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# Proposing a seven-parameter kinetics model for predicting cerussite flotation recovery

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ABSTRACT

Sulfide lead resources are being depleted and the exploitation of carbonate lead deposits is now the main focus of lead mining. Cerussite, $PbCO_3$, is majorly discarded to tailing damps because it is difficult to be processed by flotation in lead concentration units. This paper not only investigates the optimization of cerussite flotation, but it also proposes a model for predicting the recovery. Froth flotation was used for cerussite recovery from a previously existing tailing damp in the ChahGaz mine in Kerman Province, Iran. The response surface method was used for experimental design and optimization of Pb flotation in which a statistical experimental model was suggested to model flotation kinetics based on the effective parameters. The results showed that particle size, pH, solid content, $Na_2S$ dosage, collector dosage and collector type to be the most effective parameters. These parameters were applied for investigating flotation kinetics. A three-fraction (with seven-parameter) flotation model, with fast, medium and slow kinetics rate constants was obtained via 64 designed tests. The proposed model showed a good agreement with experimental data ($R^2$ more than 0.8). Also optimum conditions of cerussite flotation were set at pH= 9, $d_{80}$= 53 µm, solid content= 26%, $Na_2S$= 4000 g/t and collector dosage = 1500g/t of PAX.

**Keywords :** *Cerussite; Flotation; Kinetics; Modeling; Tailing damp*

## 1. Introduction

In order to optimize flotation reactions, the kinetics of the system should be studied [1]. An optimized kinetics model can be used to calculate the recovery of concentrate in each time [2]. From the 1930s to late 1987, various mathematical models have been introduced in order to describe the flotation process [2]. The first paper related to flotation kinetics was published by Garcia-Zuniga in Chile, in 1935 [3, 4]. He proved that recovery is an exponential function of time. Soon after, Beloglazov [5] and Schuhmann [6] developed the conditions for applying in continuous studies at a steady-state. In 1948, Sutherland [7] performed a theoretical study of the particle-bubble adhesion and derived a new equation.

The interest for flotation kinetics was revived by a paper of Arbiter [8, 9], who proposed a second-order equation to represent the result of Zuniga. Beloglazov and Sutherland obtained their result in laboratory batch tests and industrial cells [10]. There are many other studies carried out on flotation kinetics [11-69]. These numerous references show the importance of kinetics modeling in the flotation process.

The kinetics study of the flotation process includes the determination of all the factors that influence the production rate of concentrate [10, 20]. These factors are as follows:[40]

1) Chemical factors: collectors, frothers, activators, depressants, pH.
2) Equipment factors: cell design, agitation, airflow, cell bank configuration, cell bank control.
3) Operational factors: feed rate, mineralogy, particle size, pulp density, temperature.
4) Mineral factors: size and shape, degree of liberation, type of mineral surface and presence of intrusive elements on the surface of the mineral.

The best classification of flotation kinetics models that have already been presented is as follows [2]:

- Models with unique rate constant
- Models with variable rate constants, like:
- Kineticsmodels with discrete rate constant
- Kineticsmodels with a continuous rate constant distribution
- Models with an average rate of flotation

Some researchers believe that the use of kinetics models with discrete rate constant will be more appropriate than other models due to the non-homogeneity of the pulp. Also, they believe that these models have the highest correlation with experimental results in batch flotation. There are several discrete rate constant distribution models. The difference between them is the number of fractions assumed [17, 27, 30, and 70].

Some research studies have been carried out on the effect of different parameters on the flotation kinetics [2, 34-38, 41, 51-56, 59, 68]. In almost all of these studies, few effective parameters have been investigated, and there is also little research on lead flotation kinetics, especially on cerussite. This work investigates the flotation of cerussite. The parameters such as pH, particle size, solid content, $Na_2S$ dosage, collector dosage and collector type were considered as affective parameters on the flotation process. These parameters were optimized through the response surface methodology (RSM) based on central composite design (CCD) model. In addition to the optimization of flotation recovery, the kinetics model parameters of the system were investigated.

## 2. Material and methods

### *2.1. Sample, parameters, and instruments*

The sample was obtained from the tailing damp of the ChahGaz mine in Kerman Province, Iran. The specifications of the sample are presented

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in Tables 1, 2. Initially, some tests were carried out on the sample to determine the most important parameters and also their best range of variation (Fig. 1).

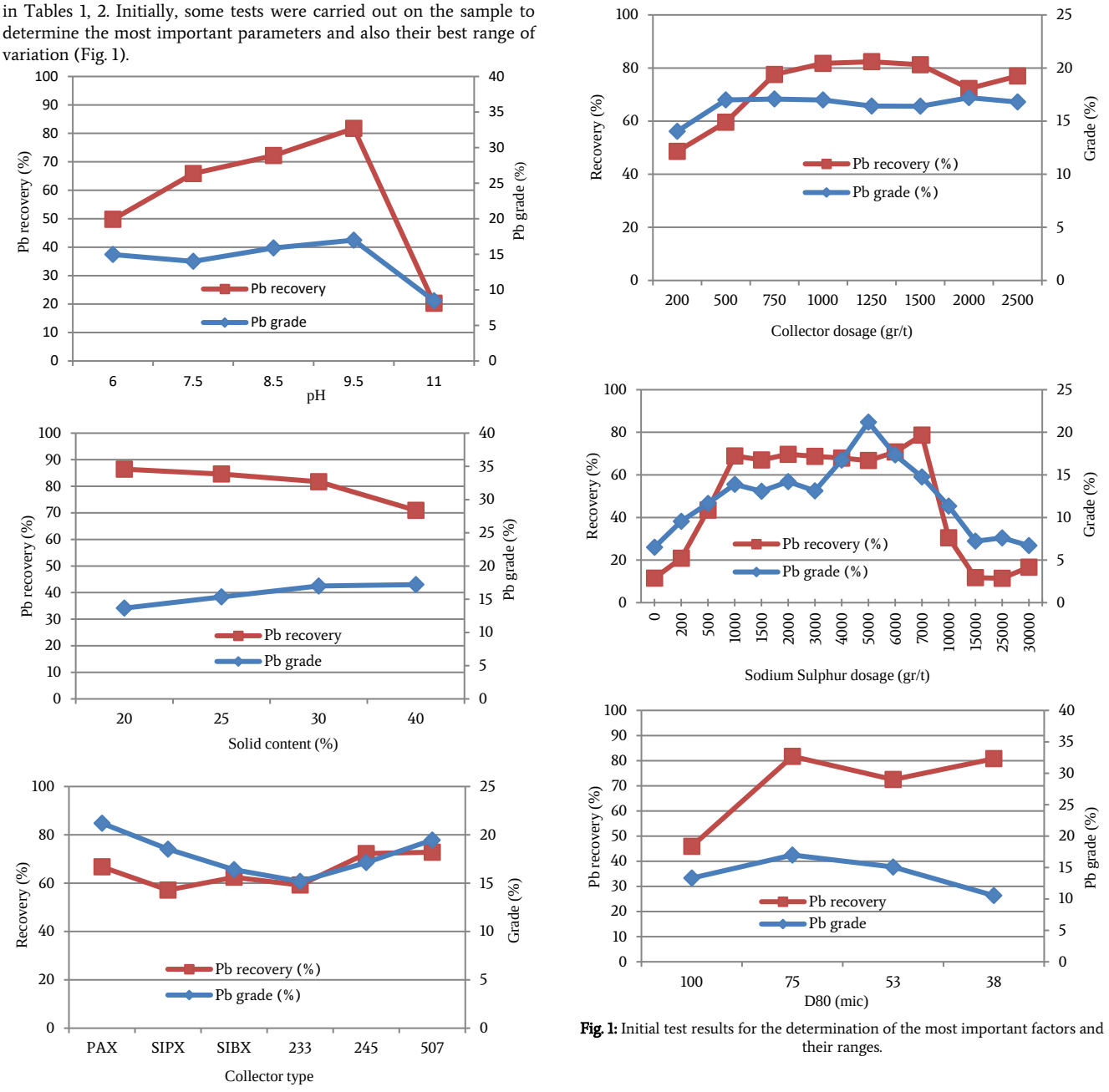



**Fig. 1:** Initial test results for the determination of the most important factors and their ranges.

**Table 1.** Chemical composition of the representative sample (XRF).

| Content | $SiO_2$ | $Al_2O_3$ | $Fe_2O_3$ | CaO | $Na_2O$ | $K_2O$ | MgO | MnO | $TiO_2$ | $P_2O_5$ | Pb | Zn | BaO | $SO_3$ | Cu | L.O.I | SrO |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **Wt. (%)** | 42.61 | 11.78 | 12.39 | 3.61 | 0.08 | 2.09 | 1.07 | 0.081 | 0.42 | 0.071 | 6.77 | 2.38 | 1.66 | 3.923 | 0.1 | 10.9 | 0.065 |

According to initial experiments, in this case, the most effective parameters on cerussite flotation are shown in Table 3. Five collector types were used in the experiments. They are PAX (Potassium Amyl Xanthate), SIPX (Sodium Isopropyl Xanthate), SIBX (sodium isobutyl xanthate), 233 (sodium diisopropyl dithiophosphate), 245 (sodium diisobuyl dithiophosphate) and 507 (sodium diisobuyl dithiophosphate and sodium mercapto benzothiazole). Then, the flotation kinetics tests were carried out. The tests were done in a Denver D-12 flotation cell with one-kilogram of sample and the Pb content of the samples was measured by atomic absorption spectroscopy (AAS) using Uniqam 939 model.

**Table 2.** Mineralogical composition of the representative sample (XRD).

| Content | Quartz | Anhydrite | Cerussite | Vermiculite | Muscovite | Montmorillonite | Clinoenstatite | Calcite | Illite | Anorthite | Chlorite | Gypsum |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **Wt. (%)** | 39.8 | 9.4 | 9.4 | 6.9 | 6.9 | 4.6 | 4.6 | 4.5 | 4.5 | 3.5 | 3 | 2.9 |

**Table 3.** The most effective flotation parameters in the tests.

| Parameters | unit | The range of test parameters | The most effective range/parameter |
|---|---|---|---|
| pH | - | 6.0-11.0 | 7.5-9.0 |
| $d_{80}$ | Micron | 38-100 | 53-75 |
| Solid Content | % | 20-50 | 25-30 |
| $Na_2S$ dosage | g/t | 0-30000 | 4000-7000 |
| Collector dosage | g/t | 200-3000 | 1500-2500 |
| Collector Type | - | PAX-SIPX-SIBX-233-245-507 | PAX-507 |

### *2.2. Experimental Design*

The response surface method is a combination of statistical and mathematical techniques, which has been widely used for modeling and analysis of the systems in which the dependent variable is affected by several parameters, simultaneously [1]. This methodology is applicable for the optimization of outputs, considering the separate effects of parameters, and analyzing the interaction between parameters [71–73]. The central composite design has been widely used as a subtype of surface response method. In this paper, a half fraction central composite design with 5 replications at the central points was used for the experiment to study the operating parameters affecting the Pb recovery. The input includes six variables with five levels consisting of upper and lower axes, factorial upper, factorial lower and central point.

Based on the initial studies on this case, the parameters considered as experimental design inputs included pH (A), particle size (B), solid content (C), $Na_2S$ dosage (D), collector dosage (E) and collector Type (F). The conditions of operating parameters are listed in Table 4. 64 experiments were designed using the central composite design method (Table 6).

**Table 4.** Levels of the operating parameters.

| Factor | Parameters | unit | Type | Level -1.68 | Low Actual | Mean | High Actual | Level +1.68 |
|---|---|---|---|---|---|---|---|---|
| A | pH | - | Numeric | 6.9 | 7.5 | 8.25 | 9.0 | 9.6 |
| B | d80 | Micron | Numeric | 44.0 | 53.0 | 64.0 | 75.0 | 84.0 |
| C | Solid Content | % | Numeric | 23.0 | 25.0 | 27.5 | 30.0 | 32.1 |
| D | $Na_2S$ dosage | g/t | Numeric | 2769.5 | 4000.0 | 5500.0 | 7000.0 | 8230.5 |
| E | Collector dosage | g/t | Numeric | 1089.8 | 1500 | 2000 | 2500 | 2910.2 |
| F | Collector Type | - | Categoric | - | PAX | 507 | - | - |

In each test, seven concentrates were taken via frothing in cumulative times 0.5, 1, 2, 4, 8, 12, 16 minutes. Each individual concentrate was weighed and analyzed separately, and ultimately, the cumulative recovery was obtained for each experiment. Finally, the results were determined for each test (Table 5).

**Table 5.** Flotation kinetics test result (Run 1) as sample.

| Products | time (min) | Wt. (%) | Pb (%) | | cumulative | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | | Assay | Distribution | Products | time (min) | Wt. (%) | Pb (%) Assay | Pb (%) Recovery |
| Feed (Cal) | 0 | 100.00 | 6.33 | 100.00 | (Cal) Feed | 0 | 100.00 | 6.33 | 0.00 |
| Concentrate1 | 0.5 | 4.76 | 47.09 | 35.45 | 1 | 0.5 | 4.76 | 47.09 | 35.45 |
| Concentrate2 | 0.5 | 3.57 | 26.41 | 14.91 | 1+2 | 1 | 8.34 | 38.23 | 50.36 |
| Concentrate3 | 1 | 4.76 | 18.12 | 13.64 | 1+2+3 | 2 | 13.10 | 30.92 | 64.00 |
| Concentrate4 | 2 | 5.86 | 9.22 | 8.53 | 1+2+3+4 | 4 | 18.96 | 24.21 | 72.53 |
| Concentrate5 | 4 | 5.74 | 4.83 | 4.38 | 1+2+3+4+5 | 8 | 24.70 | 19.71 | 76.91 |
| Concentrate6 | 4 | 3.73 | 3.77 | 2.22 | 1+2+3+4+5+6 | 12 | 28.43 | 17.62 | 79.13 |
| Concentrate7 | 4 | 4.07 | 2.60 | 1.67 | 1+2+3+4+5+6+7 | 16 | 32.50 | 15.74 | 80.80 |
| Tail | - | 67.50 | 1.80 | 19.20 | Tail | - | - | - | - |

Using the Sigma Plot 12 software and based on the experimental results, the model's parameters (responses) were extracted as shown in Table 3. These parameters were used as responses in Table 6.

### *2.3. Kinetics flotation model*

Based on the results from Sigma plot 12, a three-fraction (a seven-parameter) flotation model, with fast, medium, and slow kinetics constant was selected owing to the best agreement with the experiments' data. This model is much similar to the Jowett model that was presented in 1974. More than 100 models were fitted to the experiments' recovery values. Among them, the modified Jowett model had the highest correlation coefficient ($R^2$>99.92) with the recovery values. The model is shown in Eq. (1):

$$R=R\infty\left[Z_1\left(1-e^{k_1 t}\right)+Z_2\left(1-e^{k_2 t}\right)+Z_3\left(1-e^{k_3 t}\right)\right] \quad (1)$$

Where:

R= recovery in time t

R∞= ultimate Recovery

Z1= fast floatable fraction

Z2= medium floatable fraction

Z3= slow floatable fraction

K1= fast kinetics constant

K2= medium kinetics constant

K3= slow kinetics constant

t= time

And:

Z1+Z2+Z3= 100

## 3. Results and discussion

This new model had seven parameters influenced by the operational factors in the flotation process. Thus the effects of each operational factor and the interactions on the model parameters were investigated.

### *3.1. Optimization and ANOVA analysis of flotation results*

The analysis of variance (ANOVA) was used to investigate the effect of different laboratory parameters on model responses. ANOVA data considers the significance based on the ratio of variances according to the Fisher ratio of variances [74, 75]. The significance of the model depends on $F$ and $p$ values. Higher $F$ values and lower $p$ values ($p$<0.05) indicate the significance of the model at the confidence interval of 95% [74]. The values of $p$ and other parameters are shown in Table 7 for the significance determination of the responses.

In Table 7, Df is the degree of liberation, R2 is the correlation coefficient, and Adequate precision is the precision of the model that should be higher than 4.

**Table 6.** The results fo experiments designed by the CCD method.

| Run | Experimental parameters | | | | | | Model responses | | | | | | Recovery& Sum of Squares Errors | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | A | B | C | D | E | F | Z1 | Z2 | Z3 | k1 | k2 | k3 | R∞ | R | SS | $R^2$ |
| 1 | 9.0 | 75.0 | 25.0 | 4000.0 | 2500.0 | 507 | 21.88 | 59.77 | 18.36 | 3.76 | 0.95 | 0.09 | 84.78 | 80.8 | 0.0035 | 1.0000 |
| 2 | 8.3 | 44.0 | 27.5 | 5500.0 | 2000.0 | 507 | 10.91 | 71.24 | 17.85 | 17.83 | 0.96 | 0.11 | 79.22 | 76.6 | 0.1416 | 1.0000 |
| 3 | 6.9 | 64.0 | 27.5 | 5500.0 | 2000.0 | PAX | 1.78 | 7.08 | 91.14 | 3.91 | 0.66 | 0.001 | 88.76 | 75.1 | 0.7277 | 0.9999 |
| 4 | 8.3 | 64.0 | 27.5 | 5500.0 | 2000.0 | PAX | 30.55 | 29.85 | 39.60 | 0.78 | 0.78 | 0.02 | 93.48 | 76.1 | 3.2142 | 0.9994 |
| 5 | 8.3 | 64.0 | 27.5 | 2769.5 | 2000.0 | 507 | 35.56 | 34.75 | 29.69 | 0.97 | 0.97 | 0.04 | 92.83 | 77.7 | 0.6081 | 0.9999 |
| 6 | 8.3 | 64.0 | 27.5 | 5500.0 | 2000.0 | PAX | 33.72 | 30.96 | 35.33 | 0.83 | 0.83 | 0.14 | 79.20 | 76.1 | 0.9938 | 0.9998 |
| 7 | 7.5 | 53.0 | 30.0 | 7000.0 | 2500.0 | PAX | 22.81 | 57.33 | 19.86 | 4.13 | 0.97 | 0.09 | 82.47 | 78.5 | 0.0119 | 1.0000 |
| 8 | 9.0 | 75.0 | 30.0 | 7000.0 | 2500.0 | 507 | 43.80 | 26.47 | 29.73 | 1.47 | 0.27 | 0.27 | 82.74 | 82.2 | 5.8757 | 0.9991 |
| 9 | 8.3 | 64.0 | 27.5 | 5500.0 | 1089.8 | 507 | 6.27 | 66.67 | 27.06 | 18.38 | 1.04 | 0.16 | 80.25 | 78.7 | 0.4233 | 0.9999 |
| 10 | 7.5 | 75.0 | 25.0 | 4000.0 | 1500.0 | 507 | 35.55 | 33.39 | 31.06 | 1.40 | 1.40 | 0.17 | 81.08 | 79.5 | 0.8240 | 0.9998 |
| 11 | 7.5 | 53.0 | 25.0 | 4000.0 | 2500.0 | PAX | 37.00 | 35.89 | 27.11 | 0.98 | 0.98 | 0.18 | 77.40 | 76.1 | 0.2335 | 1.0000 |
| 12 | 9.0 | 75.0 | 25.0 | 7000.0 | 1500.0 | 507 | 41.33 | 41.08 | 17.59 | 1.24 | 1.24 | 0.11 | 86.30 | 83.4 | 2.2852 | 0.9996 |
| 13 | 8.3 | 44.0 | 27.5 | 5500.0 | 2000.0 | PAX | 8.45 | 66.00 | 25.54 | 30.48 | 0.76 | 0.13 | 78.08 | 75.5 | 0.0567 | 1.0000 |
| 14 | 7.5 | 75.0 | 30.0 | 4000.0 | 2500.0 | PAX | 10.33 | 61.91 | 27.76 | 30.48 | 0.75 | 0.16 | 78.98 | 77.2 | 0.0674 | 1.0000 |
| 15 | 8.3 | 64.0 | 27.5 | 5500.0 | 2910.2 | PAX | 13.45 | 67.29 | 19.26 | 21.36 | 0.81 | 0.15 | 78.39 | 76.9 | 0.0448 | 1.0000 |
| 16 | 9.0 | 53.0 | 25.0 | 7000.0 | 2500.0 | PAX | 15.38 | 66.83 | 17.80 | 2.72 | 0.53 | 0.04 | 88.59 | 80.0 | 1.1222 | 0.9997 |
| 17 | 9.0 | 53.0 | 30.0 | 7000.0 | 1500.0 | PAX | 9.37 | 70.54 | 20.10 | 3.01 | 0.76 | 0.14 | 82.21 | 80.3 | 0.2726 | 1.0000 |
| 18 | 9.0 | 53.0 | 30.0 | 4000.0 | 2500.0 | 507 | 53.06 | 28.19 | 18.76 | 1.46 | 0.50 | 0.11 | 81.64 | 78.7 | 0.3034 | 0.9999 |
| 19 | 8.3 | 64.0 | 27.5 | 8230.5 | 2000.0 | 507 | 31.78 | 27.08 | 41.14 | 1.23 | 1.23 | 0.20 | 76.49 | 75.1 | 0.5895 | 0.9999 |
| 20 | 9.0 | 53.0 | 25.0 | 4000.0 | 1500.0 | PAX | 15.33 | 59.38 | 25.29 | 47.51 | 1.07 | 0.23 | 79.85 | 78.9 | 0.0429 | 1.0000 |
| 21 | 9.0 | 75.0 | 25.0 | 7000.0 | 1500.0 | PAX | 7.58 | 28.53 | 63.89 | 3.41 | 0.56 | 0.00 | 89.46 | 82.4 | 0.0512 | 1.0000 |
| 22 | 7.5 | 53.0 | 30.0 | 4000.0 | 1500.0 | 507 | 4.87 | 51.41 | 43.72 | 7.31 | 0.99 | 0.21 | 78.44 | 77.4 | 0.0029 | 1.0000 |
| 23 | 8.3 | 64.0 | 27.5 | 5500.0 | 2000.0 | 507 | 35.10 | 33.46 | 31.44 | 0.94 | 0.94 | 0.18 | 78.42 | 77.1 | 0.3558 | 0.9999 |
| 24 | 8.3 | 64.0 | 23.0 | 5500.0 | 2000.0 | PAX | 52.17 | 31.49 | 16.34 | 0.87 | 0.34 | 0.08 | 80.24 | 76.4 | 1.6038 | 0.9996 |
| 25 | 7.5 | 75.0 | 30.0 | 4000.0 | 2500.0 | 507 | 28.34 | 45.35 | 26.31 | 1.77 | 0.53 | 0.04 | 90.71 | 78.2 | 2.9619 | 0.9995 |
| 26 | 8.3 | 64.0 | 27.5 | 5500.0 | 2000.0 | 507 | 33.45 | 31.08 | 35.48 | 0.91 | 0.91 | 0.16 | 79.36 | 77.1 | 5.5452 | 0.9992 |
| 27 | 8.3 | 64.0 | 27.5 | 5500.0 | 2000.0 | 507 | 35.59 | 33.80 | 30.61 | 0.82 | 0.82 | 0.10 | 82.51 | 77.1 | 0.8736 | 0.9998 |
| 28 | 8.3 | 64.0 | 27.5 | 5500.0 | 2000.0 | 507 | 54.24 | 42.50 | 3.26 | 1.04 | 0.18 | 0.03 | 81.50 | 77.1 | 0.5208 | 0.9999 |
| 29 | 7.5 | 75.0 | 30.0 | 7000.0 | 1500.0 | 507 | 36.03 | 36.09 | 27.87 | 1.09 | 0.66 | 0.13 | 83.59 | 80.9 | 0.6882 | 0.9999 |
| 30 | 8.3 | 64.0 | 27.5 | 5500.0 | 2000.0 | PAX | 2.85 | 66.99 | 30.15 | 3.86 | 0.75 | 0.11 | 80.27 | 76.1 | 0.2599 | 1.0000 |
| 31 | 8.3 | 64.0 | 27.5 | 5500.0 | 2910.2 | 507.0 | 5.57 | 67.44 | 26.99 | 3.66 | 0.84 | 0.15 | 79.91 | 78.0 | 0.0392 | 1.0000 |
| 32 | 8.3 | 64.0 | 27.5 | 5500.0 | 2000.0 | PAX | 47.45 | 50.38 | 2.17 | 1.30 | 0.25 | 0.03 | 78.22 | 76.1 | 0.2695 | 1.0000 |
| 33 | 7.5 | 53.0 | 25.0 | 7000.0 | 1500.0 | PAX | 33.69 | 20.35 | 45.95 | 1.65 | 0.45 | 0.00 | 77.28 | 78.7 | 0.8231 | 0.9998 |
| 34 | 8.3 | 64.0 | 27.5 | 5500.0 | 1089.8 | PAX | 34.13 | 31.41 | 34.47 | 1.25 | 1.25 | 0.13 | 81.82 | 78.1 | 1.6569 | 0.9996 |
| 35 | 9.0 | 53.0 | 30.0 | 7000.0 | 1500.0 | 507 | 32.63 | 28.44 | 38.94 | 1.13 | 1.13 | 0.13 | 85.49 | 81.3 | 0.2807 | 1.0000 |
| 36 | 8.3 | 64.0 | 32.1 | 5500.0 | 2000.0 | 507 | 23.69 | 27.84 | 48.47 | 2.02 | 0.78 | 0.18 | 78.75 | 76.5 | 0.0673 | 1.0000 |
| 37 | 7.5 | 53.0 | 30.0 | 7000.0 | 2500.0 | 507 | 40.31 | 25.61 | 34.08 | 2.02 | 0.46 | 0.15 | 81.11 | 78.5 | 0.0281 | 1.0000 |
| 38 | 9.0 | 53.0 | 25.0 | 7000.0 | 2500.0 | 507 | 58.09 | 17.00 | 24.91 | 2.44 | 0.63 | 0.16 | 82.74 | 81.1 | 0.0250 | 1.0000 |
| 39 | 9.0 | 75.0 | 30.0 | 4000.0 | 1500.0 | 507 | 15.44 | 47.10 | 37.45 | 15.76 | 1.23 | 0.21 | 82.09 | 81.1 | 0.7485 | 0.9998 |
| 40 | 8.3 | 64.0 | 27.5 | 5500.0 | 2000.0 | PAX | 16.18 | 65.08 | 18.74 | 5.97 | 0.95 | 0.10 | 78.89 | 76.1 | 0.0327 | 1.0000 |
| 41 | 7.5 | 75.0 | 30.0 | 7000.0 | 1500.0 | PAX | 9.63 | 49.81 | 40.55 | 15.20 | 1.22 | 0.16 | 82.41 | 79.8 | 0.4262 | 0.9999 |
| 42 | 9.0 | 75.0 | 30.0 | 7000.0 | 2500.0 | PAX | 9.79 | 22.08 | 68.14 | 3.53 | 0.69 | 0.003 | 84.78 | 81.1 | 0.0066 | 1.0000 |
| 43 | 7.5 | 75.0 | 25.0 | 4000.0 | 1500.0 | PAX | 21.71 | 50.49 | 27.80 | 3.63 | 0.71 | 0.02 | 82.05 | 78.4 | 0.2961 | 1.0000 |
| 44 | 8.3 | 64.0 | 32.1 | 5500.0 | 2000.0 | PAX | 9.15 | 64.53 | 26.32 | 6.52 | 0.90 | 0.12 | 78.71 | 75.5 | 0.0158 | 1.0000 |
| 45 | 7.5 | 53.0 | 30.0 | 4000.0 | 1500.0 | PAX | 38.00 | 37.04 | 24.96 | 1.91 | 0.53 | 0.05 | 86.51 | 76.3 | 0.4501 | 0.9999 |
| 46 | 8.3 | 64.0 | 27.5 | 5500.0 | 2000.0 | 507 | 36.10 | 34.48 | 29.41 | 1.08 | 1.08 | 0.15 | 78.13 | 76.1 | 0.3728 | 0.9999 |
| 47 | 7.5 | 53.0 | 25.0 | 7000.0 | 1500.0 | 507 | 18.08 | 60.52 | 21.40 | 4.39 | 0.78 | 0.13 | 82.11 | 79.7 | 0.0008 | 1.0000 |
| 48 | 9.6 | 64.0 | 27.5 | 5500.0 | 2000.0 | PAX | 5.45 | 67.57 | 26.98 | 11.34 | 1.13 | 0.15 | 81.18 | 78.2 | 0.0974 | 1.0000 |
| 49 | 8.3 | 64.0 | 27.5 | 5500.0 | 2000.0 | 507 | 47.36 | 23.49 | 29.16 | 1.19 | 0.19 | 0.19 | 79.40 | 77.1 | 0.4849 | 0.9999 |
| 50 | 9.0 | 53.0 | 30.0 | 4000.0 | 2500.0 | PAX | 38.59 | 36.35 | 25.05 | 1.48 | 1.48 | 0.20 | 78.55 | 77.6 | 3.4028 | 0.9994 |
| 51 | 8.3 | 84.0 | 27.5 | 5500.0 | 2000.0 | 507 | 9.14 | 13.16 | 77.70 | 2.01 | 0.69 | 0.002 | 77.84 | 77.5 | 1.0195 | 0.9997 |
| 52 | 8.3 | 64.0 | 27.5 | 2769.5 | 2000.0 | PAX | 39.21 | 29.87 | 30.92 | 1.38 | 0.65 | 0.14 | 79.40 | 76.6 | 0.1009 | 1.0000 |
| 53 | 7.5 | 53.0 | 25.0 | 4000.0 | 2500.0 | 507.0 | 6.80 | 55.37 | 37.83 | 17.42 | 0.72 | 0.16 | 79.63 | 77.1 | 0.0674 | 1.0000 |
| 54 | 8.3 | 64.0 | 27.5 | 5500.0 | 2000.0 | PAX | 76.04 | 2.32 | 21.65 | 1.43 | 1.27 | 0.14 | 78.43 | 76.1 | 0.3108 | 0.9999 |
| 55 | 8.3 | 64.0 | 27.5 | 8230.5 | 2000.0 | PAX | 33.01 | 25.78 | 41.21 | 1.22 | 1.22 | 0.13 | 78.44 | 74.1 | 0.7590 | 0.9998 |
| 56 | 7.5 | 75.0 | 25.0 | 7000.0 | 2500.0 | PAX | 22.62 | 40.18 | 37.20 | 3.70 | 0.80 | 0.15 | 82.45 | 79.6 | 0.0409 | 1.0000 |
| 57 | 8.3 | 64.0 | 23.0 | 5500.0 | 2000.0 | 507 | 6.68 | 50.58 | 42.74 | 6.76 | 1.05 | 0.17 | 79.73 | 77.5 | 0.0366 | 1.0000 |
| 58 | 7.5 | 75.0 | 25.0 | 7000.0 | 2500.0 | 507 | 34.72 | 32.00 | 33.29 | 1.42 | 1.42 | 0.15 | 83.54 | 80.6 | 0.6679 | 0.9998 |
| 59 | 9.6 | 64.0 | 27.5 | 5500.0 | 2000.0 | 507 | 33.28 | 29.78 | 36.94 | 1.45 | 1.45 | 0.14 | 85.28 | 81.6 | 0.1284 | 1.0000 |
| 60 | 9.0 | 75.0 | 30.0 | 4000.0 | 1500.0 | PAX | 47.00 | 28.31 | 24.68 | 1.83 | 0.51 | 0.13 | 82.75 | 80.0 | 0.4557 | 0.9999 |
| 61 | 6.9 | 64.0 | 27.5 | 5500.0 | 2000.0 | 507 | 7.61 | 54.42 | 37.97 | 2.57 | 1.02 | 0.14 | 79.51 | 76.2 | 0.1641 | 1.0000 |
| 62 | 8.3 | 84.0 | 27.5 | 5500.0 | 2000.0 | PAX | 19.27 | 46.42 | 34.32 | 5.30 | 1.51 | 0.20 | 77.68 | 76.4 | 0.0748 | 1.0000 |
| 63 | 9.0 | 75.0 | 25.0 | 4000.0 | 2500.0 | PAX | 28.28 | 33.63 | 38.09 | 3.01 | 0.80 | 0.18 | 81.59 | 79.8 | 0.0173 | 1.0000 |
| 64 | 9.0 | 53.0 | 25.0 | 4000.0 | 1500.0 | 507 | 14.47 | 44.40 | 41.13 | 6.78 | 1.23 | 0.19 | 81.66 | 79.9 | 0.2324 | 1.0000 |

As seen in Table 7, pH and particle size are rather affecting K1 and Z1than the others. Also, pH is more effective on K2, particle size on Z2 and the Na2S dosage at R∞. As it is shown in Table 7, the interaction of some parameters is effective on K1, K2, K3, Z1, Z2, Z3 and R∞. For example, the interaction between pH-collector type, the dosage of Na2S-collector type, pH-pH, the dosage of Na2S-dosage of collector-collector type, pH-pH-dosage of Na2S, pH-pH-d80-d80 and pH- d80-d80-collector type are significant factors that mostly affect the parameters at R∞.

Since an inadequate model could lead to misleading results, the validation of the model is an essential part of the data analysis procedure. The adequate precision ratio indicates that the precision of obtained data should be higher than 4 [1, 76, 77]. The adequate precision ratio of data for all responses was obtained more than 4, indicating the high precision of the presented model.

The correlation coefficient (R2) was obtained more than 0.8, which shows an appropriate agreement between predicted data and real ones.

**Table 7.** The analysis of variance for responses by a quadratic model.

| K1 | | K2 | | K3 | | Z1 | | Z2 | | Z3 | | R∞ | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **Source** | **p-value (Prob>F)** | **Source** | **p-value (Prob>F)** | **Source** | **p-value (Prob>F)** | **Source** | **p-value (Prob>F)** | **Source** | **p-value (Prob>F)** | **Source** | **p-value (Prob>F)** | **Source** | **p-value (Prob>F)** |
| Model | < 0.0001 | Model | 0.0441 | Model | 0.0421 | Model | 0.0383 | Model | 0.0493 | Model | 0.0126 | Model | 0.0398 |
| A | 0.0293 | A | 0.0321 | DF | 0.0088 | CF | 0.021 | B | 0.0071 | A | 0.0043 | D | 0.0073 |
| B | < 0.0001 | AB | 0.0435 | ABF | 0.0150 | A2 | 0.0006 | AF | 0.0052 | B | 0.0029 | AF | 0.0328 |
| AB | < 0.0001 | BF | 0.0187 | ADF | 0.0255 | B2 | 0.0032 | CF | 0.0433 | AF | 0.0054 | DF | 0.0187 |
| AC | < 0.0001 | CF | 0.0063 | DEF | 0.0023 | E2 | 0.005 | E^2 | 0.0101 | BF | 0.0199 | $A^2$ | 0.0330 |
| AD | 0.0006 | $A^2$ | 0.0351 | $A^2D$ | 0.0238 | ABF | 0.0215 | ABF | 0.0013 | $A^2$ | 0.0009 | DEF | 0.0127 |
| AE | < 0.0001 | $B^2$ | 0.1107 | $B^2F$ | 0.0231 | $A^2F$ | 0.0361 | A $B^2F$ | 0.0303 | $B^2$ | 0.0339 | $A^2D$ | 0.0018 |
| AF | 0.0050 | DEF | 0.0365 | - | - | $A^2B^2$ | 0.0006 | - | - | $A^2F$ | 0.0087 | $A^2$ $B^2$ | 0.0456 |
| BC | < 0.0001 | $A^2D$ | 0.0306 | - | - | - | - | - | - | A $B^2$ | 0.0125 | A $B^2F$ | 0.0363 |
| BD | 0.0004 | $AB^2$ | 0.0902 | - | - | - | - | - | - | $A^2$ $B^2$ | 0.0191 | - | - |
| BE | < 0.0001 | $A+B^2$ | 0.0422 | - | - | - | - | - | - | $A^2BF$ | 0.0016 | - | - |
| BF | 0.0026 | $A^2BF$ | 0.0047 | - | - | - | - | - | - | A $B^2F$ | 0.0045 | - | - |
| CD | 0.0004 | $A^2EF$ | 0.0436 | - | - | - | - | - | - | - | - | - | - |
| CE | 0.0003 | - | - | - | - | - | - | - | - | - | - | - | - |
| CF | 0.0011 | - | - | - | - | - | - | - | - | - | - | - | - |
| DE | 0.0467 | - | - | - | - | - | - | - | - | - | - | - | - |
| EF | < 0.0001 | - | - | - | - | - | - | - | - | - | - | - | - |
| $A^2$ | 0.0004 | - | - | - | - | - | - | - | - | - | - | - | - |
| $B^2$ | < 0.0001 | - | - | - | - | - | - | - | - | - | - | - | - |
| $C^2$ | 0.0038 | - | - | - | - | - | - | - | - | - | - | - | - |
| $E^2$ | < 0.0001 | - | - | - | - | - | - | - | - | - | - | - | - |
| Lack of fit | 0.8203 | Lack of fit | 0.9949 | Lack of fit | 0.9693 | Lack of fit | 0.9973 | Lack of fit | 0.9925 | Lack of fit | 0.9822 | Lack of fit | 0.9998 |
| $D_f$ | 46 | $D_f$ | 48 | $D_f$ | 46 | $D_f$ | 42 | $D_f$ | 43 | $D_f$ | 44 | $D_f$ | 41 |
| $R^2$ | 0.9935 | $R^2$ | 0.8779 | $R^2$ | 0.8543 | $R^2$ | 0.8050 | $R^2$ | 0.8105 | $R^2$ | 0.8589 | $R^2$ | 0.7903 |
| Adequate Precision | 41.099 | Adequate Precision | 6.247 | Adequate Precision | 6.950 | Adequate Precision | 6.161 | Adequate Precision | 5.324 | Adequate Precision | 9.268 | Adequate Precision | 6.782 |

### *3.2. Individual and interaction effects of the parameters*

If the numerical difference between the optimum amount and the amount used in the experiments for each parameter is low, the interaction will have a more significant effect on the flotation.

In Figure 2 (a to l), individual effects and interactions of parameters on K1 are shown. In figure 2.

Figure 2. (a) shows the interaction effect between pH-d80 on K1. As seen, at the smaller particle size and medium pH, K1 is maximum. pH has a direct and d80 has a reverse effect on K1. HS- ions adhere to the surface of the cerussite properly and activate it. For higher and lower values of pH, respectively, S-2 and H2S are the dominant species of S. Therefore, in an average alkaline pH, there is the highest amount of HS- [78]. Also in this study, with increasing pH, the flotation rate will be increased due to the maximum value of S in the form of HS- and faster absorption on cerussite. By increasing the particle size, the degree of liberation and the specific surface are both reduced. Therefore, the absorption of chemicals on cerussite will be decreased, which will reduce K1. As particle size decreases due to the interaction between these two parameters and the higher effectiveness of pH, K1 is reduced as well. The highest values of K1 occurred for small-sized particles and moderate pH values.

Figure 2 (b to e) shows the interaction effect between pH and the solid content, Na2S, as well as the type and dosage of the collector. The maximum quantity of K1 can be obtained for high pH values and low solid contents, low Na2S dosages, low collector dosages, and by using PAX, respectively.

In figure 2(b), for low percentages of solids content, the result is the same as (a). However, the condition is different for high solids content percentages. With increasing the solid content, pulp turbulence and collisions increases, and subsequently, particle backload occurs.

In figure2 (c), K1 increases by decreasing the dosage of sodium sulfide. By increasing the dosage of sodium sulfide, the sulfur ions are preferably absorbed onto the collector, and K1is reduced[79].

In figure 2 (d), by increasing the collector dosage and forming micelles, it loses some properties, which is not suitable for flotation.

In figure 2 (e), when collector 507 was used, different values of pH did not change K1, but by using collector PAX, and K1 increased as pH increased.

Figure 2. (f to h) shows the interaction effect between particle size and solid content, Na2S, and collector dosage. The maximum quantity of K1 can be obtained in low values of particle size and other parameters are not so effective.

In figure2 (g), the effect of particle size is very significant, and different dosages of sodium sulfide have an insignificant effect on K1.

In figure 2(h), the conditions are similar to (g). At particle size 75 microns, as the dosage of the collector decreases, K1 reduces as well, which could be because of inadequate collector dosage.

Figure 2. (i and j) shows the interaction effect between the solid content and the collector dosage as well as the collector type. The maximum quantity of K1 can be obtained at a solid content of %30; other parameters, however, are not affected by this factor.

Figure 2 (i) shows that as the collector dosage increases, K1 increases, which suggests the presence of a sufficient amount of the collector. Figure 2 (j) shows that the use of 507, instead of PAX, would increase the solid content percent which results in decreasing K1.

Figure 2. (k) presents the interaction effect between the collector dosage and Na2S type. The maximum quantity of K1 can be obtained at high values of both factors but with slight effectiveness, which can be due to the appropriate dosage of these two parameters.

Figure2. (l) shows that the maximum quantity of K1 can be obtained at high PAX dosages or low 507 dosages. When 507 is used, due to the simultaneous use of the two collectors, K1 decreases, but the result is differed when PAX is used. Also, lesser dosages of 507 would form a critical micelle.

In summary, the effective parameters on the maximum quantity of K1

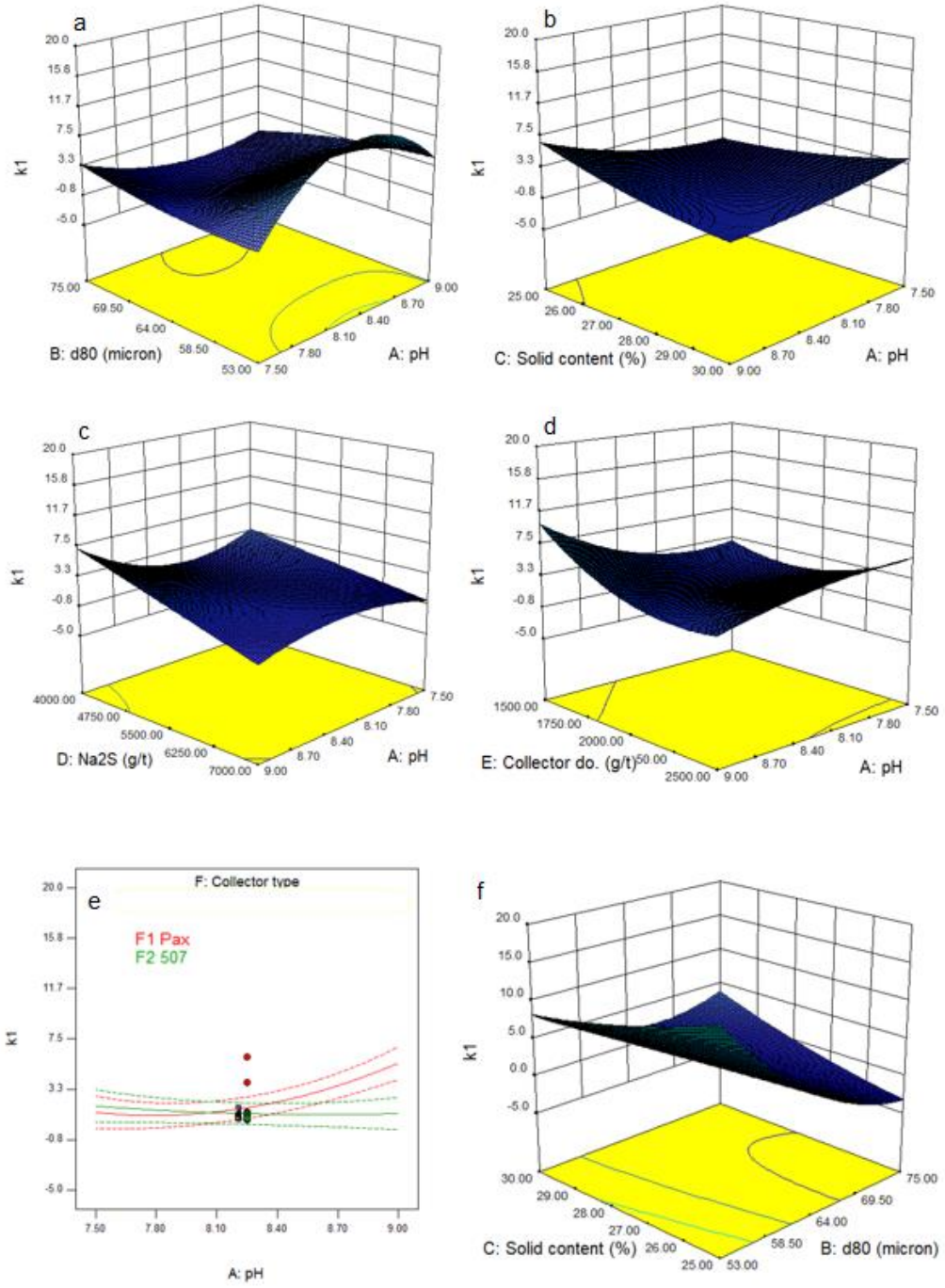
happened at low particle size values, high pH values, high solid content values, the use of PAX, high collector dosages and $Na_2S$.

From figure 3. (a) can see that as pH increases, K2 increases subsequently similar to K1. In figure 3. (b), the interaction effect between particle size and pH as well as the maximum quantity of K2 can be obtained at average values of both factors.

By decreasing the particle size due to increasing the degree of liberation, the flotation process is improved, but the excessive reduction of particles would result in increasing fine particles, and subsequently, it would not be suitable for the flotation process. On the other hand, by increasing the value of pH, the collector is hydrolyzed and loses its properties. At low pH values, however, it precipitates on the surface of cerussite and prevents the connection between the bubble and cerussite [78]. Accordingly, the highest K2 value is obtained at mean pH and particle size values.

In figure 3. (b, c), the interaction effect between the collector type and the particle size as well as the solid content and the maximum quantity of K2 can be obtained at high particle sizes and solid contents in the presence of PAX.

Therefore, the effective parameters on the maximum quantity of K2 would be high pH values, high particle size values and the use of PAX.



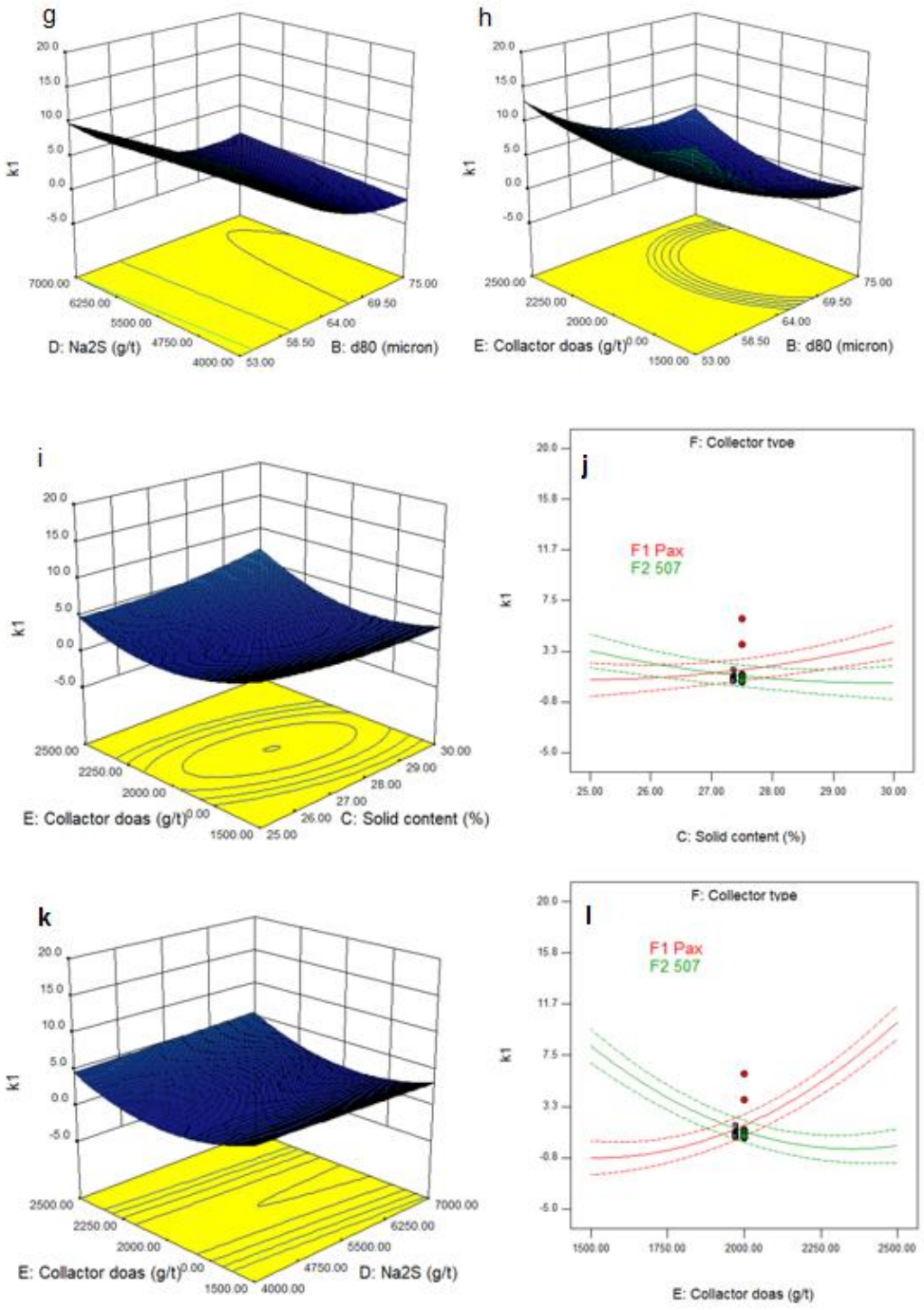



**Fig. 2.** The effect of different parameters on the fast kinetics rate constant (Other parameters were held at the central level).

Figure 4 shows the only interaction effect between $Na_2S$ and the collector type that affects $K_3$. The maximum quantity of $K_3$ can be obtained at high $Na_2S$ dosages in the present of 507. This can be due to the presence of a sufficient amount of sodium sulfide. On the other hand, the use of 507, which is a combination of two different collectors, can float more ore varieties.

Figure 5 also shows the only interaction effect between solid content and the collector type that effect on Z1. The maximum quantity of Z1 can be obtained at high solid content values in the presence of 507 and low solid content values in the presence of PAX.

As the percentage of solid content and the probability of particles that collide with bubbles and chemicals increase, the use of 507 (two collectors) and the possibility of particle flotation with a rate of $K_1$ increase.

Figure 6 shows the effect and interaction of parameters on $Z_2$. As the figure shows, when the particle size increases, $Z_2$ is reduced. For the interaction between the collector type with pH and solid content, in the presence of PAX, the maximum quantity of $Z_2$ can be obtained at high pH and solid content values and in the presence of 507, their values are vice versa.

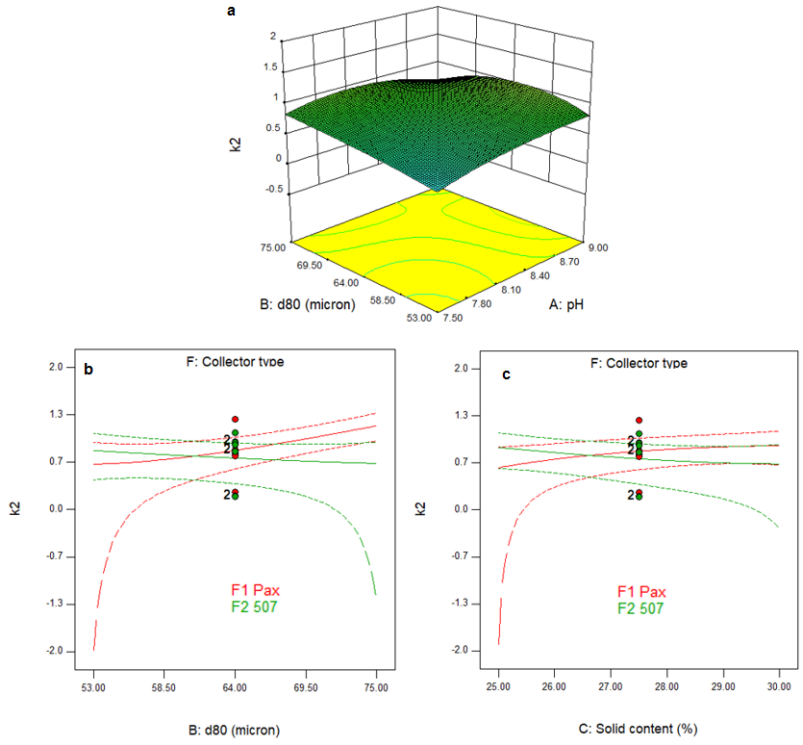



**Fig. 3.** The effect of different parameters on the medium kinetics rate constant (Other parameters were held at the central level).

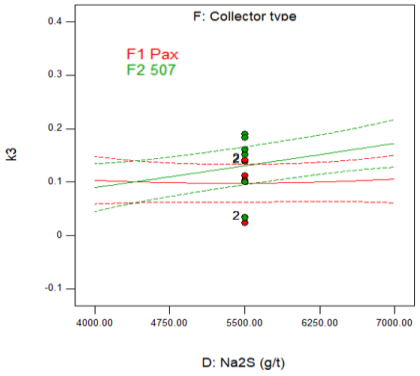



**Fig. 4.** The effect of different parameters on the slow kinetics rate constant (Other parameters were held at the central level).

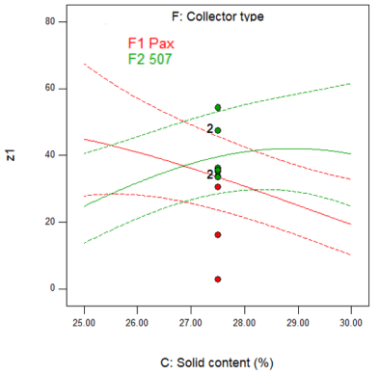



**Fig. 5.** Effect of different parameters on the fast floatable fraction(Other parameters are held at the central level).

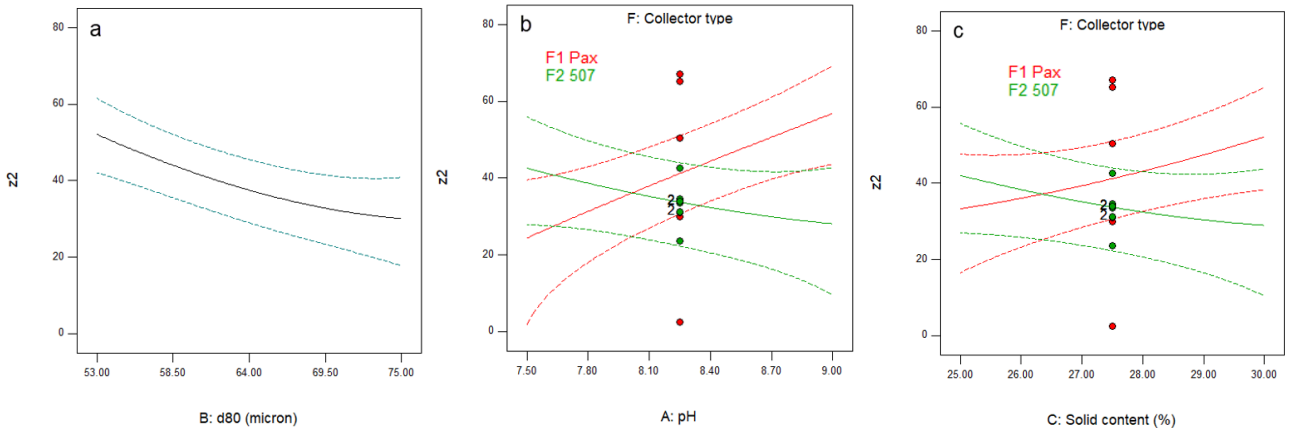



**Fig. 6.** The effect of different parameters on the medium floatable fraction (Other parameters were held at the central level).

The interaction effect of parameters on $Z_3$ are shown In figure 7. As seen, with the increase of particle size, $Z_3$ is increased and with an increase of pH, $Z_3$ is reduced. In the interaction between collector type with pH and particle size, in the presence of PAX, the maximum quantity of $Z_3$ can be obtained in a low value of pH and in the presence of 507, pH is not an effective factor. On the other hand, in the presence

of 507, the maximum quantity of $Z_3$ can be obtained at high particle size values, and in the presence of PAX, particle size is not an effective factor.

Figure 7 (a) shows that by increasing the value of pH, the percentage of particles that float with a rate of $K_3$ reduces. These materials will float at $K_1$ and $K_2$ rates. This is due to the maximum amount of S as HS by increasing pH and more absorption of HS on the surface of cerussite.

It is concluded from Figures 6 (a) and 7 (b) that by increasing the particle size, $Z_2$ is reduced and added to $Z_3$. This can be due to the increase in the particle size, which reduces the specific surface, and finally decreases bubble collision.

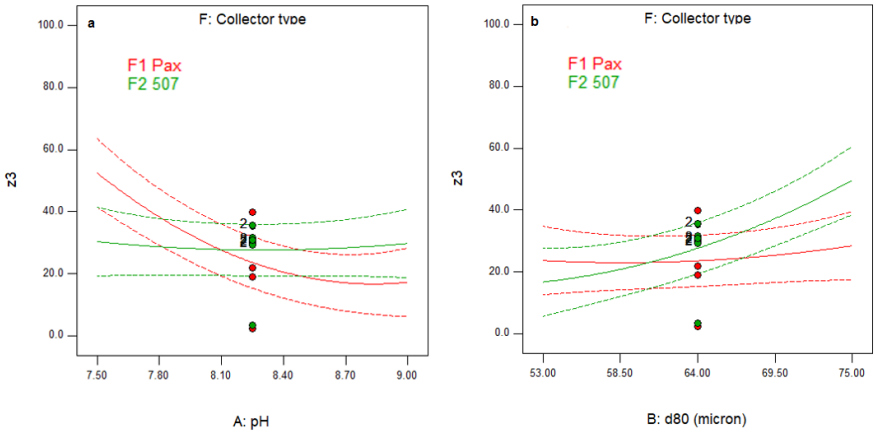



**Fig. 7.** Effect of different parameters on the slow floatable fraction(Other parameters were held at the central level).

Figure 8 shows the interaction effect of the parameters at $R_\infty$. As $Na_2S$ increases, R∞ reduces in the interaction between collector type with pH and Na2S, in the presence of PAX. The maximum quantity of R∞ can be obtained at low pH values and in the presence of 507, pH is inverse. On the other hand, in the presence of 507, the maximum quantity of R∞ can be obtained at low Na2S dosages, and in the presence of PAX, the dosage of Na2S is not effective.

In Figure8 (b), the final recovery was reduced by increasing the dosage of sodium sulfide. This is due to the tendency of excess sodium sulfide in the pulp to absorb the collector and also the property of soluble sulfur ions as the depressant. In Figure 8 (a), pH has a positive effect using 507, and a negative effect using PAX. In Figure 8 (b), when PAX is used, changing the dosage of sodium sulfide does not affect the final recovery, but when 507 is used, as the sodium sulfide content increases, the final recovery reduces, as shown in Figure 8 (b).

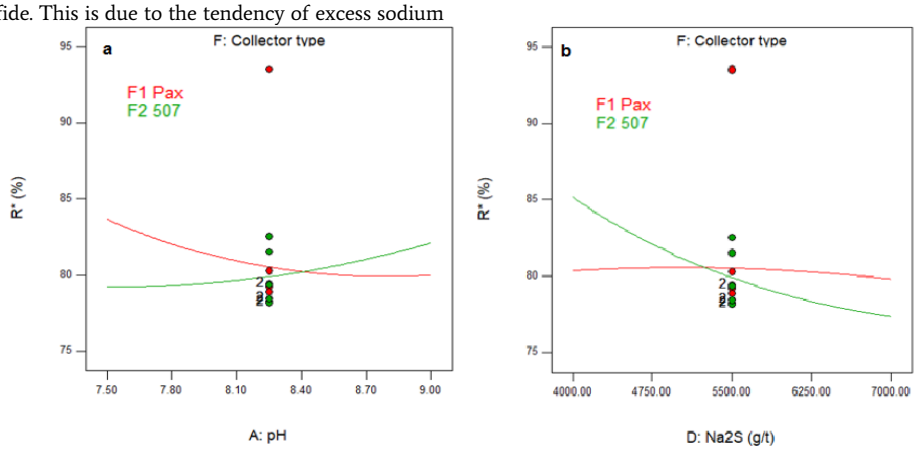



**Fig. 8.** The effect of different parameters on ultimate Recovery (Other parameters were held at the central level).

### *3.3. Perturbation graphs*

The perturbation graphs of each response are shown in figure 9. In these figures, the steeper the slopes of each diagram, the more effective the parameter. Therefore, the most effective parameter on $K_1$ is particle size, the least effective parameter on $K_2$ is solid content, the most effective parameters on $K_3$ are pH and particle size, and the most effective parameter on $R_\infty$ is the dosage of $Na_2S$.

### *3.4. The proposed model parameters*

The kinetics parameters of lead flotation related to the effective parameters are as equations (2) to (8).

K1= +1.56 +0.87A -5.63B -1.61AB -2.20AC -0.98AD -2.69AE -1.17AF +3.47BC +1.02BD+1.46BE+1.28BF+1.03CD +1.05CE -1.43CF +0.50DE -4.78EF +0.98A2 +3.73B2 +0.75C2 +2.90E2 +3.85ABF +3.33ACF +0.82ADF +1.18AEF -1.51BCF -0.68BDF -2.38BEF -1.08CDF -2.91CEF +5.20A2B -2.95A2D -1.85A2E -0.67A2F -3.70A2B2 -1.80A2BF +1.37A2CF +5.43A2EF +0.94AB2F (2)

(K2)2= +0.67 +0.26 A -0.16 AB -0.29 BF -0.19 CF +0.16 A2 +0.12 B2 +0.16 DEF -0.32 A2D -0.24 AB2 -0.37 A2B2 +0.44 A2BF -0.29 A2EF (3)

K3= +0.11 +0.020DF +0.022ABF +0.020ADF +0.029DEF -0.037A2D -0.017B2F (4)

(Z1)0.36= +3.56+0.42CF -0.42A2 -0.35B2 -0.33E2 -0.27ABF +0.21A2F +0.87A2B2 (5)

(Z2)1.25= +94.62-34.18 B -35.77 AF -24.62 CF +20.55 E2 (6)

+27.42 ABF +31.64 AB2F

Z3= +25.58 -8.95A +9.42B +8.67AF +7.02BF +6.84A2 +4.00B2 -4.44A2F +9.05AB2 -10.51A2B2 - 12.05A2BF -10.56AB2F (7)

(R∞)-3= +1.948×10-6 +1.453×10-7D -1.121×10-7AF +1.249×10-7 DF -6.733×10-8 A2 +8.590×10-8 DEF - 2.071×10-7 A2D -1.200×10-7 A2B2 +1.304×10-7 AB2F (8)

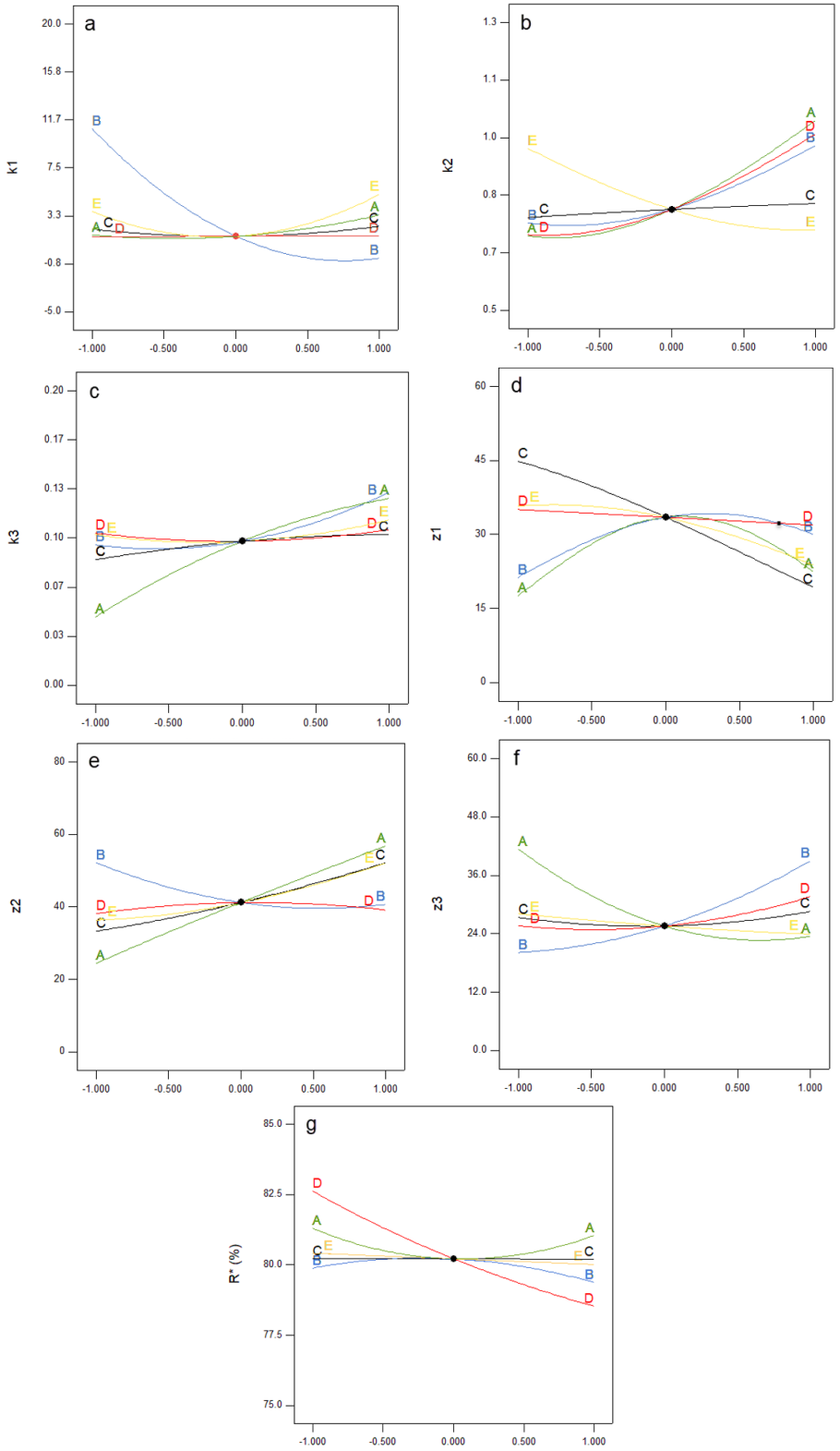



**Fig. 9.** Perturbation graph of each response (A: pH, B: $d_{80}$, C: solid content, D: $Na_2S$ and E: collector dosage, all parameters were held at the central level).

### 3.5. Optimum conditions of the experiment

In the surface response method, the maximum and minimum conditions of responses can be determined with high accuracy using the statistical approach and optimization at the designing space [73-75]. The first differentiation of Eq. (2-8), which was determined with ANOVA, presents the maximum $K_{1,2,3}$ and $R_\infty$ of Pb. The optimum conditions are summarized in Fig. 9. Based on the experimental design outputs, the optimum conditions of pH: 9, $d_{80}$:53 µm, solid content: 26%, $Na_2S$: 4000g/t and collector: 1500g/t of PAX, resulted in maximum $K_{1,2,3}$ and ultimate recovery (Table 8). These conditions were tested as the validation test in the laboratory and the results showed only a 3.5% deviation from the prediction.

**Table 8.** Optimum conditions of the experiments.

| Ex. Parameter | Best Condition | Model Parameter | Best Result |
|---|---|---|---|
| pH | 9.0 | k1 | 38.30 |
| $d_{80}$ | 53.0 | k2 | 1.06 |
| Solid Content | 26.3 | k3 | 0.24 |
| $Na_2S$ | 4000.0 | z1 | 24.0 |
| Collector Dosage | 1500.0 | z2 | 55.0 |
| Collector Type | PAX | z3 | 21.0 |
| - | - | $R_\infty$ | 80.43 |

## 4. Conclusions

In this paper, a seven-parameter kinetics model was proposed for the recovery prediction of cerussite flotation. The effect of flotation parameters on the model parameters was investigated. The effects of operating parameters such as pH, solids content, particle size, $Na_2S$ dosage, collector dosage, and collector type were studied and the optimization was conducted through the response surface methodology based on the central composite design (CCD) model. A three-fraction (with seven-parameter) flotation model, with fast ($K_1$), medium ($K_2$) and slow ($K_3$) kinetics rate constants was obtained through 64 designed tests. The proposed model showed a good agreement with the experimental data ($R^2$ more than 0.8). Based on the analysis of variance, the most effective parameter on $K_1$ is particle size, the least effective parameter on $K_2$ is solid content, the most effective parameters on $K_3$ are pH and particle size, and the most effective parameter on $R_\infty$ is the dosage of $Na_2S$. The optimum conditions were achieved as follows: pH: 9, $d_{80}$: 53 µm, solid content: 26%, $Na_2S$: 4000g/t and collector: 1500g/t of PAX, resulted in maximum $K_{1,2,3}$ and ultimate recovery.

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