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Modeling and Optimization of water recovery the sludge from beneficiation process of phosphate ore using Experimental Design Methodology

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Abstract

The objective of this work is to present modeling and optimization of water recovery from the sludge beneficiation process of phosphate ore using experimental design methodology. This sludge is produced from a process of beneficiation of a phosphate ore. By a screening design (Hadamard Matrix), we evaluated the effect of five parameters on this rate. The data analyses of this screening design showed that only three parameters have an effect on this rate. By a Box-Behnken design, we were able to find the values of these three parameters to achieve until 49% of water. This result has a great impact both economic and environmental.

1. Introduction

Most industrial processes consume and discharge a lot of water. However, mineral processing treatments considered as the largest water users large water users. Water has become scarce in lately years. All national and international organizations insist on the fact that it should be preserved.

On the other hand, the beneficiation processes of phosphate ores by washing and floating uses a lot of water that generates huge quantities of sludge. This sludge is stored in ponds. By decantation, a quantity of water can be recovered. However, decanting is a too slowly process too slowly. In addition, the presence of fine particles slows down the decantation. This causes serious problems:

1. Little water recovery.
2. Large area to store sludge.

Several studies focused on this theme [1-3]. Some methods were tested: ultrasonic [4-5], thermal [6-7], biological [8] and acid and alkaline treatment [9-10]. Some authors [11-14] used the combination of two or more of the previously methods for to improving sludge water recovery.

It should be noted that coagulation process followed by flocculation are the most used [15-19].In this regard several works [17-21] were carried out. They treated the impact of several process parameters (coagulant, flocculent, pH, nature and quantity of ferric and aluminum salts ...). However, all these studies launched on the sludge, don't focus on the one generated by phosphate ores treatment. Moreover these studies only worked with one or two parameters using one-factor-at-a-time approach (OFAT). Besides that, OFAT generally requires more experimental runs, has less accuracy and is not able to estimate factor interactions when compared with designed experiments

As far as we are concerned, we will evaluate the effect of several parameters on the process of recovery of washing and flotation water from a phosphate ore beneficiation process. Then we will look for parameters considered influential for a maximum recovery.In order to better organize our experiments, we use the design of experiment.

2. Experimental details

2.1.*Sludge and flocculent*

Sludge:

We used sludge gotten from a washing and flotation water from phosphate ore beneficiation process. The sludge

total solid was about 30%.
Flocculent:
We used in this study a commercial anionic polyelectrolyte..

2.2 *Design of experiment*
Traditionally, a scientist carries out experiments way by varying the parameters the ones after the others. This method gives results but is very expensive in time because it inevitably requires the realization of a great number of experiments. This is why it is important to help the scientist to achieve his experiments with design of experiment (DOE). By this mythology, the scientist knows how to plan experiments. The experimental step will help him to structure his research in a different way, to valid his own assumptions, with better understanding the study phenomena, and to solve the problems. Therefore, in this study the experiments will be conducted using design of experiment. In the current study, the ranges of input parameters are selected as in table 1:

Table 1: Factors and their levels for screening design

Factors		Levels	
		Low level (-1)	**Up level (+1)**
X_1	Stirring time (s)	30	120
X_2	Flocculant amount (g/t)	300	500
X_3	Flocculant concentration (g/l)	0.4	1
X_4	Stirring type (speed)	Low	High
X_5	Add Flocculant	Fractional	Full

Parameters number submitted to this study is five. It seems wise to start with a screening design (Plackett & Burman) [22-27]. Screening design was employed to screen factors that may have significant effects on response(s) because it is the most efficient and available method to carry out multifactor experiments. The significant factors can then be used to develop a model to optimize and predict the response, if needed. The regression model (Eq. (1)) of a screening design can be written as:

$$Y = a_0 + \sum_{i=1}^{n} a_i X_i \quad (1)$$

Where Y is the response, a_0 is the mean of all treatment combinations, a_i is half of the effect estimated corresponding to significant effects, Xi are coded variables that represent significant effects and take on values between -1 and +1.
In this work, we are interested in water recovery rate from washing and flotation sludge of a phosphate ore beneficiation process. The response is the water recovery rate. The screening design chosen for the current study requires 8 experiments for 5 factors. We summarized in table 2 the experimental design and the response after we carried out the experiments.

3. Resulats and Discussion

3.2.Screening design
All the experiments that were carried out in the experiment design showed in table 2flollow the same procedure, under the operating conditions indicated by the coding scheme (high and low levels of each factor). Data analysis provided us factors which are statistically significant to the water recovery rate. The results of the experiments allowed us to develop the regression model describing the interrelationship between operational variables and response by equations including linear terms. The regression coefficients are presented below :

$$\mathbf{Y = 28.11 + 3.85\, X_1 + 4.36\, X_2 + 3.06\, X_3 + 1.91\, X_4 - 1.48\, X_5}$$

Data analysis the screening design, represented in the figure 1, led us to the following conclusions:

- As shown in this figure 1 three variables: Stirring time and Flocculant amount and Flocculant concentration, were significant factors with positive effect.
- Stirring type and Add Flocculant were non-significant factors.

The figure 2 illustrates the standardized Pareto charts ($P < 0.05$) of main effects. Pareto analysis confirms the previous conclusions.

Table 2 : Screening design and response

Exp n°	X1 Stirring time (s)	X2 Flocculant amount (g/t)	X3 Flocculant concentration (g/l)	X4 Stirring type (speed)	X5 Add Flocculant	Y Water recovery rate (%)
1	120	500	1	Low	Full	36.58
2	30	500	1	High	Fractional	32.83
3	30	300	1	High	Full	25.63
4	120	300	0.4	High	Full	22.75
5	30	500	0.4	Low	Full	21.60
6	120	300	1	Low	Fractional	29.66
7	120	500	0.4	High	Fractional	38.88
8	30	300	0.4	Low	Fractional	16.99

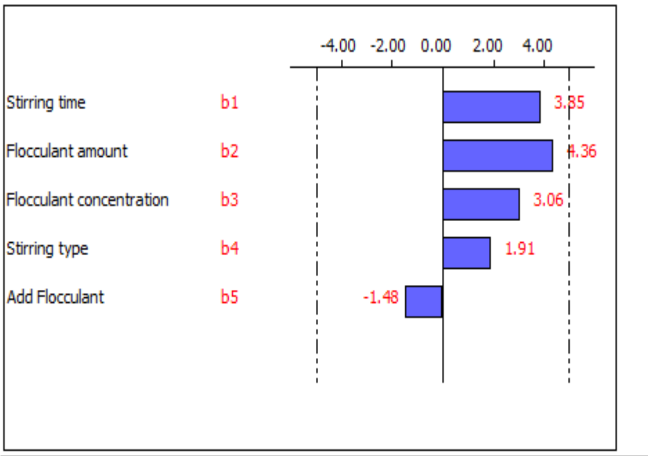

Figure1 : Effects graph

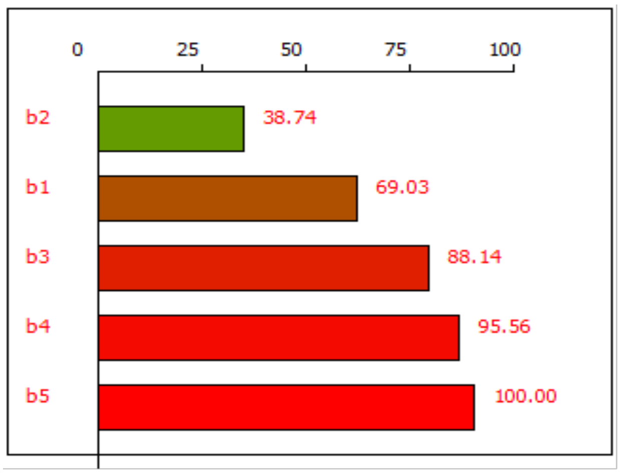

Figure 2 : Pareto chart

Through the investigation of the data obtained from screening design, Stirring time and Flocculant amount and Flocculant concentration were the significant variables for water recovery rate of from the washing and flotation sludge of an ore enrichment process of phosphate. Due to non-significant effect of other variables on the response, Stirring type and Add Flocculant were fixed to high speed and fractional add or the next stage of response surface methodology.

3.2.Optimization of significant variables by response surface methodology

A Three-level central composite design (CCD) was employed to investigate the interactions between the significant experimental factors and to identify their optimal values and represented in table 3.

Table 3: Values natural variables of the Central Composite design.

Factors		Levels	
		Low level (-1)	**Low level (-1)**
X_1	Stirring time (s)	30	120
X_2	Flocculant amount (g/t)	300	500
X_3	Flocculant concentration (g/l)	0.4	1

Central composite design (CCD) as branch of response surface methodology (RSM) has high efficiency for optimization variables and draws a mathematical equation, which correlates the response to the variables as sole and interaction. The CCD was established in three levels (-1, 0, +1) for each significant factor. The upper and lower values for each factor were set as in the screening design. The response surface regression procedure using the second order polynomial (Eq. (2)) was used to analyze the experimental data obtained:

$$Y = a_0 + \sum_{i=1}^{n} a_i X_i + \sum_{i=1,j>i}^{n} a_{ij} X_i X_j + \sum_{i=1}^{n} a_{ii} X_i^2 \quad (2)$$

Several works have used the response surface methodology[28-30] to treat the industrial sludge.

We chose the Box-Behnken design. We summarized in table 4 the experiment plan and measured the results after the tests were carried out

Table 4: Experiment plan and measured results.

Exp n	X_1 Stirring time (s)	X_2 Flocculant amount (g/t)	X_3 Flocculant concentration (g/l)	Y Water recovery rate (%)
1	30	300	0.7	**33.70**
2	120	300	0.7	**41.76**
3	30	500	0.7	**44.93**
4	120	500	0.7	**48.96**
5	30	400	0.4	**38.02**
6	120	400	0.4	**46.08**
7	30	400	1	**37.44**
8	120	400	1	**45.22**
9	75	300	0.4	**33.41**
10	75	500	0.4	**40.61**
11	75	300	1	**31.39**
12	75	500	1	**43.49**
13	75	400	0.7	**40.32**
14	75	400	0.7	**42.91**
15	75	400	0.7	**41.47**
16	75	400	0.7	**42.63**

The second order model can be expressed as a function of the process parameters (Stirring time, Flocculant amount and Flocculant concentration) and Water recovery rate. The relationship is defined between the Water recovery and the process parameters.

We have established a mathematical model between the water recovery rate and the process parameters using the experimental results. Using the response surface methodology, Water recovery rate model (with uncoded levels) is shown in the following equation:

$$\mathbf{Y = 41.83 + 3.49X_1 + 4.72X_2 - 0.07X_3 + 2.49X_1^2 - 1.98X_2^2 - 2.63X_3^2 \quad (3)}$$

In those kinds of studies, before use this model, a statistical validation is required. The validation process is based on the following four tests:

- ✓ Normal Probability Plot;
- ✓ Analysis of variance ANOVA ;
- ✓ Coefficient of determination R^2 ;
- ✓ Adjusted coefficient of determination R^2_{ajs}.

The residue graph, representedin figure 3, clearly shows that the residue line up a straight. Therefore, that plot indicates data are normally distributed data.

In order to test and judge the adequacy and goodness of fit of mathematical models, the ANOVA technique was used to evaluate the significance of the mathematical models and the process variables involved at 95% confidence interval. It can be seen from the table 5 that the probability values (P -value) for the model are smaller than 5%, which demonstrates that the model terms' contribution to the for water recovery rate from washing and flotation sludge of a phosphate ore beneficiation process is significant.

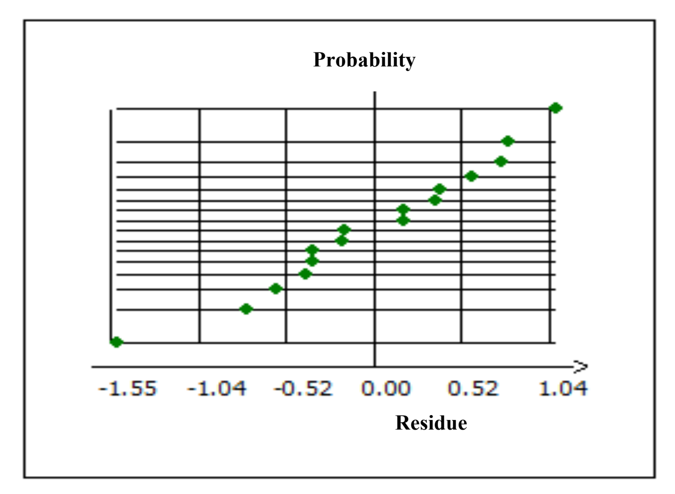

Figure 3: Normal Probability Plot

Table 5: ANOVA results for the response Y

Variation source	Sum of squares.	degrees of freedom	Mean of squares	F value	Prob > F
Regression	353.5766	9	39.2863	35.0262	**0.017**
Residual	6.7298	6	1.1216		
Total	360.3064	15			

There are other criterias that are widely used to illustrate the goodness of fit and adequacy of fitted mathematical models, such as R^2 and adjusted R^2_{ajs}. R^2 shows how well terms (data points) fit a curve or line. Adjusted R^2_{ajs} also indicates how well terms fit a curve or line, but adjusts for the number of terms in a model. The highest value of R^2 and adjusted R^2_{ajs} (close to 1) are recommended. The results presented in table 6 show that the R^2 and adjusted R^2_{ajs} are close to 1, indicates that most of the variation in water recovery rate from washing and flotation sludge of a phosphate ore beneficiation process could be predicted by the developed mathematical models.

Table 6: Quality of the mathematical model

$R^2 = 0.953$		**$R^2_{ajs} = 0.922$**

Based on the mathematical model given by Eqs. (**3**), the study of the various process parameters effects was made in order to analyze the optimal operating conditions that can be used for achieving the maximum water recovery rate from washing and flotation sludge of a phosphate ore beneficiation process.
The goal in the optimization of water recovery rate from washing and flotation sludge of a phosphate ore beneficiation process was to obtain the maximum rate of water in the range of the study conditions. In order to gain a better understanding of the results, the predictive model is presented in the figure 4 as the two and three-dimensional response surface plots.

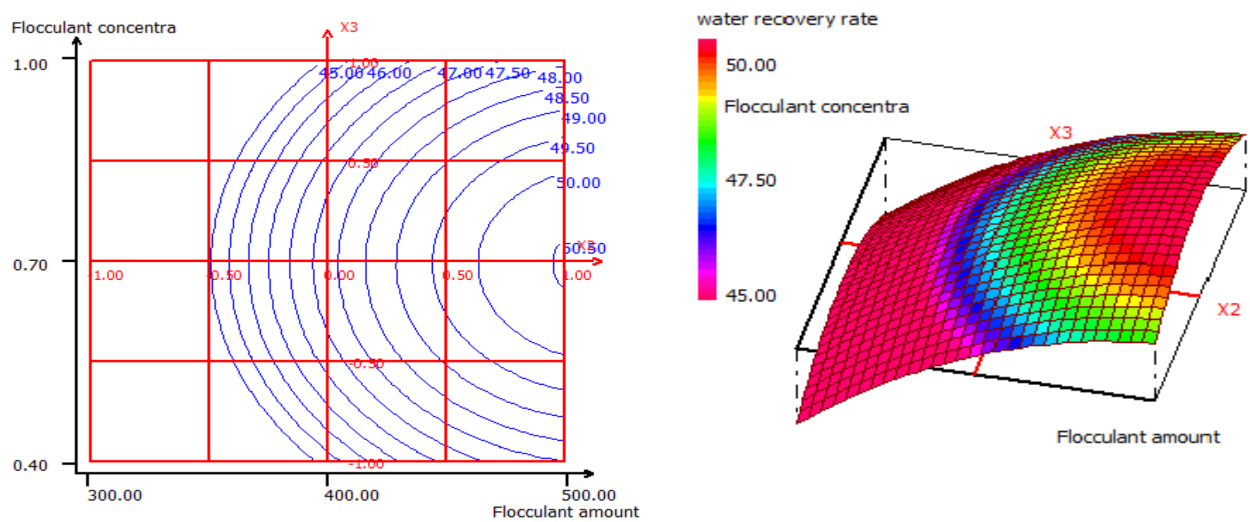

Figure 4: Response surface and contour plot (2D & 3D) for water recovery rate (at stirring time = 120s)

This figure is carried out for a stirring time of 120s. It is clear that for this duration we can recover up to 49% of water and this for several adjustments of the amount of flocculent and its concentration. Table 7 shows an example of a parameter adjustment that allows recovering up to 49% ± 2 of the water recovery rate.

Table 7 : Optimal point of water recovery rate

Factors	value	Water recovery rate (%)
Stirring time (s)	**120**	**49 ±2**
Flocculant amount (g/t)	**498**	
Flocculant concentration (g/l)	**0.75**	

Conclusions

In this work, our goal was to optimize the water recovery rate from washing and flotation sludge of a phosphate ore beneficiation process. First we evaluated the effect of five (05) parameters on this rate. We used a screening plan that has delivered three of the five that have a significant effect on the water recovery rate.

Then, we used a response surface design (Box Behnken) to study three parameters effect. Thanks to this design, we were able to find the adjusting parameters to recover until 49%water recovery rate from washing and flotation sludge of a phosphate ore beneficiation process.

It should be noted that the outcome of this project has several economic and environmental impacts. Thanks to this project, we were able, in a short time, to recover a large quantity of water which will be used later in the same process. Besides that, increasing water recovery rate will considerably reduce the demand the sludge storage area.

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