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Enhancing the Technical Qualifications of Egyptian White Sand Using Acid Leaching; Response Surface Analysis and Optimization

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Abstract: White silica sand samples were collected from Zafarana area along the Red sea coast, Egypt. The samples were mixed and quartered to obtain representative sample for characterization. The silica content in the sample is 99.441% and the iron content is 0.112%. Such geochemical qualifications don't match the technical specification of ceramic, optics, silicon metals and solar cells. RSM in conjunction with CCRD was used to study the removal of iron from silica sand using oxalic acid in terms of three operating parameters (Contact time, Acid concentration, Temperature). The model F values indicated the high significance of the design, also the good agreement between the Actual and the predicted results ($R^2 > 0.9$) indicated suitability of second order quadratic polynomial model to represent the removal process. The best removal process (82%) was achieved at 8gm per ton oxalic acid concentration, 95°C temperature and 120 min leaching time. The final product exhibit high silica content (99.683% SiO_2) and lower iron content of (0.017%) which match the requirements of ceramic, silicon carbide, silicon metal and the production of silicon for solar cells.

Keywords: White Sand, Oxalic Acid, Response Surface Methodology, Optimization

1. Introduction

The world demand of high quality silica sources increase gradually with time for their applications in traditional and high-tech applications [1]. High quality silica resources of silica sand, hydrothermal quartz and pegmatitic quartz become backbone raw material in several advanced applications such as semiconductors, high temperature lamp tubing, telecommunications and optics, microelectronics, and solar silicon applications [2], [3], [4].

Natural silica resources in general contain impurities of other minerals and oxides. Such impurities have a strong influence on the chemical quality and in turn the technical qualification of the silica raw materials [5]. Among the common impurities in silica sand are iron oxide impurities which cause the highest damage by both their color and properties [6]. Iron impurities are prohibitive to some advanced applications such as optical fibers, silicon for solar cells, semiconductors, microelectronics and refractories [7]. The main ferruginous minerals which present as impurities are iron oxides, siderite, pyrite, rutile, tourmaline and mica [8, 9].

Therefore such deposits require several beneficiation and upgrading techniques to match the technical specifications of high advanced industrial applications. Physical and chemical beneficiation methods such as gravity and magnetic separation, attrition, flotation and acid or alkaline leaching are used to remove iron impurities from quartz sands [10], [11], [12]. Chemical processing to remove the iron oxide contaminants appear to be highly efficient as compared to physical beneficiation [13].

Leaching of iron bearing impurities can be performed using, H_2SO_4, HCl, HF, phosphoric acid and oxalic acid. Liu et al., (1996) [14] used a mixture of 10% HF acid and 90% H_2SO_4, HCl and HNO_3 for 3–12 h; they obtained 96.3% of iron removal. From a raw sand containing more than 420 ppm Fe, a product containing 84 ppm Fe was achieved after two days of leaching using H_2SO_4 with an initial concentration of 25 g/l and subsequent attrition using NaOH solution and gravity separation [15].

Phosphoric acid also used as one of the best leaching agents in the purification of iron impurities from the quartz. It has advantages of a high leaching rate, low cost of production, simplified flow sheet, and of less harm to our target product. The optimal leaching rate is up to 77.1% where the iron content reduced from 0.048% to 0.014% in the conditions of temperature 80°C, ratio of solid to liquid 10%, and quartz sand size of 100 mesh for 120 min [16]. Also oxalic acid show high efficiency in leaching of iron impurities from silica sand. Veglio et al., [17] leached iron from the silica sand by oxalic acid and obtained 45– 50% iron removal in the conditions of temperature 80°C, average size 160 µm, 3 g/l oxalic acid and 10% (w/v) ratio of solid to liquid for 3 h. Du et al., [7] used the oxalic acid in leaching iron and obtained 75.4% leaching rate in the conditions of 4 g/l oxalic acid, 95°C, 500 rpm/150 W and the iron content reduced from 0.06% to 0.045%.

Egypt is blessed with very high reserve of different types of silica resources [18]. The main problem facing the production of high quality silica of high technical qualification is the present of some mineral contaminants. So the aim of this paper is to (1) enhance the technical qualification of Egyptian white san sample by reducing the iron bearing impurities, and to (2) set the optimum condition for maximum removal of iron bearing impurities depending on response surface methodology (RSM) in conjunction with central composite rotatable design (CCRD).

2. Experimental Work

2.1. Materials and Methods

White silica sand samples were collected from Zafarana area along the Red sea coast, Egypt. Oxalic acid was delivered from Sigma Aldrich Company, Egypt. The complete chemical analysis of the silica sand sample was performed by Pnalytical Axios Advanced XRF technique at Central Metallurgical Research and Development, Egypt. The present mineral phases were detected from X-ray powder diffraction pattern of the silica sand sample using a Philips APD-3720 diffractometer with Cu Kα radiation, operated at 20 mA and 40 kV in the 2θ range of 5–70 at a scanning speed of 5°/min. the nature of the iron impurities was investigated using binocular microscope.

2.2. Leaching of Iron

Response surface and Central Composite Rotatable Design of quadratic polynomial model was designed as function of three operating parameters (leaching contact time, concentration of oxalic acid and the temperature). The design was used to detect the effect of the operating parameters, interaction between them and the optimum conditions for maximum removal of iron by oxalic acid leaching.

The experiments were performed according to tests suggested by the statistical design. The tests were performed in terms of the upper and lower limits for the selected variables (Table 1).

Table 1. *Upper and lower values of the parameters.*

Factor	Name	Low actual	High actual	Low coded	High coded
A	Contact time	10 min	120 min	-1	1
B	Acid Conc.,%	1g/l	8g/l	-1	1
C	Temperature	45ºC	95ºC	-1	1

The experiments were carried out in a 250 ml round bottom flask where 20 g from the ground quartz was added into the oxalic acid solutions. The solid/ liquid ratio is fixed at 1.5 and the stirrer speed adjusted at 500 rpm. Also pH value adjusted at 2.5 by nitric acid solution (0.1 N) for all the testes. At the end of each test the solid is removed by filtration using Whatman 45 µm pore size filter paper. The results were evaluated by following iron concentration in the filtrate solutions using a spectrophotometric method (DIN EN 1189, 1996) with a Lamp UV/VIS Spectrophotometer (M160 PC) in Nanophotonics and Applications Lab, Beni Suef University. The removal efficiency of iron oxide from the original value was calculated as follow:

$$\text{Removal of Fe.}, \% = \frac{(C_0 - Ce)100}{C_0} \quad (1)$$

Where C_0 is the iron content in the sand and C_e is the iron dissolved in the solution.

The data obtained were fitted to a second-order polynomial equation:

$$Y = \beta_o + \sum_{i=1}^{4} \beta_i X_i + \sum_{i=1}^{4} \beta_{ii} X_i^{2} + \sum_{i=1}^{3} \sum_{j=i+1}^{4} \beta_{ij} X_i X_j \quad (2)$$

Where Y is the amount of dissolved iron (mg/L) and the removal efficiency (%); β0, βi, βii, βij are constant coefficients Xi are the uncoded independent variables. Subsequent regression analyses, analyses of variance (ANOVA) and response surfaces were performed using the Design Expert Software (Version 6.0.5). Optimal reaction parameters for maximum removal were generated using the software's numerical optimization function.

3. Results and Discussion

3.1. Characterization

Results of XRF chemical analysis of the quartz sample were listed in Table 2. The SiO_2 content is 99.441%, the present impurities represent about 0.56% (5600 ppm) and Fe_2O_3 is about 0.112%. Therefore, the qualifications of the sample do not match the specifications of high quality quartz for advanced and high technical applications such as ceramic (< 0.02% Fe_2O_3), silicon carbide (less than 0.1 Fe_2O_3), optics (< 0.014% Fe_2O_3), silicon metal (0.01 to 0.02 Fe_2O_3) and the production of silicon for solar cells (< 0.04% Fe_2O_3).

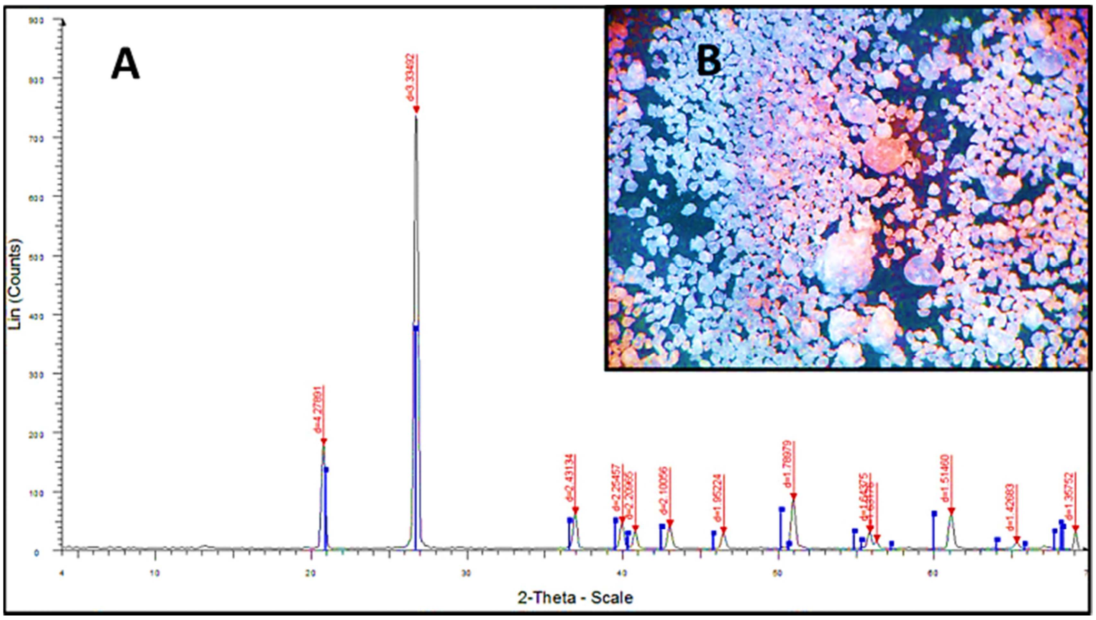

Figure 1. *Shows (A) XRD pattern of the sand sample and (B) sand grains under the binocular microscope.*

Table 2. *Major oxides of the sand sample.*

Oxides	%
Na_2O	0.033
K_2O	0.040
CaO	0.048
MgO	0.052
Al_2O_3	0.077
Fe_2O_3	0.112
TiO_2	0.031
P_2O_5	0.042
SO_3	0.058
Cl	0.024
Cr_2O_3	0.020
ZrO_2	0.014
MnO_2	0.029
SiO_2	99.441

The XRD pattern of the sand sample revealed that, the sample is composed mainly of quartz minerals with no indications for the presences of other mineral phases (Fig. 1A). The petrographic study using the binocular microscope showed the iron bearing impurities as free iron oxide particles and sand grains stained by iron oxide (Fig. 1B).

3.2. Removal of Iron from Sand

3.2.1. Significance of the Model

Table 3 summarizes the experimental runs designed with the CCRD and the resulted responses (Dissolved iron (%) and Removal Efficiency (%). Second order quadratic polynomial model was selected to represent the relations between the selected parameters and the required responses. Model F-values for the required responses were obtained from the analysis of variance (ANOVA). The model F-values were 52.13 and 47.37 for the percentage of dissolved iron and the removal efficiency, respectively. Therefore the model is highly significant with only a 0.01% noise [19].

Table 3. *Results of the designed experimental runs.*

Run	Contact time (min)	Acid Conc., (gm/T)	T (°C)	Iron dissolved (%)	Removal efficiency (%)
1	120.00	8.00	95.00	0.092	82
2	120.00	1.00	95.00	0.066	59
3	60.00	4.00	45.00	0.064	57
4	120.00	1.00	45.00	0.061	55
5	60.00	4.00	75.00	0.066	59
6	60.00	4.00	95.00	0.073	65
7	120.00	4.00	75.00	0.069	62
8	10.00	4.00	75.00	0.060	54
9	60.00	1.00	75.00	0.059	53
10	10.00	1.00	45.00	0.051	46
11	10.00	1.00	95.00	0.056	50
12	60.00	4.00	75.00	0.067	60
13	10.00	8.00	45.00	0.068	61
14	10.00	8.00	95.00	0.075	67
15	120.00	8.00	45.00	0.074	66
16	60.00	8.00	75.00	0.079	71

The regression plotting of the predicted and experimental values for the dissolving of iron and the removal efficiency, respectively, show great agreement with determination coefficient ($R^2 > 0.9$) for the required responses (Fig. 2). This gave an indication about the significance of the quadratic polynomial model and its suitability to represent the actual relations between the required responses and the selected variables [20].

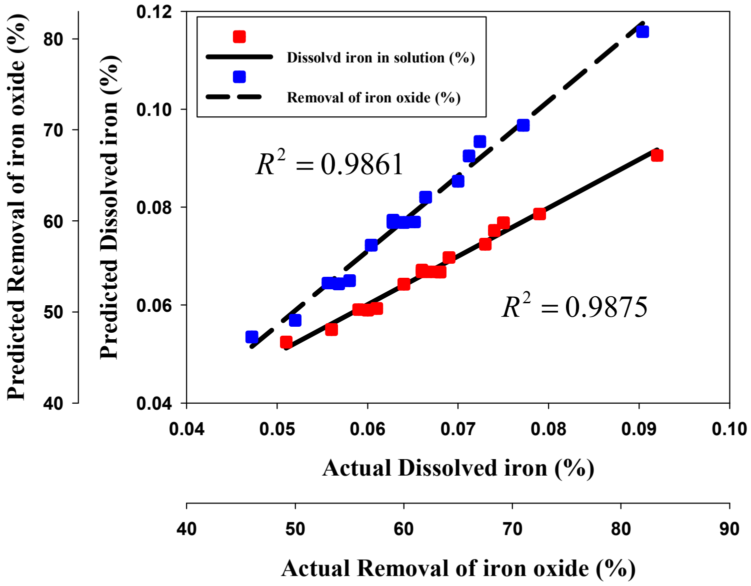

Figure 2. *R^2 values for the actual and predicted values for removal of iron from silica sand.*

The mathematical equations of the quadratic polynomial model, which represent the relations between the required responses (dissolved iron (%) and the removal efficiency (%) and the selected variables, were obtained from Design Expert Software (Version 6.0.5). The mathematical regression equations for the removal efficiency for coded units as follows:

$$Y = + 60.66 + 4.56 X A + 8.33 X B + 3.87 X C- 2.44 X A^2 + 0.79 X B^2 + 1.78 X C^2 + 0.27 X A X B +1.20 X A X C + 1.75 X B X C$$

Where Y is the removal efficiency, A is the contact time, B is the acid concentrations and C is the temperature.

3.2.2. Effect of the Parameters and Interaction

The results revealed that, the amount of dissolved iron and in turn the removal efficiency increased gradually with increasing the contact time from 10 minute to 120 min (Fig. 3A and B). Also the removal efficiency increased sharply with increasing the oxalic acid concentration from 1gm/T to 8gm/T. The effect of temperature also was recorded by increasing the amount of dissolved iron with increasing the temperature from 45°C to 95°C. (Fig. 3A and B)

The interaction between the operating parameters play major role in dissolving of iron from silica sand and the optimum value for each variable controlled mainly by the other variables. The interaction between the operating parameters was represented by contour and 3D response surface diagrams.

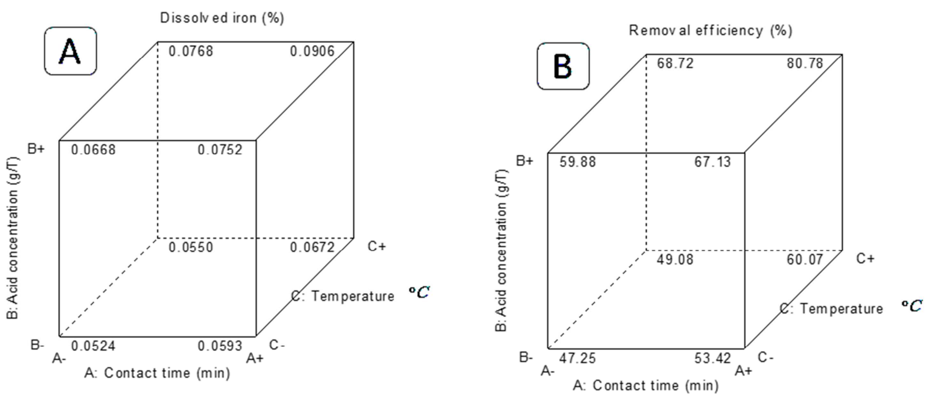

Figure 3. *The effect of the operating parameters on (A) the removal efficiency of iron impurities and (B) amount of dissolved iron in the oxalic acid solution.*

Response surface plots in terms of two selected factors at any one time maintaining all other factors at fixed levels are suitable in understanding the interaction effects of these two factors and represented by contour or 3D diagrams for dissolving of iron and the removal efficiency. The elliptical shape of the curve indicates good interaction of the two variables and circular shape indicates no interaction between the variables.

3.2.3. A. Interaction Between Contact Time and Oxalic Acid Concentration

The interaction between the contact time and the concentration of oxalic acid is represented in Fig. 4 by contour diagrams for the dissolving of iron and in Fig. 5 by 3D diagrams for the removal efficiency. The interaction effect of contact time on the percentage of dissolved iron was investigated at fixed condition of oxalic acid concentration (4gm/T) and temperature (75°C).

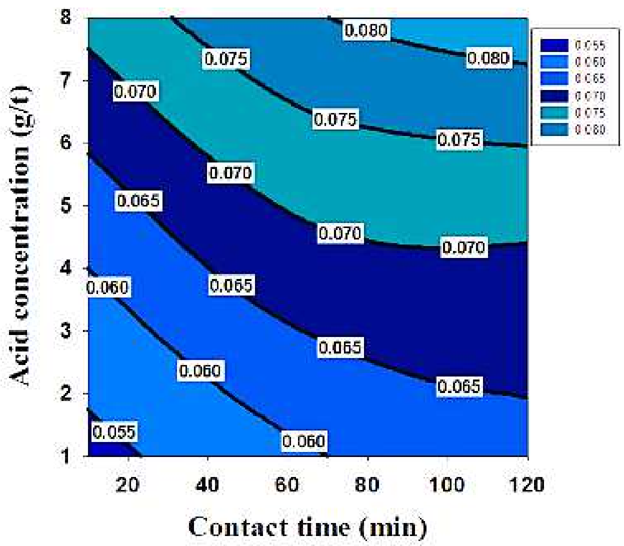

Figure 4. *Contour diagram for the interaction effect between the contact time and oxalic acid concentration on dissolving of iron.*

Under these conditions the percentage of dissolved iron increased from 0.06% to 0.069% with increasing the leaching time from 10 minute to 120 minute. These results match increasing in the removal efficiency from 54% to 62% (Fig. 5). The interaction effect of the acid concentration appears in decreasing the removal capacity with decreasing the initial concentration from 4gm/T to 1gm/T at fixed temperature 75°C.

With decreasing the oxalic acid concentration to 1 gm/T, the percentage of dissolved iron was reduced. The increasing with time was detected to be from 0.051% to 0.061% with increasing leaching time from 10 min to 120 min. This match reducing in the raising of the removal efficiency with the change in time from 46.6% to 55.7% instead of increasing from 54% to 62% at oxalic acid concentration 4 gm/T. Therefore the maximum effect of time can be obtained at the upper limit of oxalic acid concentration (8 gm/T).

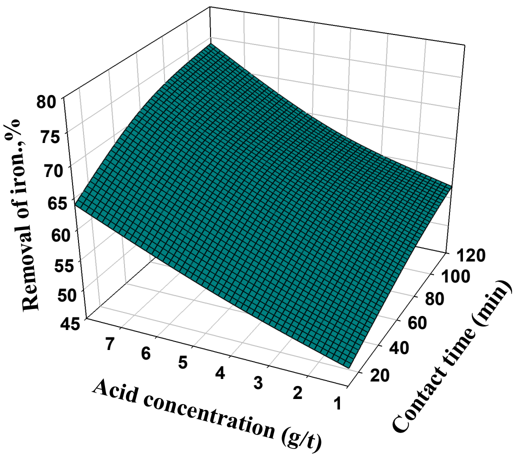

Figure 5. *3D diagram for the interaction effect between the contact time and oxalic acid concentration on the removal efficiency of iron.*

At oxalic acid concentration (8 gm/T), the percentage of dissolved iron increased from 0.07% to 0.082%, which associated with increasing in the removal efficiency from 63.4 to 73.6%. This occurs with increasing the leaching time from 10 min to 120 min.

3.2.4. B. Interaction Between Contact Time and Temperature

The interaction between the leaching time and the temperature was represented by contour diagram for the dissolving of iron on oxalic acid solution (Fig. 6) and by 3D diagrams for the removal efficiency of iron from silica sand (Fig. 7). Studying the interaction effect between the leaching time and the temperature was performed at fixed acid concentration 8gm/T and fixed leaching time 120 min. Under such conditions the dissolving of iron from the silica sand increased from 0.074% to 0.092% with increasing the operating temperature from 45°C to 95°C (Fig. 6). Such values match increasing in the removal efficiency of iron from 66 to 82% (Fig. 7). This represents the removal behavior at the optimum leaching time (120 min).

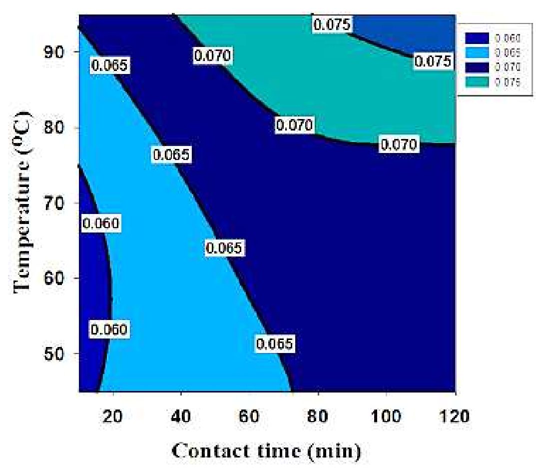

Figure 6. *Contour diagram for the interaction effect between the contact time and Temperature on dissolving of iron by oxalic acid solution.*

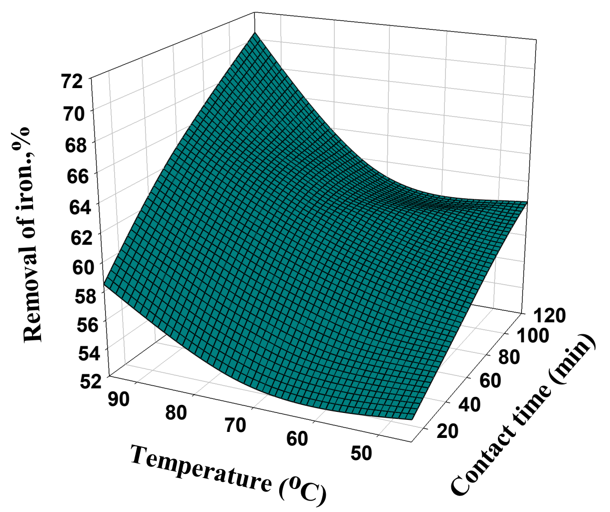

Figure 7. *3D diagram for the interaction effect between the oxalic acid leaching time and the temperature on the removal efficiency of iron.*

The interaction effect of leaching time on the removal capacity of iron bearing impurities with temperature was investigated at 8gm/T oxalic acid concentration and at lower leaching time of 10 min. Under the previous leaching time, the dissolving of iron bearing impurities from sand was increased from 0.068% to 0.075% with increasing the temperature from 45°C to 95°C. The previous results match increasing in the removal efficiency of iron oxide from 61 to 67%. The obtained results for the effect of temperature on the removal of iron at different leaching time revealed that, the removal capacity reach the maximum value at the upper limit of temperature (95°C) and the upper limit of leaching time (120 min).

3.2.5. C. Interaction Between Temperature and Oxalic Acid Concentration

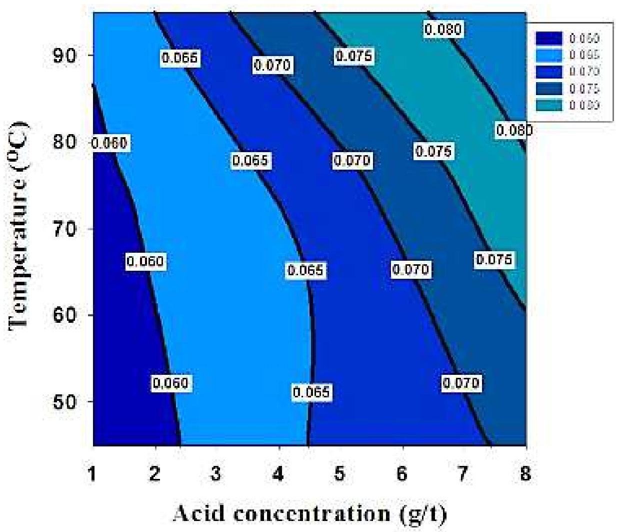

Figure 8. *Contour diagram for the interaction effect between oxalic acid concentration and temperature on dissolving of iron by oxalic acid solution.*

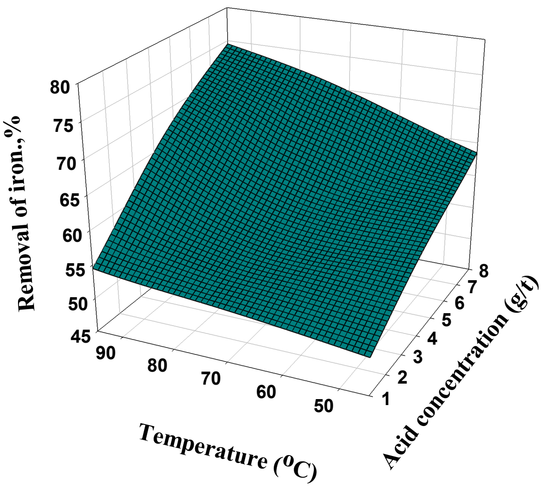

Figure 9. *3D diagram for the interaction effect between the oxalic acid leaching time and the temperature on the removal efficiency of iron.*

The interaction between the oxalic acid concentrations that used in leaching of iron from silica sand and the operating temperature during the process was represented by contour diagram for the dissolving of iron on oxalic acid solution (Fig. 8) and by 3D diagrams for the removal efficiency of iron from silica sand (Fig. 9). The interaction between oxalic acid concentration and the temperature in °C was investigated at fixed conditions of leaching time (120 min) and temperature of (95°C).

As stated previously the removal of iron bearing impurities from Egyptian white sand increase sharply with increasing the oxalic acid concentration under any operating conditions of temperature and time. Thus, the optimum experimental conditions for maximum removal of iron bearing impurities can be achieved at the upper limit for all the operating variables.

3.2.6. D. Optimization

Taking advantage of the quadratic programming, the predicted optimum conditions for maximum iron removal from silica sand using oxalic acid leaching can be obtained using Design Expert's optimization function in terms of the upper and lower limits for the selected variables (contact time, concentration of oxalic acid and temperature). The optimum solution for the operating conditions is 120 min contact time, 95°C temperature and 8gm/T oxalic acid concentration. Under these conditions the removal efficiency increased to the maximum value (82%).

3.3. Qualification of the Final Product

The chemical analysis for the final product after treatment in the optimum conditions listed in Table 4. The resulted data revealed that the silica content was raised to 99.683% i.e the present impurities reduced to about 0.32% (3200 ppm) from 0.56% (5600 ppm) in the original white silica sand sample. Also the iron content with reduced to 0.017% which match the requirements of ceramic (< 0.02% Fe_2O_3), silicon carbide (less than 0.1 Fe_2O_3), silicon metal (0.01 to 0.02 Fe_2O_3) and the production of silicon for solar cells (< 0.04% Fe_2O_3). i.e treatment of Egyptian silica sand deposits can enhance their technical qualifications to match the specifications of some advanced applications.

Table 4. *Major oxides of the final product after leaching by oxalic acid.*

Oxides	%
Na_2O	0.035
K_2O	0.038
CaO	0.041
MgO	0.022
Al_2O_3	0.059
Fe_2O_3	0.017
TiO_2	0.010
P_2O_5	0.032
SO_3	0.010
Cl	0.020
Cr_2O_3	0.010
ZrO_2	0.013
MnO_2	0.012
SiO_2	99.683

4. Conclusion

Egyptian silica sand sample with low silica content of 99.441% and higher iron content of 0.112% was investigated for advanced applications as well as the applicability of oxalic acid leaching of iron bearing impurities to upgrade its technical qualifications.

The sample composed mainly of quartz minerals and iron bearing impurities present as iron oxide or quartz coating iron oxide. The technical qualifications don't match the requirements of ceramic, optics, silicon metals and solar cells applications. RSM associated with CCRD was used to study the removal of iron from silica sand using oxalic acid in terms of three variables (Contact time, Acid concentration, Temperature). The model F values and the good agreement between the predicted removal of iron and the experimental results indicated the high significance of the design and the second order quadratic polynomial model to represent the removal process. The operating conditions of 8 gm/T oxalic acid concentration, 95°C temperature and 120 min leaching time are the beast conditions for maximum removal of iron (82%). The final product exhibit high silica content (99.683% SiO_2) and lower iron content of (0.017%) which match the requirements of ceramic, silicon carbide, silicon metal and the production of silicon for solar cells.

Leaching of Egyptian white silica sand by oxalic acid can upgrade its quality and its technical qualifications to match the specifications of some advanced and high technical applications.

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