The Optimization of the Adsorption of the Tannic Acid on the Fluorite Mineral Surface using a Response Surface Methodology

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Abstract: The separation flotation of fluorite containing calcite by depression using the tannic acid is complex due to similar surface properties of calcium minerals. The improvement of this process requires an essential evaluation of the adsorption process. Therefore, the objective of this study is the optimization of the adsorption of the tannic acid onto fluorite. We conducted this experimental study by Central Composite Design of response surface methodology. We used experimental results obtained to develop a statistical model at a confidence level of 95 % using Statgraphics centurium software 18. This model revealed that the initial tannic acid concentration and the solution pH were the most significative parameters. Therefore, we exploited the obtained model to reach optimal conditions, allowing to achieve a maximum adsorbed tannic acid amount.

1 INTRODUCTION

Fluorite is an essential industrial mineral used to produce essentially hydrofluoric acid (Zhang and Song 2003; Gao et al. 2018; Zhang et al. 2018b). It usually coexists with other calcium-containing minerals, including calcite (Chen et al. 2019; Gao et al. 2019). Generally, physicochemical properties of calcium minerals such as fluorite and calcite are similar. Both interact similarly with anionic reagents (Liu et al. 2016). Thus, the valorisation of fluorite containing calcite as a gangue mineral is a very industrial problem and remains a challenging to overcome (Ren et al. 2017a; Gao et al. 2019).

In general, flotation is the physicochemical technique that permits to separation between fluorite and calcite minerals (Rutledge and Anderson 2015; Liu et al. 2016; Chen et al. 2017; Zhang et al. 2018a). The principle of this technique is the tannic acid adsorption on the fluorite mineral surface (Zhang et al. 2018c). To improve the separation process between calcium minerals, it is so important to evaluate this primary step before reaching flotation. To our knowledge, there are few published studies about the evaluation of the tannic acid adsorption process on fluorite surface. Most of them showed a strong interaction between phenolic tannin groups and ion calcium onto fluorite using optimization conventionnel method of adsorption (Ren et al. 2017b; Wei et al. 2017; Gao et al. 2018; Zhang et al. 2018a). Thus, the use of an efficient method to design and optimize the adsorption process is necessary.

In this work, we optimized the tannic acid adsorption process on the fluorite surface by a response surface methodology using Statgraphics centurium 18 software. This methodology permit to obtain a statistical model at a confidence level of 95 %. This model has been then exploited to determine the effect of reactional parameters (initial tannic acid concentration, solution pH and temperature) on the studied adsorption process. Finally, we used three-dimensional response surface methodology to determine optimal conditions of adsorption investigated.

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Table 1: Experimental range of independent variables and their real and coded values.

Variables	Levels and ranges		
	-1	0	1
C: Initial tannic acid concentration (mg/L)	20	60	100
pH: Solution pH	5	7,5	10
T: Temperature (K)	303	318	333

2 SETTINGS: ADSORPTION EXPERIMENTAL DESIGN

we conducted adsorption experiments by mixing 1 g of fluorite and 100 mL of distilled water using a thermostatic water bath equiped with an electric shaker under different conditions of the initial tannic acid concentration, the pH and the temperature. The mixture was analysed after equilibrium by the spectrophotometer Ultra Violet Visible to obtain the adsorbed tannic acid amount using the following expression:

$$Q = \frac{(C_i - C_e)}{m} * V \quad (1)$$

Where Ci and Ce (mg/L) are the initial and the equilibrium tannic acid concentration, respectively. V (L) is the solution volume, and m (g) is the adsorbent mass.

We selected operating parameters, including initial the tannic acid concentration, the solution pH and the temperature to evaluate their effect on the adsorption capacity. We chose the experimental range of each operating parameter based on preliminary experiments. We coded real factor values as -1, 0 and 1. Table 1 presented the experimental range, real and coded values of these parameters.

We conducted experimental tests under Central Composite Design CCD. We calculated the number of CDD experiments by the following formula:

$$N = 2^k + 2 * k + N_0 \quad (2)$$

Where k is the variable number, and N0 is the replicate number of the central value of the experimental range. We replicated this value three times (*N_0=3*) to estimate the experimental error and data reproducibility. Using thus the equation (2) and knowing that the variable number is 3 (*k=3*), we obtained 17 tests. Table 2 summarized CDD with its experimental results. We performed this experimental design using Statgraphics Centurium 18.

Table 2: CDD and experimental results.

Row	C (mg/L)	pH	T (K)	Q (mg/g)
1	20	5	303	0,035
2	100	5	303	7,56413
3	20	10	303	0,0337
4	100	10	303	6,2756
5	20	5	333	0,02865
6	100	5	333	3,9976
7	20	10	333	0,016203
8	100	10	333	2,6506
9	20	7,5	318	0,01024
10	100	7,5	318	3,62935
11	60	5	318	2,31678
12	60	10	318	1,94653
13	60	7,5	303	1,76912
14	60	7,5	333	0,85021
15	60	7,5	318	1,20021
16	60	7,5	318	1,1984
17	60	7,5	318	1,1996

3 RESULTS AND DISCUSSIONS

3.1 Statistical Modelling

In the present research, experimental results of Table 2 were exploited to obtain a statistical model using Box-Cox procedure (Box P 1964). We studied this model by analysis of variance ANOVA using Statgraphics Centurium 18. Table 3 summarized obtained results of this analysis.

Based on the high correlation coefficient (0,9994) and the low mean absolute error (0,027), the Box-Cox model provides a good description of tannic acid adsorption onto fluorite. In addition, ANOVA results indicate that all parameters are significantly based on the low P-Value (less than 0,05) except the second term of temperature.

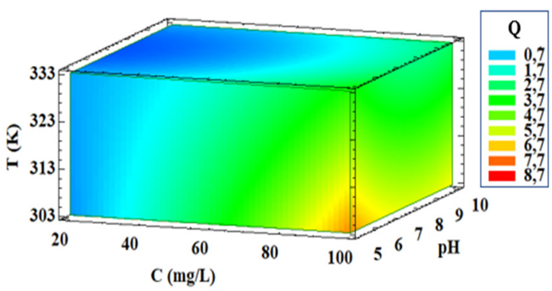

Figure 1: Contours of the estimated response surface.

Table 3: ANOVA analysis.

Source	Sum of Squares	Df	Mean Square	P-Value
C	28,9465	1	28,9465	0,0000
pH	4,97127	1	4,97127	0,0000
T	1,09445	1	1,09445	0,0000
C*C	1,52303	1	1,52303	0,0000
C*pH	0,0307715	1	0,0307715	0,0071
C*T	0,192934	1	0,192934	0,0000
pH*pH	0,33356	1	0,33356	0,0000
pH*T	0,0150994	1	0,0150994	0,0361
T*T	0,0032496	1	0,0032496	0,2768
Model	37,1108	9	4,12343	0,0000
Residual	0,019088	8	0,002386	
Total	37,1299	17		

The obtained statistical model is expressed by:

$$Q = 0{,}16*C - 0{,}46*pH - 0{,}004*T - 0{,}0005*C*C - 0{,}0004*C*pH - 0{,}0002*C*T + 0{,}05*pH*pH - 0{,}001*pH*T + 0{,}00001*T*T \quad (3)$$

3.2 Optimization

To optimize the tannic acid adsorption process onto fluorite, we used a three-dimensional (3D) response surface methodology. Figure 1 illustrates the contours of estimated response surface. Table 4 summarize obtained optimal conditions of the studied process. The optimization results show that we reach a maximum tannic acid adsorption onto fluorite using an initial tannic acid concentration of 100 mg/L, a pH of 5 and a temperature of 30 °C. Thus, we can compare these results with those obtained in our previous work (Tangarfa et al. 2021) of the optimization of the tannic acid adsorption onto calcite. We obtained a maximum adsorbed tannic acid amount onto calcite under an initial tannic acid concentration of 175 mg/L and a pH of 8. This means that the obtained optimal conditions of the adsorption corresponding to fluorite and calcite minerals are so different and selective. Therefore, we can use these results in our next works to separate between fluorite and calcite by flotation.

4 CONCLUSIONS

The experimental study of the tannic acid adsorption onto fluorite as a function of initial tannic acid concentration, pH and temperature was carried out and optimized by CDD of response surface methodology. We considered obtained results to get a validated statistical model checked by ANOVA analysis. Based on the high correlation coefficient and the low mean absolute error, we indicated that the Box-Cox model described well the tannic acid adsorption onto fluorite. Furthermore, we revealed based on the low P-Value (less than 5%) that the initial tannic acid concentration and the solution pH influenced the studied adsorption. Optimization results using 3D response surface methodology showed that we reached a maximum adsorbed tannic acid amount of about 7 mg/g using an initial tannic acid concentration of 100 mg/L, a pH of 5 and a temperature of 30 °C. The all above finding provides valuable results in mineral processing, allowing to enhance the fluorite valorisation by flotation using the tannic acid as a depressant.

Table 4: Optimal conditions of the tannic acid adsorption on the fluorite surface.

Q (mg/g)	C (mg/L)	pH	T (K)
6,99	100	5	303

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