Optimization alkaline leaching of silicon element from bauxite ore

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A R T I C L E I N F O

A B S T R A C T

The effect of alkaline leaching on the recovery of silicon element from the Iraqi Bauxite Ore. was examined NaOH concentration, particle size, and stirring speed using the program MINITAB 16. NaOH concentration (X1) (1,2, and 4M), particle size (X2) (53,75, and 150µm), and stirring speed (X3) (250,500, and 750 rpm). The best recovery percentage was found to be (94.1097) when the variables were (X2= 53µM), (X1 = 4M), and (X3=750 rpm). The NaOH concentration (X1), particle size (X2), and stirring speed (X3) have a substantial influence on the process of recovery. However, NaOH concentration (X1) and particle size (X2) have a significant effect compared with the stirring speed (X3) on the recovery process.

1. Introduction

Bauxite is a reddish clay-based rock that is most typically found in subtropical and tropical locations. Silica, aluminum oxide compounds (alumina), titanium dioxide and iron oxides make up the majority of bauxite. Red mud or bauxite refinery residue (BRR) is a highly hazardous residue produced during the extraction of alumina from bauxite. The global production of red mud is estimated to be over 150 million tons per year A.Muhanad [1]. Despite the existence of recycling methods, bauxite residue is discarded as waste. It comprises raw materials for the manufacturing of silicon, iron, and aluminum. Titanium oxide, hematite, and goethite are abundant in Iraqi bauxite. Titanium oxide and iron should be eliminated to meet the industrial criteria Hanny et al. [2]. In Iraq, the only identified bauxite resources are the North Hussainiyat karst bauxite deposits in the Western Desert. They have been discovered by the Iraqi Geological Survey in the year 1990 ranging from a few meters to 35 meters thick in Ubaid Formation (early Lower Jurassic) fossil karsts of varying

sizes, coupled with kaolinite, bauxite kaolinite, quartz sandstone, and flint-clay. The thickness of a few highly bauxite profiles can reach 100 meters Their et al. [3]. The sequence of treatments is determined by the raw mineral, chemicals. Those processes primarily center on extraction, which involves separating the impurities that come with the metal and other mechanical components. Shredding processes, wet and dry screening, magnetic separation, gravity separation approaches, electrostatic separation float, and other physical techniques that take use of variations in the chemical and physical surface qualities of the metal and its impurities may be adequate choices. The effectiveness of such operations is determined in large part by the selection of acceptable working conditions for each approach [1] as well as the metal ratio in the raw materials and the technique or procedures used for separating and concentrating them. The aim of this study is to use a hydrometallurgical process for recovering silicon from Iraqi bauxite ore. The recovered silicon

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element is considered as a valuable material for technological and industrial applications.

2. Experimental work

2.1. Atomic absorption spectroscopy

The dissolved Si element in the solution of leaching process, was determined by the atomic absorption spectroscopy type (nova AA 350), at the laboratories of Ibn-Sina state company.

2.2. Materials and method

The Bauxite ore samples were provided by The Iraqi geological survey, which came from the Al-Hussain at area in Western Iraq's Anbar province. The SiO_2 concentration was (14.2) wt%. The chemical composition related to bauxite ore is shown in **Table 1**. The XRF analysis of bauxite ore done in the Iraqi-Germany laboratory, College of Geology at the University of Baghdad. The chemical analysis was achieved by X-ray fluorescence (Shimadzu 1800, XRF). A 1 kg bauxite ore sample was comminuted by laboratory ball mill and jaw crusher, they were done in the mineral processing laboratory of the Department of Production Engineering and Metallurgy, and the bauxite powder was split into equal samples using a laboratory jones riffles splitter for chemical composition and leaching experiment testing.

Table 1. Chemical composition of Iraqi bauxite ore.

Composition	AL_2O_3	SiO_2	Fe_2O_3	Zr_2O_3
Content Wt. %	6.52	14.2	2.169	0.3
Composition	P_2O_5	TiO_2	CaO	
Content Wt. %	0.332	0.452	0.161	

2.3. Leaching Process

The leaching of element from bauxite ore by (NaOH) was performed in a 250 ml, necked round bottom reaction glass vessel. The reaction vessel was heated using the magnetic stirrer hot plate. The stirring of mixture was done by the magnetic stirring coated by Teflon. The leaching efficiency (L%) can be calculated using the following equation Alghanmi et al. [4]:

$$L\% = (C1 \times V)/(Co \times W) \times 100 \tag{1}$$

Where:

L % represents the leaching efficiency's percent, and $C1$ represents the concentration of a metal in solution in g/l. Co represents the concentration of a metal in solid in wt% and V represents the volume of leaching solution in l. W represents the weight of solid sample in gm.

3. Design of Experiments

The elements recovery from bauxite ore is significantly influenced by the process factors. **Table 2** illustrates the independent controllable process factors that have been found to have a substantial impact on leaching. The goal of leaching manipulations is to investigate the effects of specific parameters (leaching agent concentration (X1), particle size (X2), and

speed string (X3)) for determining their optimal values for maximum Si dissolution from bauxite ore, while keeping other variables constant, such as temperature (25 Celsius) and liquid to solid ratio (2:1). **Table 2** lists all of the factors which the present research. Also, the leaching procedure and responses are utilized to determine the value of silicon elements from the Iraqi bauxite ore. **Table 3** shows the experimental outcomes as well as the experimental strategy (Matrix).

Table 2. The factors and their levels employed in the experiments

Factors	Notations	Levels		
		-1	0	+1
NaOH Concentration (X1)	C (M)	1	2	4
Particle Size (X2)	P (μm)	53	75	150
Speed String (X3)	S.S (rpm)	250	500	750

Furthermore, the response surface 'Y' for k factors is represented via the 2nd-order polynomial regression equation Marzouk et al. [5].

$$Y = b_o + \sum_{i=1}^{k} b_i X_i + \sum_{i=1}^{k} b_{ii} X_i^2 + \sum_{i=1}^{k} b_{ij} X_i X_j \tag{2}$$

Where:

b_o = Constant,

b_i = Linear term coefficient,

b_{ii} = Quadratic term coefficients,

b_{ij} = Interaction term coefficient

In which b_i, b_{ii}, and b_{ij} are the coefficients that rely on the main and interaction effects of the factors, and b0 represents the average of responses. Also, the coefficients were estimated with the Minitab 16 program. After finding the coefficients, the mathematical model was created. At a 95% confidence level, all of the coefficients were evaluated for significance.

Table 3. Experimental design matrix and observed values of the recovery

No	X1	X2	X3	C	P	S. S	Y=Recovery Si element %
1	-1	-1	0	1	53	500	83.4
2	1	-1	0	4	53	500	93.8
3	-1	1	0	1	150	500	83
4	1	1	0	4	150	500	89.7
5	-1	0	-1	1	75	250	82.7
6	1	0	-1	4	75	250	85.6
7	-1	0	1	1	75	750	84.1
8	1	0	1	4	75	750	90.1
9	0	-1	-1	2	53	250	86.2
10	0	1	-1	2	150	250	84
11	0	-1	1	2	53	750	87.6
12	0	1	1	2	150	750	86.4

13	0	0	0	2	75	500	85.2
14	0	0	0	2	75	500	85.2
15	0	0	0	2	75	500	85

4. Results and Discussion

4.1. Test of significance

The coefficients of the regressed model have been tested for their significance with a use of RSM (i.e., Response Surface Methodology) in MINITAB program, as can be seen in **Table 4.** Utilizing the level of significance (b = 0,050), coefficients that have p-values > (b = 0.050) are not significant (b23, b22, and b33), and will be removed from the model., the left coefficients (bl, b2, b3, b11, b12 and bl3). As a result, the derived mathematical model for Si recovery might be written as a following:

$$Y = 71.214 + 1.885\,X1 - 0.2736\,X2 + 0.0089\,X3 + 0.021\,X11 + 0.0772\,X12 + 0.0022\,X13 \qquad (3)$$

In which the variables (X1), (X2) and (X3) represent the NaOH concentration, particles size, and stirring speed, respectively, and (Y) represents the recovery Si element. It is noted from equation (3), the NaOH concentration has the greatest influence on the recovery, whatever the case, the NaOH concentration and particles size have the best influence on the recovery, while the effects of stirring speeds (X3) are less in comparison to (X1, and X2).

Table 4. Response surface regression: Y versus X1, X2and X3

Term	Coef	SE Coef	T	P
Constant	71.2145	08.9337	10.2100	00.0000
C	01.8849	02.9056	00.6490	00.0050
P	-00.2736	00.1327	-02.0620	00.0040
S.S	00.0089	00.0171	00.5230	00.0030
C*C	00.0208	00.4715	-00.0440	00.0040
P*P	00.0013	00.0006	02.1570	00.0090
S.S*S.S	-00.0000	00.0001	-00.6930	00.5190
C*P	00.0772	00.0111	-00.6540	00.0020
C*S.S	00.0022	00.0023	00.9630	00.0050
P*S.S	00.0000	00.0001	00.1920	00.0070

4.2. Analysis of variance (ANOVA):

The ANOVA test has been utilized in order to identify the important design parameters that influence on the recovery of Si element. **Table 5** shows that the (p-value) is not more than 5% based on the significant level (0.050) and applying the regression model's test (F-test), indicating that the model of regression is significant [6]. This outcome is acceptable. R-sq Adjusted equals 70.22%, indicating the independent variables (X1, and X2). The rest is a result of other factors like the random error and explains that (70.22%) of the variables ocuur in the variable (Y). In the case when the

determination coefficient is near to one of the best, the findings are likely to be satisfactory.

4.3. The main effect plot of recovery Si element

Figure 1 exhibits the influence of particle size, NaOH concentration, and stirring speed on the recovery of Si from bauxite for values between (+1, -1). It can be seen that there is a gradual increase in the recovery with the increase in NaOH concentration, which is due to the excellent attack of sodium hydroxide on bauxite ore, resulting in the decomposition and disintegration of elements [1].

Table 5. Analysis of variance for Y

Source	DF	Seq SS	Adj MS	F-value	P-value
Regression	9	131.185	14.5761	4.67	0.003
Residual Error	5	15.615	3.1231		
Total	14	146.8			
S=1.76722		R-sq=89.36		R-sq(adj)=70.22	

From figure 1, it can be seen that the recovery increases as the particles size is decreasing, and the highest percentage of (L %) obtained is at 53 μm. The reason for increasing the (L%) with the decrease in particle size is due to the increasing of the surface area exposed to reaction with NaOH, the rate of transmission of materials is large and thus increases the reaction rate Zheng et al. [7]. **Fig. 1** specifies that the leaching of the Si element increases gradually. However, when increasing the speed for more than 500 rpm, the leaching efficiency starts to decrease. During the leaching process, the heterogeneous reaction occurs at the interface, which is between the phases of solid and liquid, at the boundary between the two phases, and the layer of diffusion is created. With regard to solid in the aqueous phase, such layer includes a stationary aqueous layer, the diffusion layer might be thinned through excessive stirring, yet not totally removed, in which the leaching effectively elevated with the increase in stirring speed. Increasing the stirring speed causes an increase in the leaching efficiency because of the suspension of mineral particles and a decrease in the thickness of the mass transfer boundary layer on the particle surface Shabani et al. [8]. While, the noted lower efficiency of leaching with a higher stirring rate of more than 500 rpm might be ascribed to the violent agitations causing particles to adhere to the flask's inner wall. Such an approach decreased the efficiency of leaching Wang et al. [9].

4.4 Normal probability plots

The normal probability plot of the residuals is shown in **Fig. 2.** The residuals of data are essentially distributed normally since the total points make an approximately straight line, as shown by the normal probability plots created with the computer software MINITAB. The distribution and spread of residual are shown in **Fig. 3** which happens in a random form on the two sides of a line representing the value of 0 (a line separating the negative and positive residuals). This is because the error of result is a random error, which cannot be monitored in a specific form, as it is

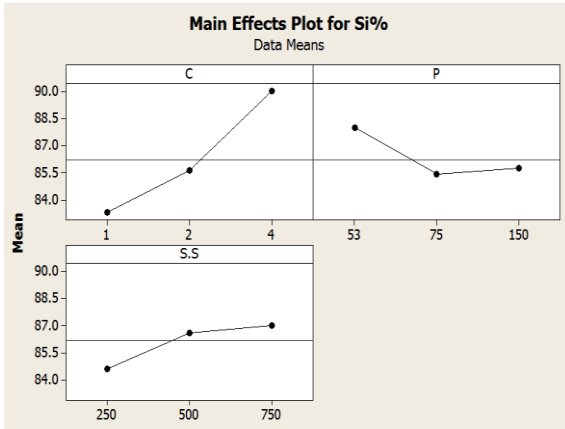not decreasing or increasing or only exists on one side. Thus, such residuals do not have a constant variance

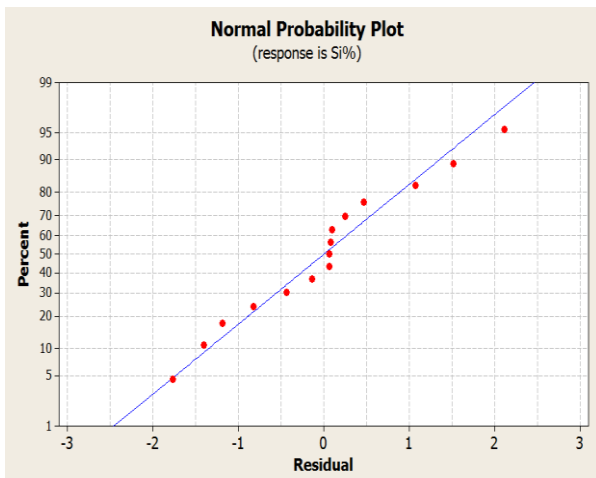**Figure 1. Main effect plot of residuals of recovery Si element**

Figure 2. Normal probability plot of residuals of recovery Si element
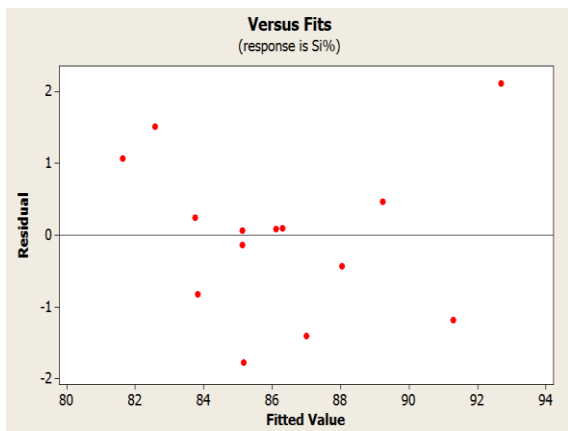

Figure 3. Plot of residuals vs value of recovery Si element

4.4. Optimization of recovery of Si

The optimization chart for the recovery of Si at various values for three factors (X1, X2, and X3) is shown in Fig. 4. The best setting of each one of the parameters is displayed in the middle of the top row, whereas optimization of the result has been displayed in the left column. Each factors behavior curve is depicted below. As illustrated, an optimal run at a concentration of NaOH (4 M), stirring speed (750 rpm) and a particle size of bauxite ore (53µ m) might lead to the silicon element recovery (94.1097%). The values of particle size (X2), sodium hydroxide concentration (X1), and stirring speed (X3) produced with the use of programs were applied, resulting in a silicon element recovery of (95.88%), which is substantially identical to that achieved by the program.

4.5. Response surface analysis

Fig. 5 A, B, and C shows three-dimensional plots and figure 5 D, E, and F shows the contour plots for the effect of factors (X1, X2, and X3) on the response Y (recovery Si element from bauxite ore). Where, it was noted that there is a sharp increase in the recovery with an increase in the concentration of sodium hydroxide (X1) compared with other factors(X1) and (X2). As for the particles size (X2) and the stirring speed (X3), they have almost the same effect, where the increased recovery is clear with the decrease in the volume of the raw particles and the increase effect than the combined effect of sodium hydroxide(X13) and the stirring speed or the combined effect of the stirring speed with the size of the particles(X23).in the stirring speed. On the other hand, it was observed that the combined effect of sodium hydroxide and the size of the particles(X12) have a greater effect.
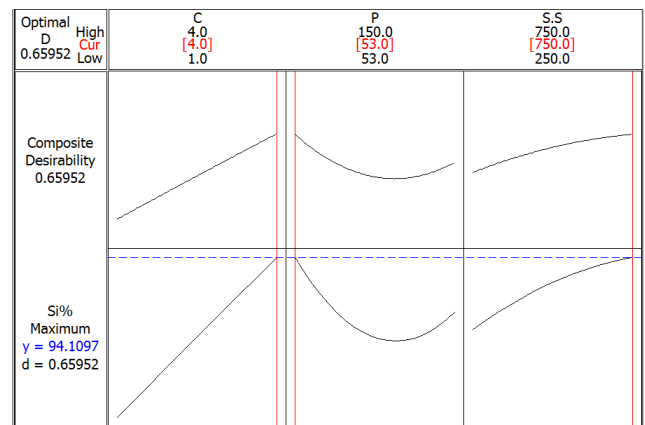

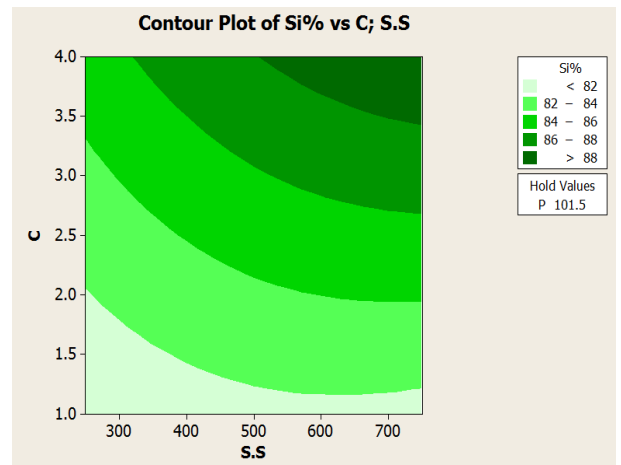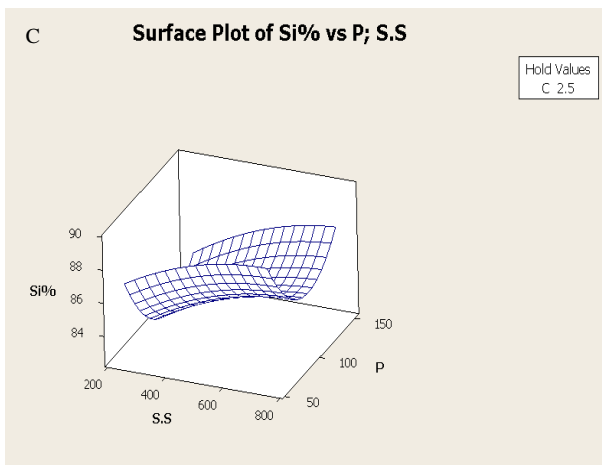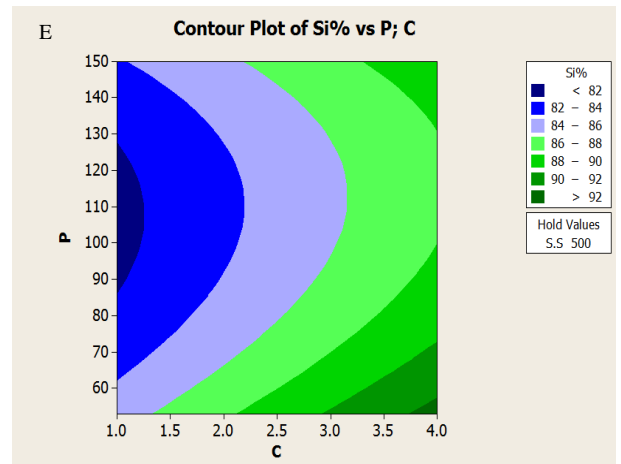**Figure 4. Optimization chart for maximum of recovery Si element.**
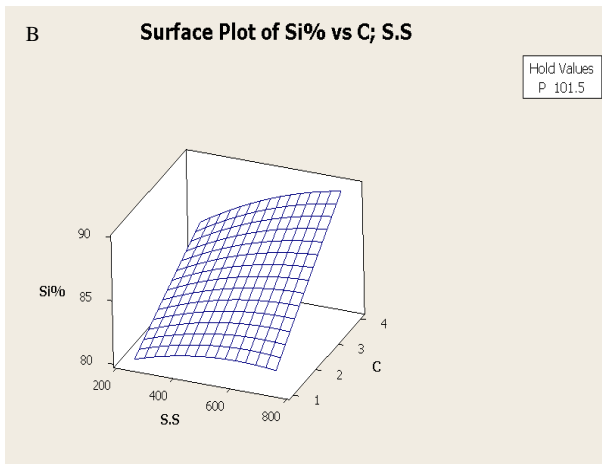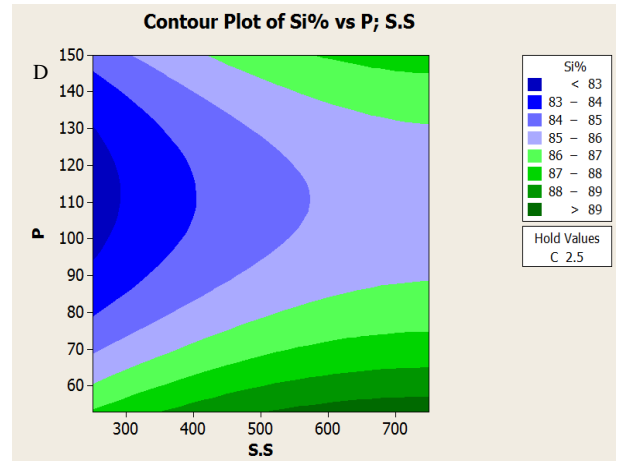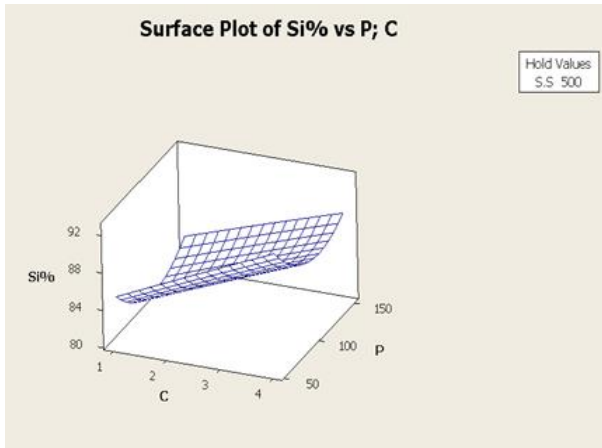
Figure 5. Three-dimensions and contour plots of recovery Si.

5. Conclusions

1. Regression models might be represented by Eq. 3, which X1 has a considerable impact on the recovery of Si element. It must be noted that X1 has the greatest impact in comparison to the influence of X2 and X3.

2. The combined influences of NaOH and the size of the particles (X12) have greater effect as a compared with the combination effect of sodium hydroxide (X13) and the stirring speed or the combined effect of the stirring speed with the size of the particles (X23).

3. The increase in the concentration of NaOH (X1) and decrease in particles size of bauxite (X2) and increase in the stirring speed(X3) caused the increased improvement of the recovery of Si.

4. The optimal recovery of Si element has been obtained at NaOH concentration (X1) 4M, particles size (X2) 53um, and stirring speed (X3) 500 rpm, in which the recovery of silicon element (93.8 %).

5. It is possible to obtain an increase in the recovery of the silicon element (94.1097 %) when using the variables, NaOH concentration (X1) 4M, particles size (X2) 53um, and stirring speed (X3) 750 rpm.

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