Flotation tests using sorghum flour as a pyrochlore depressant

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Abstract

Cornstarch is one of the most used depressants in mineral flotation operations. However, corn is a costly input for the Brazilian mineral industry, since its main destination is to serve the animal and human food market and exports. In this study, sorghum and millet, two cheaper starch sources, were tested in order to reduce the mining dependence on corn. A simplex lattice design mixture {3,4} was used to evaluate the best proportion of corn, millet, and sorghum flours to be used as a pyrochlore depressant in relation to the industrially adopted cornstarch (Stargill 6172 supplied by Cargill). Flotation tests were carried out on a bench scale Denver flotation cell. All tests were performed in triplicate in a single rougher stage. The results found suggest that sorghum flour was a stronger pyrochlore depressant compared to corn and millet flours, with an average Nb_2O_5 content of 1.72 ± 0.05% and recovery of 80.75 ± 4.10%. These values were close to the ones found with Stargill 6172 (1.32 ± 0.02 and 80.95 ± 1.13%, Nb_2O_5 content and recovery, respectively), the industrially adopted depressant. The marked price and the easiness in the production of the flour in relation to the extraction of starches makes the sorghum flour a potential pyrochlore depressant, replacing the industrially used cornstarch.

Keywords: flotation reagents; depressant; sorghum; mixture design.

1. Introduction

Niobium's main use is steel microalloying, and accounts for 80% of the world's niobium production, comprising over 10 percent of the world's steel production (over 80 million tons per year). Niobium ore production and exportation play an essential role in the Brazilian economic development. This metal demand has increased in the last ten years, putting niobium ore in a featured position due to world steel production and consumption (Alves & Coutinho, 2015). Brazil has the world's largest niobium reserves (85%), followed by Canada (14%), and the United States (1%) (USGS, 2020). Araxá, located in the state of Minas Gerais, has the world's largest niobium deposit and is the world's largest niobium producer, operated by Companhia Brasileira de Metalurgia e Mineração (CBMM), followed by Catalão, in the state of Goiás, operated by Niobras, a CMOC Brazil group company (Ferreira Filho & Costa, 2019).

Pyrochlore ($(Na,Ca)_2Nb_2O_6(OH,F)$) is the most important niobium bearing mineral. Some of the other minerals present at the Brazilian pyrochlore deposits are barite ($BaSO_4$), magnetite (Fe_3O_4), goethite ($FeO(OH)$), limonite ($FeO(OH){\cdot}nH_2O$), monazite ($(Ce,La,Nd,Th)PO_4$), apatite ($Ca_5(PO_4)_3(OH, F, Cl)$) and quartz (SiO_2)

(Oliveira *et al*., 2001). Usually, this mineral is associated with carbonates and silicates (Ni *et al*., 2012) and its processing circuit is quite complex (Gibson *et al*., 2015), consisting of many stages: classification, magnetic separation, desliming, and many stages of flotation (Chelgani, 2013).

Flotation is one of the most important methods used for mineral enrichment worldwide. It is based on selectively turning hydrophilic minerals into hydrophobic ones (Quast, 2016). In most cases the mineral of economic interest is the one to be floated, therefore called a direct flotation. However, it is possible to sink the mineral of economic interest and float the gangue minerals (reverse flotation). Nowadays Niobras has up to four flotation stages. The flotation route is composed of carbonate flotation, silicate flotation, and reverse niobium flotation followed by a direct pyrochlore flotation. The carbonates flotation is carried out in alkaline pH, using saponified fatty acids as collector and gelatinized cornstarch as depressant (Bulatovic, 2010; Gibson, Kelebek, and Aghamirian, 2015). Eventually a sulfate flotation can be performed at the end of the process (Silva *et al*., 2017). The pyrochlore floatation is carried out using a cationic collector (amine) in an acidic pH. The particle size in this flotation system must be above 15 µm, and therefore, the slimes are removed after milling by cycloning (Gibson *et al*., 2015). According to Gibson, Kelebek, and Aghamirian (2021), the lack of knowledge regarding the role of different modifiers used in pyrochlore flotation can lead to inappropriate use, resulting in poor flotation.

According to Pawar *et al*. (2008), starch is the major source of energy stored as a carbohydrate in plants and it is composed of two polysaccharides: a linear one (amylose, with medium to high molecular weight), and a branched one (amylopectin, high molecular weight) (Pearse, 2005). Both the forms of starch are polymers of α-D-glucose. Natural starch contains 10-20% amylose and 80-90% amylopectin (Pawar *et al*., 2008). The amylose/amylopectin ratio is not constant, changing from one vegetable to another (Tester, Karkalas, and Qi, 2004). Cornstarch is the most common depressant used in Brazil. It is used not only for iron ore, but for many different ores (Peres & Correa, 1996).

The cultivation of corn in Brazil has two purposes: to meet the internal demand, being used as animal feed (pigs, cattle, and chicken), or industrialized for human consumption and, in this case, the production is sufficient for the domestic market. The surplus is exported as a commodity, obeying international quotations (Reis *et al*., 2016). The mineral industry has used low quality cornstarch, or even corn derivatives, and suffered with corn price fluctuations. One possible solution is to find another starch source to reduce the mineral industry dependence on corn.

To date, studies show the use of starches and flours from sorghum (*Sorghum bicolor* (L.) Moench) (Silva *et al*., 2019a; Silva *et al*., 2019b) and millet (*Pennisetum glaucum* (L.) R. Br.) (Silva, Sousa, and Silva, 2021) as depressants to reduce dependence on corn. However, none of these studies tested blends of corn flours (CF), sorghum flours (SF), and millet flours (MF) as a pyrochlore depressant. The present work presents for the first time the flotation results for a blend of the three flours through a simplex lattice mixture design (SLMD).

2. Materials and methods

2.1 Niobium ore samples

The ore and the water samples used in this work were donated by Niobras. The ore samples were collected after desliming, prior to the addition of any chemicals for flotation. The samples were then dried in an oven at 70 °C for 24 hours, homogenized and quartered with a Jones splitter into samples with 900 g on average.

2.2 Reagents and flotation tests

The collector (Lioflot-502A, a fatty acid supplied by Miracema-Nuodex) and depressant (Stargill 6172, a modified cornstarch supplied by Cargill) adopted at Niobras were used as benchmarks and prepared as recommended by the company. The collector solution was prepared adding 16 g of Lioflot-502A in 150 mL of distilled water in a 300 mL volumetric flask. The solution was kept under magnetic stirring and a solution of 6 g of NaOH to 50 mL of distilled water was slowly added to it. After the complete homogenization of the solution, its volume was completed to 300 mL with distilled water. The depressant was prepared adding 50 g of Stargill 6172 to 200 mL of distilled water in a 500 mL volumetric flask. The solution was kept under magnetic stirring for 5 min at 1.200 RPM until the starch complete solubilization. Then a solution of 23 g of NaOH in 50 mL of distilled water was slowly added to the depressant solution and kept under magnetic stirring for 15 minutes for the starch to complete gelatinization. The solution volume was completed to 500 mL with distilled water.

Graniferous sorghum (type 1G100 Dow Agrosciences) and pearl millet (type ADR 300) grains were supplied by the Agroceres, harvested from a farm located in Ipameri, Goiás, Brazil. The flour preparation and gelatinization were done as proposed by Silva *et al*. (2019a). The authors showed that sorghum and millet need a smaller volume of NaOH solution to be gelatinized then the Stargill 6172, since a highly viscous solution was obtained if the same amount of NaOH solution used in the Stargill 6172 gelatinization was used with sorghum or millet.

The flotation tests were performed in a single rougher stage in a bench scale Denver flotation cell manufactured by CDC, using a cell with 2 L (internal volume) to study the first flotation in the niobium process route (AKA carbonate flotation). All tests were carried out in close agreement with Niobras operational parameters (see Table 1). Both depressant and collector were daily prepared to avoid the retrogradation of the starches and any alterations of the collector, as suggested by the suppliers. A NaOH solution (10% w/v) was used as a pH modifier. All the tests were carried out in triplicate in the Modelling and Mineral Processing Research Lab (LaMPPMin).

Table 1 - Bench scale flotation tests parameters.

Parameter	Value
Solids % (conditioning)	55
pH during conditioning	11
Agitation speed (conditioning, RPM)	1400
Depressant dosage (g/t)	800
Depressant conditioning (min)	7
Collector dosage (g/t)	100
Collector conditioning (min)	3
Solids % (flotation)	42
Airflow rate (L/min)	4.0
pH during flotation	10.3
Agitation speed (flotation, RPM)	1300
Flotation time (min)	2 – 4

2.3 Experimental design and statistical analysis and modeling of experimental data

A simplex lattice mixture design (SLMD) for three components and a 4th degree polynomial {3,4} was used to evaluate the effect of dosages of CF, MF, and SF on the niobium recovery (RNb_2O_5). The experimental design produced 15 experiments, three single-flour tests, nine two-flour blends, and nine three- flour blends, as presented in Table 2. The effect of SLMD on the niobium recovery was analyzed using the method of least square for multiple regression. The linear, full quadratic, special cubic, full cubic, special quartic, and full quartic models were tested for the niobium recovery, and the fitted models were subjected to an analysis of variance (ANOVA) with $p < 0.05$. The best-fit equations were evaluated after dropping the non-significant terms. All statistical calculations were carried out using the statistical software Minitab version 21.1.1.

Table 2 - Flotation tests experimental design according to the SLMD.

Experiment	Flour dosages (g)			Coded flour dosages		
	CF	MF	SF	CF	MF	SF
1	2.500	0.000	0.000	1	0	0
2	0.000	2.500	0.000	0	1	0
3	0.000	0.000	2.500	0	0	1
4	1.250	1.250	0.000	½	½	0
5	0.000	1.250	1.250	0	½	½
6	1.250	0.000	1.250	½	0	½
7	0.625	0.000	1.875	¼	0	¾
8	0.625	1.875	0.000	¼	¾	0
9	0.000	0.625	1.875	0	¼	¾
10	0.000	1.875	0.625	0	¾	¼
11	1.875	0.625	0.000	¾	¼	0
12	1.875	0.000	0.625	¾	0	¼
13	1.250	0.625	0.625	½	¼	¼
14	0.625	1.250	0.625	¼	½	¼
15	0.625	0.625	1.250	¼	¼	½

3. Results and discussion

3.1 Flotation tests

Figure 1 presents the contour plot of the influence of the flours' dosage on the niobium recovery. The binary and ternary interaction between the flours behaved in an antagonistic way compared to the pure components for niobium recovery. The average value of the niobium recovery was reduced in both cases, regardless the flour dosage. A higher niobium recovery indicates a more effective flotation. The best results were found for single starches, namely 100% SF (80.75 ± 4.10%), 100% MF (77.25 ± 4.16%), and 100% CF (75.03 ± 1.36%). This result shows the high potential that SF has as a pyrochlore depressant, adding it to the list of minerals already tested in literature, such as hematite, silicates, and apatite (Silva, 2018; Silva *et al.*, 2019b). A Student's t-test was conducted to test the niobium recovery using the three flours and Stargill 6172. Sorghum flour and Stargill 6172 were statistically similar for a 95% confidence interval ($p>0.05$), and different from the other two flours.

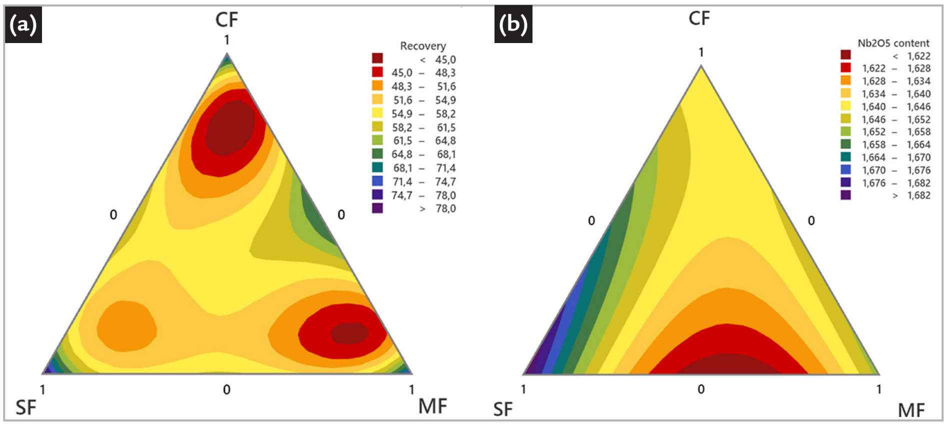

Figure 1 - Contour plot of the flours dosage for niobium (a) recovery and (b) content.

According to Silva (2018), sorghum has a higher amylopectin content than corn. The relationship between the levels of amylose and amylopectin present in starches used as depressants in mineral flotation should be considered as an important characteristic that can affect the process. Cereda *et al.* (2001) state that the greater the content of amylopectin in starches, the better the mineral separation. Matos, Alexandrino, and Ferreira (2020) observed higher values of iron ore yield and recovery using pure amylopectin when compared to pure amylose and cornstarch. Pinto, Araújo, and Peres (1992) showed, through microflotation tests in a Hallimond tube, that pure amylopectin is a stronger hematite depressant than pure amylose. According to the authors, pure amylose is a weaker depressant, followed by amylopectin and starch for apatite. These results indicate that starches with higher levels of amylopectin should be stronger depressants.

The flours were tested integrally, which means that no separation or purification stages were carried out. The proteins present in sorghum (such as kafirin) could have contributed to the pyrochlore depression. According to Duodo *et al.* (2003), kafirin is similar to zein, the corn protein, since they both are prolamine proteins. Studies conducted by Correa (1994) and Peres & Correa (1996), show that zein had a depressant effect on hematite. Even more, Tohry *et al.* (2021) showed that tannins, which are also present in sorghum flour, were effective depressants in the hematite flotation in the presence of quartz.

Regarding to the Nb_2O_5 content, the best results were also found for pure sorghum flour (1.72 ± 0.05%) with an average yield of 38.49 ± 1.68%. In these tests the average Nb2O5 content in the flotation feed was 1.38 ± 0.02%. A Student's t-test was conducted to test the yields using Stargill 6172 and sorghum flour, indicating that the two depressants are statistically similar for a 95% confidence interval ($p>0.05$), and different from the other two flours.

Figure 2 presents a plot using the industrial results from the cleaner stage at Niobras. Unfortunately, it was not possible to collect samples after the rougher stage, and therefore, the cleaner stage was used in comparison with the lab results. The points indicate the average niobium recovery results from the bench flotation tests: the yellow line is the average niobium recovery in the industrial cleaner stage and the blue and green lines represent its plus one and minus one standard deviation. Although the flours were predominantly composed of starch, they also had other components, such as proteins, lipids, among others. Therefore, considering that the sorghum grain was composed of 75 to 79% of starch, it is possible to notice that the starch dosage when using sorghum was lower than that used in Niobras with Stargill 6172.

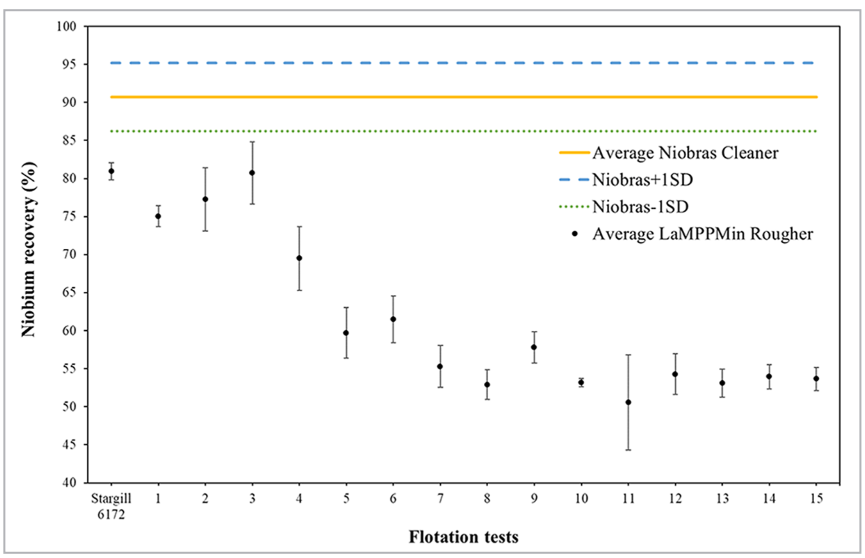

Figure 2 - Comparison between the niobium recovery average results in all flotation tests.

3.2 Statistical analysis and modelling of experimental data

The first statistical analysis performed consisted of choosing the most appropriate model for the niobium recovery, based on the results of the ANOVA test and the lack of fit of the models. The insignificant terms in the models were dropped, since the models were analyzed hierarchically. SLMD {3,4} made it possible to adjust from the linear model to the complete quartic model, as presented in Table 3. The models with significant confidence interval of 95% were the quadratic and the full quadratic models (with $p < 0.05$). However, for the quadratic model, the lack of fit was significant ($p > 0.05$). Because of that, this model was disregarded. Table 4 shows the ANOVA results of the full quadratic model and its coefficients are shown in Table 5.

Table 3 - Models fit summary for the niobium recovery.

Model	p-Value	Lack of fit	R^2 (%)	Predicted R^2 (%)
Linear	0.853	< 0.001	0.760	0.000
Quadratic	<0.001	< 0.001	57.750	43.480
Special cubic	0.260	< 0.001	59.160	43.240
Full cubic	0.951	< 0.001	59.560	34.610
Special quartic	0.741	< 0.001	59.170	42.610
Full quartic	<0.001	0.679	92.760	87.860

Table 4 - ANOVA for the full quartic model for niobium recovery.

Source	Degrees of freedom	F-Value	p-Value	Predicted R^2 (%)
Regression	8	57.69	0.000	87.86
Linear	2	4.05	0.026	
Quadratic	3	74.89	0.000	
CF*SF	1	89.82	0.000	
CF*MF	1	17.32	0.000	
SF*MF	1	118.04	0.000	
Full quartic	3	58.07	0.000	
CF*SF*(-)2	1	33.38	0.000	
CF*MF*(-)2	1	112.15	0.000	
CF*MF*(-)2	1	22.77	0.000	
Residual	36			
Lack of Fit	6	0.66	0.679	
Pure Error	30			
Total	44			

Table 5 - Coefficients of the full quartic model estimated for niobium recovery.

Term	Coefficient	Standard error of the coefficient	T-Value	p-Value	VIF
CF	75.43	1.56	*	*	2.49
SF	81.26	1.56	*	*	2.49
MF	77.31	1.56	*	*	2.49
CF*SF	-71.60	7.55	-9.48	0.000	3.16
CF*MF	-31.44	7.55	-4.16	0.000	3.16
SF*MF	-82.08	7.55	-10.86	0.000	3.16
CF*SF*(-)2	-220.20	38.1	-5.78	0.000	2.16
CF*MF*(-)2	-403.60	38.1	-10.59	0.000	2.16
SF*MF*(-)2	-181.90	38.1	-4.77	0.000	2.16

All model coefficients were significant, as confirmed by the p-value ($p < 0.05$) and because the calculated standard error was much lower than the value of the coefficient itself. All interactions between the flours were related in an antagonistic way. This means that the coefficients showed negative results for the interaction effects. Therefore, the combinations between the flours impair the process, decreasing the niobium recovery. According to Mendes (2019), the value and sign of the coefficients indicate the influence of each component, as well as the synergism and antagonisms that act between them. Regarding the variance inflation factor (VIF), which is a measure of the amount of multicollinearity in regression analysis, the coefficients varied between 2.16 and 3.16, considered satisfactory, and consequently, not incurring in multicollinearity. Authors, such as Myers & Montgomery (2002) are more rigorous and stipulate that the value of the VIF cannot exceed 4 or 5 units.

Models with high Predicted R^2 values have better predictive capabilities. Thus, the complete quartic model explained 87.86% of the data variation around the mean, and the rest of the data variation is explained by the residues. The residues are distributed symmetrically (the number of points is similarly distributed above and below the zero line). This behavior indicates that the proposed model can be considered adequate to the data. Furthermore, the points are randomly distributed, characterizing homoscedasticity of effect-sizes (constant variance) (see Figure 3). Figure 4 shows the plot of normal distribution of the residuals. Since most of the points were located close to each other and alongside a line, it is possible to assume that the error of the proposed model follows a normal distribution for the niobium recovery. According to Montgomery & Runger (2014), errors follow a normal distribution, if and only if, about 95% of the standardized residues are contained in the range (-2, +2). The found results are in accordance with the authors statement, since only two points stood out from this analysis. However, this fact was not considered harmful to the model representativeness, since the found p value was higher than 0.05 ($p = 0.072$).

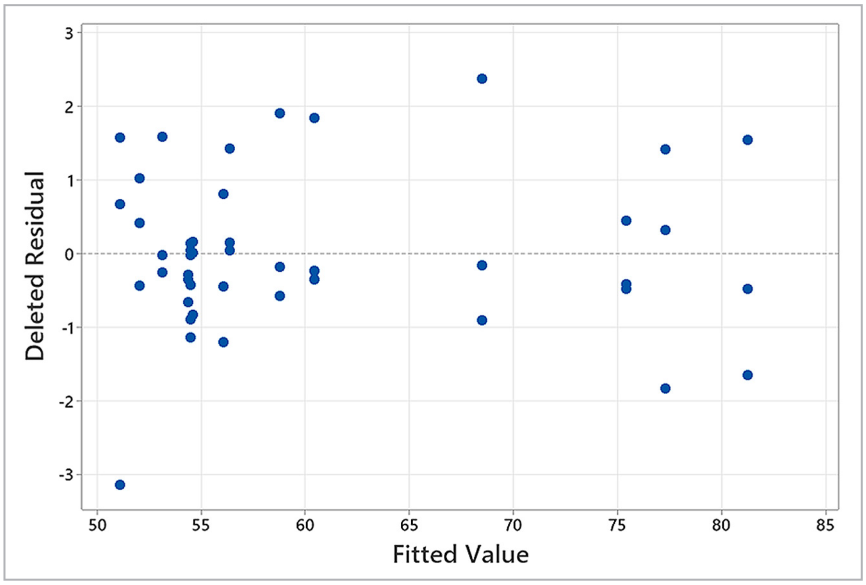

Figure 3 - Deleted residual vs. model fitted values.

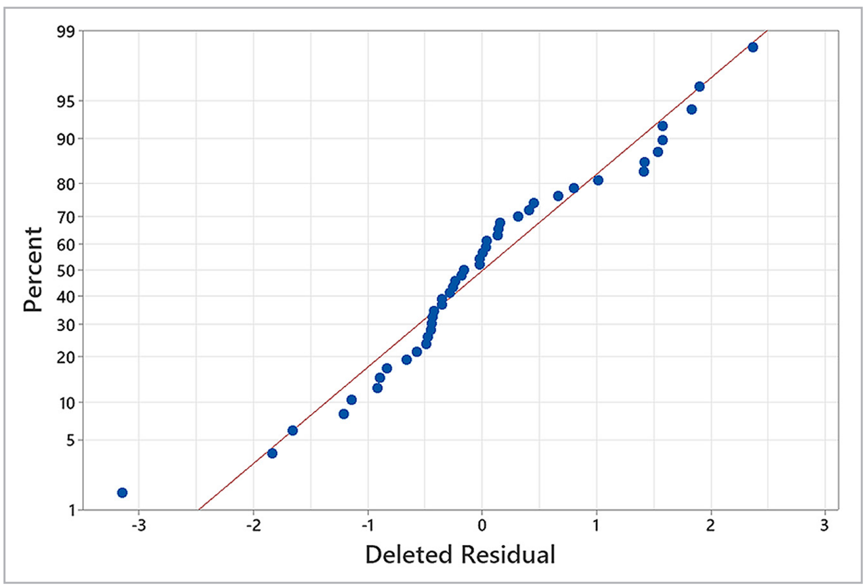

Figure 4 - Normal distribution of the model residuals.

4. Conclusions

Bench scale flotation tests were carried out to find a starch based depressant replacement for corn flour, or even cornstarch, to be used in the carbonate flotation, the first flotation step in the niobium processing route. The results indicate that sorghum flour had results similar to the industrial cornstarch. The mixture of the three starch sources were also investigated, but no increase in the niobium recovery, or content, was found. Although the flour mixtures could reduce the depressant cost, they were not synergistic in the tested conditions. Another conclusion is regarding the use of a flour instead of a starch. The flour production is an easier and cheaper process when compared to the starch one. Therefore, the use of sorghum flour replacing the cornstarch could lead to a cost reduction in the niobium flotation.

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