistical parameters for the validation of the purposed model.

The statistical parameters							
Std. Dev.	Mean	C.V. %	PRESS	R-Squared	Adj R-Squared	Pred R-Squared	Adeq Precision
0.070	3.46	2.02	0.23	0.9782	0.9699	0.9520	37.389

The effect of main parameters (i.e., the leaching temperature, time, liquid to solid ratio, and the concentration of acid) are shown in Figure 4. In this plot, the slope or curvature of a gradient in one factor shows that the response can be sensitive to this variable. On the other hand, the straight line indicates the lack of sensitivity of the response to such variable.

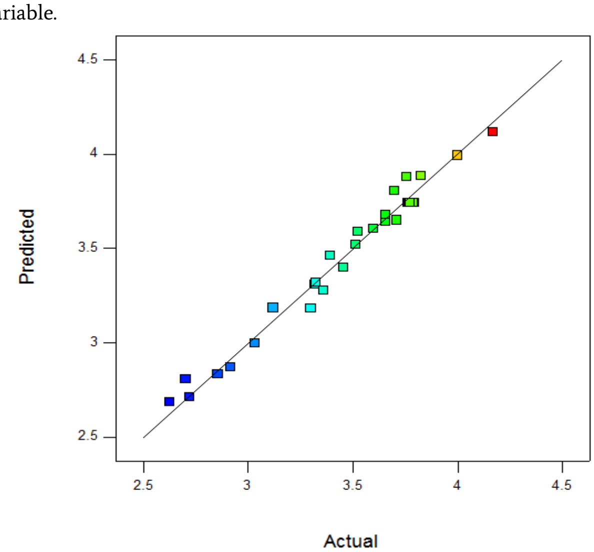

Figure 3. The plot of predicted versus observed values for TiO2 extraction.

In the extraction of titanium dioxide from roasting slag, the temperature gradient slope (A) indicated this parameter was effective in extraction, and increased the extraction rate by its growth (Figure 4). The gradient of the time graph (B) was low, indicating this parameter was effective in extraction, but its effect was less than the other studied parameters (Figure 4). The graph of the ratio of liquid to solid (C) was not linear, so that it could be divided into two sections of steep and low-slope parts (Figure 4). The first part was a steep curve which showed the high impact of the studied parameter in this limit. And the second part had a lower gradient curve which indicated that the effect of this parameter had been reduced. In fact, this curve showed that increasing the liquid-to-solid ratio from one point to the next did not have much effect on the TiO_2 extraction, and it can be inferred that with an increase in the ratio of liquid to solid, the extraction was constant. The acid concentration plot (D) was a curve. This curve was divided into two parts (Figure 4). At the beginning of the curve, there was a positive gradient indicating this parameter was effective in the TiO_2 extraction and also extraction rate increased by increasing the acid concentration. The second part of the curve was also steep (negative) emphasizing the effectiveness of the concentration of acid in the extraction of TiO_2. It also showed that an increase in acid content could reduce the titanium dioxide extraction.

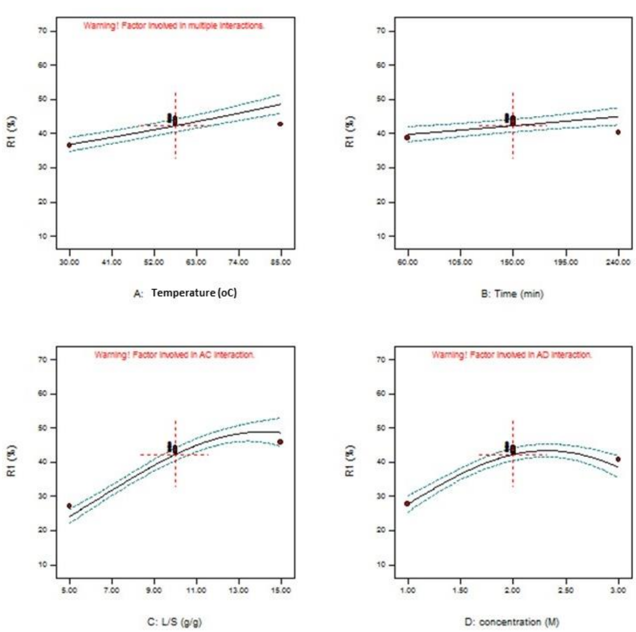

Figure 4. The effect of the main parameters (i.e., the leaching temperature, time, liquid to solid ratio, and the concentration of acid) on TiO_2 extraction.

In order to investigate the interactive effect of the studied parameters on the extraction of titanium dioxide, a 3-dimensional model of response surface and related logistic diagram was designed which is presented in Figure 5. Also, the control lines were useful in the type identification of interactions between the studied variables. The effect of the important variables and their interactions is shown in Figure 5.The interaction between the temperature and liquid to solid ratio in the extraction of titanium dioxide was studied too, at which time and concentration of acid was fixed at 150 minutes and 2 M, respectively (Figure 5.a). The extraction was considerably influenced by the interaction between temperature and the ratio of liquid to solid. By increasing these two parameters, the extraction will be increased. The time and liquid-to-solid ratio were maintained fixed. The interaction between temperature and concentration of acid on the extraction of titanium dioxide was investigated (Figure 5.b). Increasing the temperature and concentration would cause an increase in TiO_2 extraction. This process continued to the acid concentration about 2.5 M. Thus, about 50% of TiO_2 was extracted at 85°C and 2.5 molar of acid concentration.

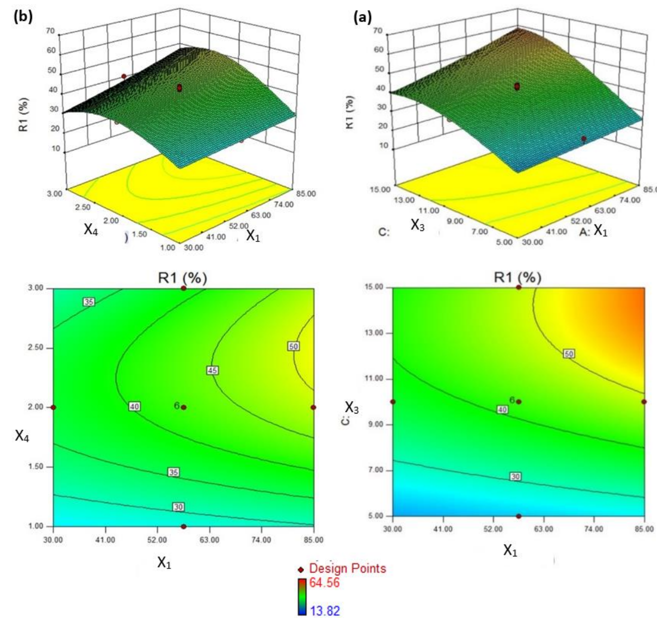

Figure 5. The contour plot and response surface plot showing the effect of the temperature and liquid to solid ratio (a), and the effect of the temperature and concentration (b) on the extraction of TiO2 when all other variables were kept at their center level.

Regarding the main aim of this research work which was to maximize the extraction of titanium dioxide, the optimum conditions were predicted using design of experiments method. The results obtained with numerical optimization were 65.93% retrieval, based on the model at 84.78°C, 233.16 minutes; liquid to solid ratio was14.85 and the concentration of 2.43 M (Figure 6). Experimental TiO_2 extraction was obtained 81.32%, at 85°C, 235 minutes, liquid to solid ratio 15, and the acid concentration of 2.4 M. Due to the small number of factors in the retrieval and the trend of the model on the whole data, the predicted and actual extraction values were different.

An arbitrary test was carried out to evaluate the model at a temperature of 50°C, 120 minutes, a liquid to solid ratio of 15, and an acid concentration of 2.4 M. The experimental TiO_2 extraction was 49.18%, and the predicted extraction was 45.92% indicating that the model was able to accurately predict the extraction of titanium dioxide.

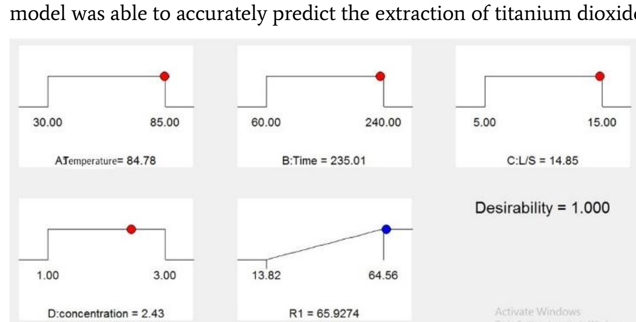

Figure 6. The proposed levels for the parameters to maximize the extraction of TiO2.

6. Conclusion

In this research work, the effect of leaching temperature, time, liquid to solid ratio, and concentration of acid was investigated, using RSM and CCD techniques. Extraction of TiO_2 as a function of affecting parameters was developed by computer simulation, using the application of a least squares method using DX10 software. The quadratic mathematical model and a statistical analysis (ANOVA) for the extraction of TiO_2 were carried out to investigate the effects of the individual variables and their interactions.

The final model showed that the effects of the individual variables, their quadratic terms, and the interactions among the variables were statistically significant, and the coefficient of multiple determinations was $R^2 = 0.9829$. The proposed regression model based on the coded factors was:

$$\ln(Y) = 3.74 + 0.14x_1 + 0.061x_2 + 0.35x_3 + 0.16x_4 + 0.054x_1x_3 + 0.072x_1x_4 - 0.21x_3^2 - 0.26x_4^2$$

According to the modeled Equation and ANOVA results, liquid-to-solid ratio had the largest effect on the extraction of titanium dioxide. The acid concentration and the leaching temperature were the next effective parameters, respectively. The effect of other parameters was lower than the expressed parameters (C> D> A> AD).

The predicted models were presented in the form of 3-dimensional plots of response surface helping to have a better understanding of the effect of the variables on TiO_2 leaching process.

The results showed that the RSM was a useful technique for evaluating the extraction of TiO_2 in the leaching process. The maximum TiO_2 extraction of 81.32% was obtained at 85°C, 234minutes; liquid to solid ratio of 15 and the concentration of 2.4 M.

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