

Hematite and quartz microflotation using millet starch as depressant

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Abstract

Brazil is among the world's largest iron ore producers. The standard concentration method is reverse cationic flotation using amines, and their derivatives, such as quartz collector and corn (as either grits, flour, or starch) as the hematite depressant. Corn is considered cheap, abundant, and available all around the country. However, its demand has been abruptly increasing over the last few years, mainly due to the emerging of new attractive markets, such as ethanol production. In order to propose a feasible replacement for corn, hematite and quartz samples from the Brazilian Iron Quadrangle were tested in a modified Hallimond tube using millet starch as depressant for the first time (corn and sorghum starch were used as benchmark). Tests were carried out with four different depressant dosages and four different pHs, all in triplicate. It was possible to reach quartz floatability above 98% and hematite depressability above 93% for millet and sorghum starches. This fundamental study shows that replacing corn with millet poses a real opportunity to reduce operational costs.

Keywords: flotation reagents; depressant; hematite; quartz; starches.

1. Introduction

The world's iron ore production has increased approximately 280 million tons from 2012 to 2017. According to Araujo *et al.* (2005), concentrates with iron content above 60% are required to fit the steel making industry requirements. The main concentration method for iron ore enrichment is the reverse cationic flotation, in which the iron ore, typically hematite, is depressed (Filippov *et al.*, 2010; Ma *et*

al., 2011) using starch, dextrin, sodium silicate, or CMC, inhibiting the adsorption of the collectors on the iron ore minerals surface (Bai *et al.*, 2019). The gangue minerals, mainly composed of quartz, are recovered using amine-based collectors. Peres and Correa (1996) showed that minerals, such as iron oxides, have an affinity with starch and other polysaccharides, which facilitates their depression during

the reverse cationic flotation. According to the Brazilian Mining Agency (ANM, 2018), approximately 265 Mtons of iron ore concentrate were produced in the Iron Quadrangle in 2016, consuming around 106 ktons of cornstarch (CS), considering an average dosage of 400 g of CS per ton of iron ore processed. CS is the standard depressant in Brazil, mainly due to its availability and market price (Araujo *et*

al., 2005).

Starches from different botanical sources could be used as depressants as long as they are available in the market with acceptable price and sufficient volume to meet the mining company demands (Peres and Correa, 1996; Turrer and Peres, 2010; and Silva *et al.*, 2019b).

Millet is diverse and broadly adapted crops. Pearl millet (*Pennisetum glaucum*), here addressed as millet, is an annual cespitose summer grass, with a high production of tillers and a vigorous regrowth after cutting or grazing. Showing a high drought tolerance, millet has

been considered a rudimentary plant. No other cereal has a big harvest under similar climatic conditions, especially in high temperatures or in dry conditions. According to Graybosch and Baltensperger (2009), these features lead millet to become one of the main crops in semi-arid areas of Asia and Africa, where other major crops tend to fail. Millet flour has been used in the food industry to prepare cakes, biscuits, and porridge (Kulkarni *et al.*, 2018). However, there is no record of its human consumption in Brazil, being cultivated as a fodder grass.

Research must be carried out to

reduce, or to find, a suitable replacement for CS in the mineral industry. The present article shows for the first-time, the experimental results of hematite and quartz flotation using millet starch (MS) as depressant. CS and sorghum starch (SS) (*Sorghum bicolor* (L.) Moench) were used as benchmark. CS was used because it is the standard depressant in Brazil in a wide range of mineral flotation processes. SS, on the other hand, is a widely farmed crop cultivated in Brazil for animal feeding, with high economical and technical potential to be adopted in the mineral industry, as shown by Silva *et al.* (2019a).

2. Materials and methods

2.1 Mineral samples

Mineral samples from the Brazilian Iron Quadrangle were used in this study. Sample preparation and characterization can be found elsewhere (Silva *et al.*,

2019b). According to the authors, the samples had a purity of around 97% and 95%, for hematite (total Fe around 67.6%) and quartz, respectively. The isoelectric

point (IEP) for hematite was determined at pH 6.95. No IEP was determined in the tested pH range (3.5 to 12.5) for the quartz samples.

2.2 Starches extraction and characterization

Pearl millet grains (type ADR 300 from ATTO Adriana Seeds) used in this study were provided by Fertigran, a company located in the city of Catalão, State of Goiás. Graniferous sorghum grains (type 1G100 from Dow AgroSciences) were farmed in Ipameri, Goiás, Brazil and provided by Agroceres. Straw residues and other impurities were removed by dry sieving. The starch extraction and characterization were performed as proposed by Silva *et al.* (2019a). Cargill supplied the CS samples (Amigel 12100). The starch granule morphology was analysed using SEM JSM-6610 from Jeol at Labmic/UFG. Samples were fixed with carbon tape on aluminium supports and then

metallized with a thin gold layer (350 Å). The starch density was measured by pycnometry and xylene as the liquid phase. The starch pH was measured according to the methodology proposed by the Adolfo Lutz Institute (1985).

Starches are water-insoluble, requiring a preparation stage prior to their use as a depressant, known as gelatinization (Liu *et al.*, 2017). It is possible to perform the starch gelatinization by two procedures (thermal or alkaline). Alkaline gelatinization is normally performed using sodium hydroxide (Santiago-Ramos *et al.*, 2017). The depressant gelatinization was performed by adding 2.7 mL of

sodium hydroxide at 10% (w/v) to a solution of 20.0 mL of distilled water and 1.0 g of starch. The solution was kept under magnetic stirring until complete gelatinization. The depressant/NaOH ratio was adopted as proposed by Silva *et al.* (2017). The collector preparation consisted of a dilution of 1.0 g of it in distilled water to a total volume of 100 mL (1% w/v) under magnetic stirring, as recommended by the manufacturer. The collector and depressant solutions were prepared daily to avoid any degradation of the reagents, especially starch retrogradation. Hydrochloric acid and sodium hydroxide, both at 1% solution, were used as pH modifiers.

2.3 Microflotation tests

Figure 1 shows the experimental setup of the modified Hallimond tube, with internal volume of 320 mL. The Stat-Ease DesignExpert 12 was used to plan and analyse the flotation results. An incomplete factorial design 4³ was adopted, with three independent variables (pH, depressant dosages and type) and four levels of each variable, except for the depressant type. Four pHs (9, 9.5, 10, and 10.5) were tested, as proposed in literature in close agreement with the industrial practice for hematite flotation, mineral recovery optimization, and preferred adsorption of the amine onto the quartz surface (Pinto *et al.*, 1992, Santos and Oliveira, 2007, and Silva *et al.*, 2019b).

Four depressant dosages were tested (5, 10, 20, and 40 mg/L). In addition, blank tests (without depressant) were also carried out at the same four pHs. Flotigam EDA (an ether amine) manufactured by Clariant was used as collector in two different dosages: 20 mg/L for hematite and 5 mg/L for quartz, as proposed by Silva *et al.* (2019b). The authors discovered that the hematite floatability was, as expected, lower than the quartz floatability under the same operational conditions and in the absence of depressant. Therefore, a high collector dosage was used to produce similar floatability between the two minerals, allowing a better understanding of the depressant influence.

The air pressure was kept at 10 psi and the airflow at 40 cm³/min, producing a superficial air velocity of 0.02 cm/s. These values were adopted in order to minimize the hydraulic entrainment (Guimarães Júnior *et al.*, 2015). The mineral samples used, weighed 1.0 g and were wet sieved at -149+105 µm (or -100+150 #). Although this particular particle size is coarser than the industrial practice, hydraulic entrainment tests were performed by Guimarães Júnior *et al.* (2015) for both minerals and the lowest value was obtained at this granulometry. Even more, Ma *et al.* (2011) found good results working with iron ore samples from Brazil with d_{80} of 129 µm by reverse anionic flotation.

$$\text{Hematite}_{\text{Depressability}} = 100 \cdot \frac{M_d}{M_f + M_d} \quad (2)$$

3. Results and discussion

3.1 Starches characterization

MS extraction produced a white powder, with average yield of $38.6 \pm 0.45\%$, almost 50% higher than SS ($25.82 \pm 4.08\%$). Figure 2 shows SEM/SEI images for the three starches.

Although the predominant shape of the starch granules was oval or semi-spherical, some grains with varied shapes could be seen. Watson and Eckhoff (2009) reported similar results for large sorghum granules

with polygonal and spherical shapes. Regarding the grain texture, the starches had a smooth surface, with an occasional presence of superficial pores. No protein ribbons were found.

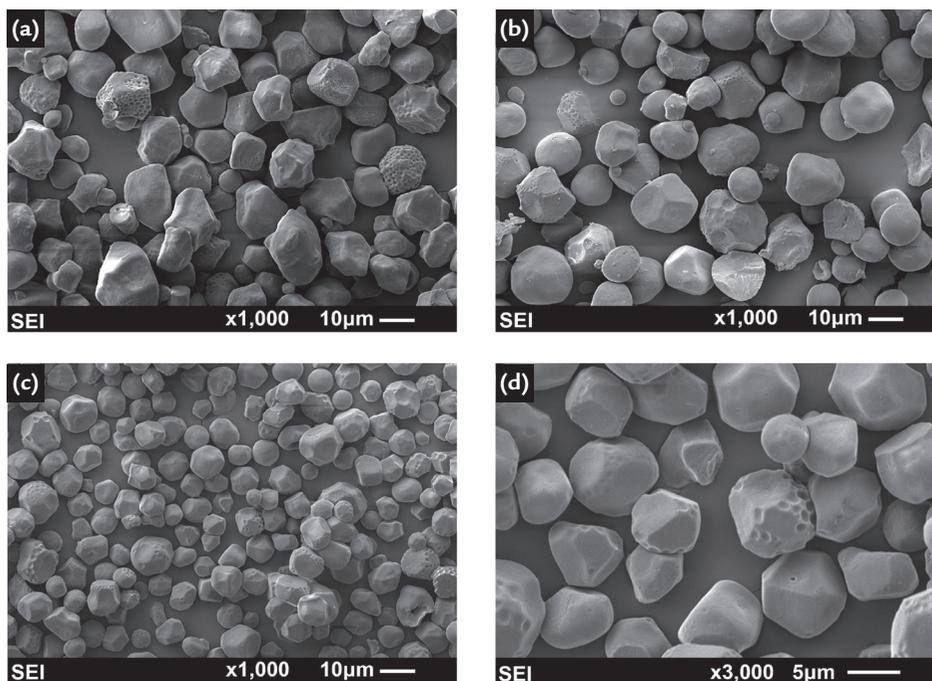


Figure 2 - SEM/SEI images for (a) CS, (b) SS, and (c) MS 1,000x magnification and (d) 3,000x magnification.

Table 1 shows the starch characterization results. Starches are mainly composed of Amyloses (AM) and Amylopectin (AP), in a ratio of approximately 1/3 (Peres and Correa, 1996). This ratio was observed for MS

($1/3.23 \pm 0.1$) and SS ($1/2.43 \pm 0.8$), but not for CS ($1/1.26 \pm 0.1$), suggesting that these two starches have higher potential as a depressant than CS. Triacylglycerol (popularly designated as oil or lipid) present in the starch acts as an

antifoaming agent, which impairs the flotation process if its content is higher than 1.5% (Peres and Correa, 1996). All tested starches had lipid content below 1.5%, with emphasis to MS (0.12 ± 0.05).

Table 1 - Depressants characterization.

Botanical sources	Cornstarch	Millet starch	Sorghum starch
Amylose (AM) (%)	38.73 ± 0.8	20.80 ± 0.30	25.46 ± 0.8 ^[a]
AM molecular weight	105 to 3×10^6 ^[b]	1.6×10^6 ^[c]	1.1×10^7 ^[d]
Amylopectin (AP) (%)	48.81 ± 0.95	67.10 ± 0.62	61.77 ± 0.86
AP molecular weight	107 to 2×10^9 ^[b]	1.3×10^7 ^[c]	3.0×10^8 ^[d]
AM/AP	$1/1.26 \pm 0.1$	$1/3.23 \pm 0.1$	$1/2.43 \pm 0.8$
Lipids (%)	0.80 ± 0.10	0.12 ± 0.05	0.49 ± 0.1 ^[a]
Proteins (%)	0.87 ± 0.05	1.00 ± 0.05	0.85 ± 0.05 ^[a]
Ashes (%)	0.51 ± 0.11	0.10 ± 0.03	0.12 ± 0.1 ^[a]
Solubility index (%)	2.35 ± 0.42	1.96 ± 0.27	1.43 ± 0.2 ^[a]
Swelling power (g/g)	7.93 ± 0.25	8.93 ± 0.47	9.88 ± 0.2 ^[a]
Starch pH	5.29 ± 0.19	6.52 ± 0.08	6.54 ± 0.06
Density (g/cm ³)	1.46 ± 0.03	1.39 ± 0.04	1.48 ± 0.04

^[a] Silva *et al.*, 2019a; ^[b] BeMiller, 2018; ^[c] Madhusudhan and Tharanathan, 1996; ^[d] Matalanis *et al.*, 2009.

Additional study is required to understand the role of some starch components in mineral flotation, e.g. zein, the most abundant protein in corn, shows a depressing ability for hematite as strong as AP (Peres and Correa, 1996). MS protein content (1.00 ± 0.05) was approximately 15% higher than the other starches. MS and SS showed similar swelling power at

high temperatures ($90\text{ }^\circ\text{C}$), 8.93 ± 0.47 g/g and 9.88 ± 0.2 g/g, respectively, and solubility index ($1.96 \pm 0.27\%$ and $1.43 \pm 0.2\%$, respectively), in agreement with the results found by Tyl *et al.* (2018) for MS. According to Silva *et al.* (2019a), the solubility index is directly proportional to the protein amount. The swelling power, on the other hand, is directly proportional

to the amount of AM and AP, which partially explains the MS results. Starch densities were statistically similar and in agreement to the results available in literature (Silva *et al.*, 2019a). It can be seen from the results that the starch pH values are close to neutrality, compatible with native starches that did not undergo any fermentation or modification process.

3.2 Quartz microflotation results

Figure 3 shows the average quartz floatability as a function of the depressant dosage for the tested pHs. The highest results for quartz floatability were obtained for MS with dosages greater than or equal to 20 mg/L. This result suggests that MS adsorption on the quartz surface is lower than the other two starches. In general, an

increase in the depressant dosage resulted in a decrease in quartz floatability for CS and SS. It is possible to notice that quartz floatability was less susceptible to pH variation with MS. The average quartz floatability ranged from $42.53 \pm 3.19\%$ to $90.90 \pm 1.77\%$ with CS, while with MS ranged from $91.71 \pm 0.55\%$ to $98.19 \pm 0.10\%$, and from

$34.49 \pm 2.60\%$ to $98.10 \pm 1.69\%$ with SS. The highest average quartz floatability ($98.19 \pm 0.10\%$) was obtained with 40 mg/L of MS at pH 10.5. In the absence of the depressants the average quartz floatability ranged from 96.01 ± 0.22 (pH 10) to $97.28 \pm 1.12\%$ (pH 9.5). The addition of the depressant reduced the quartz floatability, with a few exceptions.

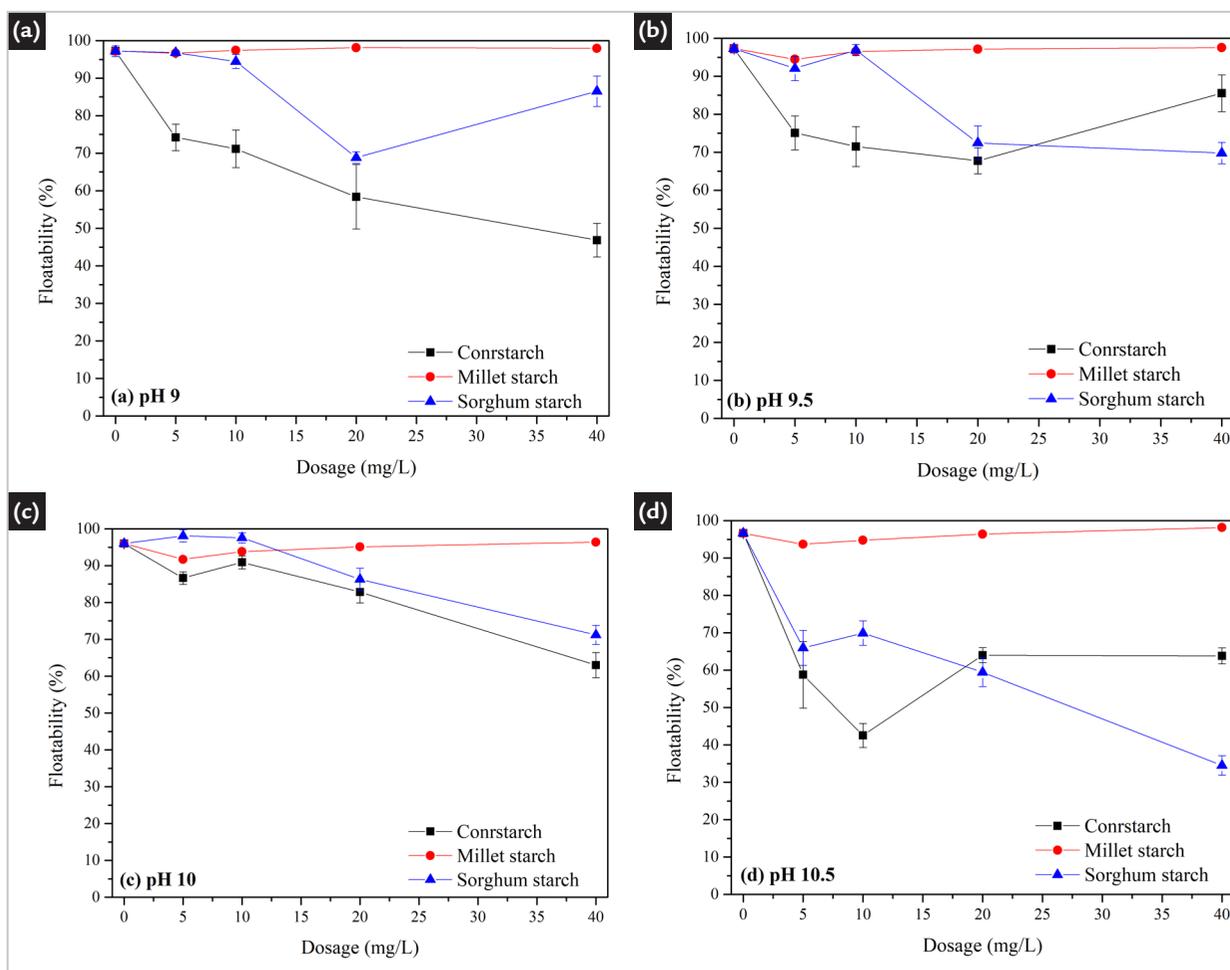


Figure 3 - Average quartz floatability as a function of the depressant dosage at four different pHs: (a) 9, (b) 9.5, (c) 10, and (d) 10.5.

Veloso *et al.* (2018) determined the isotherm adsorption of unmodified regular CS (73% AP and 27% AM) by igneous iron ore samples. The authors found that at pH 10.5, the depressant presented lower adsorption in the silicates (composed of albite, chamosite, diopside, microcline, quartz, and epidote), allowing them to be

floated by the collectors. This behaviour was not seen in the carried-out tests. The average quartz floatability at pH 10.5 dropped from $96.63 \pm 0.55\%$ without CS to $42.53 \pm 3.19\%$ with the addition of 10 mg/L, which was the lowest quartz floatability found at this pH for CS. In general, an increase in the depressant dosage pro-

moted a decrease in the quartz floatability, indicating an interaction between them. Since in a reverse flotation the non-floated quartz will depress, contaminating the concentrate, it is expected that such decrease in the quartz floatability will promote a similar decrease in the hematite grade.

A cubic model was chosen to model

the flotation results for quartz and for hematite (Tables 2 and 4, respectively). Figure 4 shows the 3D model for quartz flotation. The normality of the results was checked by plotting the normal probability versus studentized residuals (the residual is defined by difference between the observed and the predicted data). This plot indicates whether the residuals follow a normal distribution, in which case the points will

follow a straight line. All the experimental results were approximate to the continuous line, which could be due to the absence of evident problems on design normality. Therefore, the adopted regression model was considered adequate (F-value of 30.85 and p-value < 0.0001), with only a 0.01% chance that the F-value was due to noise as shown in Table 3 (model terms not significant are not shown). All three tested

parameters (pH, depressant dosage, and starch type) were significant for quartz floatability, as expected (Silva *et al.*, 2019b; Pavlovic and Brandao, 2003). Therefore, it is safe to assume that the three parameters had influence in the quartz floatability. The first order interactions were significant for pH and the starch type, and depressant dosage and starch type, but not significant for pH and depressant dosage.

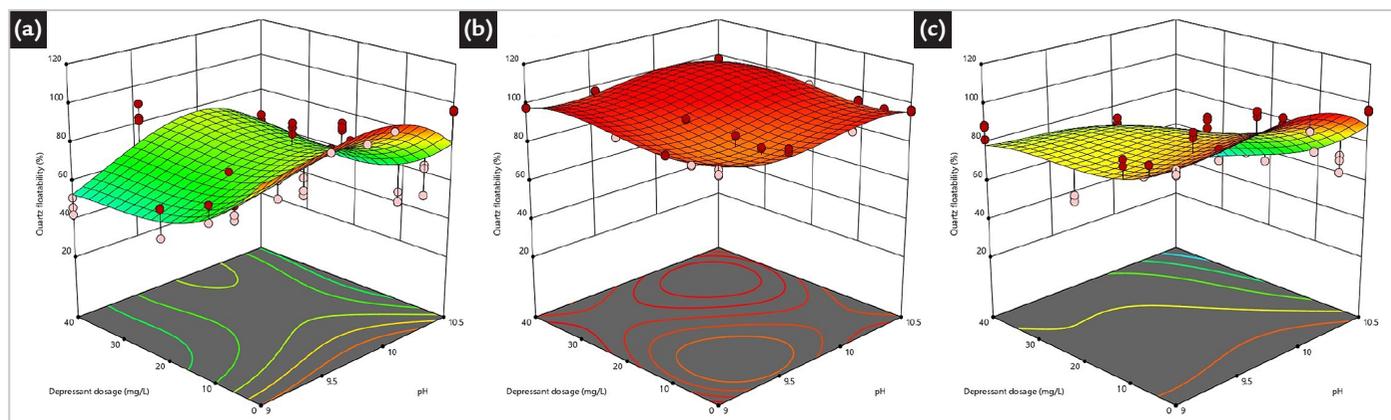


Figure 4 - Quartz response surface for (a) cornstarch, (b) millet starch, and (c) sorghum starch.

Table 2 - Models fit summary for quartz flotation results.

Model	Sequential p-value	Lack of Fit p-value	Adjusted R ²	Predicted R ²	Conclusions
Linear	< 0.0001	< 0.0001	0.4568	0.4342	
2FI	< 0.0001	< 0.0001	0.5763	0.5422	
Quadratic	< 0.0001	< 0.0001	0.6700	0.6379	
Cubic	< 0.0001	< 0.0001	0.7779	0.7447	Suggested
Quartic	< 0.0001	< 0.0001	0.8668	0.8325	Aliased

Table 3 - ANOVA for Cubic model for the quartz flotation. Model terms not significant are not shown.

Source	Sum of squares	Degrees of freedom	Mean square	F-value	p-value
Model	39344.62	21	1873.55	30.85	< 0.0001
A-pH	1675.04	1	1675.04	27.59	< 0.0001
B-Depressant dosage	6852.19	1	6852.19	112.85	< 0.0001
C-Starch type	14421.70	2	7210.85	118.75	< 0.0001
AC	1718.73	2	859.36	14.15	< 0.0001
BC	4537.51	2	2268.75	37.36	< 0.0001
A ²	2859.78	1	2859.78	47.10	< 0.0001
B ²	1673.97	1	1673.97	27.57	< 0.0001
ABC	1405.76	2	702.88	11.58	< 0.0001
A ² C	1950.10	2	975.05	16.06	< 0.0001
B ² C	1112.22	2	556.11	9.16	0.0002
A ³	608.03	1	608.03	10.01	0.0019
B ³	316.32	1	316.32	5.21	0.0238
Residual	9594.05	158	60.72		
Lack of Fit	8616.74	38	226.76	27.84	< 0.0001
Pure Error	977.31	120	8.14		
Cor. Total	48938.67	179			

3.3 Hematite microflotation results

The average hematite depressability as a function of the depressant dosage is shown in Figure 5. The hematite average depressability in the absence of the depressants ranged from $10.94 \pm 1.75\%$ to $17.08 \pm 1.75\%$ (at pH 10.5 and 9, respectively). This result indicated a low hydraulic entrainment for the particle size distribution adopted in the experimental set up, which agrees with the results found by Guimarães Júnior *et al.* (2015). Average hematite depressability with CS ranged from $59.09 \pm 0.75\%$ to $91.62 \pm 0.10\%$, while with MS ranged from $84.53 \pm 1.28\%$ to $96.58 \pm 0.14\%$, and from $80.49 \pm 1.52\%$ to $93.93 \pm 0.66\%$ with SS. The highest average

hematite depressability ($96.58 \pm 0.14\%$) was obtained with 40 mg/L of MS and pH 9. It is possible to notice that hematite flotability remained relatively stable for dosages greater than or equal to 10 mg/L for MS and SS.

Yang and Wang (2018) suggested that although AM is more readily adsorbed onto a hematite surface, AP plays a more important role than AM in determining the starch depressing ability in hematite flotation. However, the same authors affirm that when comparing AP from different sources, the one with more, longer branches would have stronger ability to depress hematite. This observation partially explains the SS

results, but not the MS ones. One possible explanation for the MS results could be its AM/AP ratio, with AP content around 37% and 9% higher than CS and SS, respectively. AP depressing ability is due to the presence of hydroxyl groups in the chemical structure of the starch (Pinto *et al.*, 1992; Pavlovic and Brandao, 2003). According to Veloso *et al.* (2018), CS shows higher depressing ability for hematite at pH 10.5, which is consistent with the results found with high dosages of the depressant (20 and 40 mg/L). At these conditions, the average hematite depressability was $81.27 \pm 1.68\%$ and $91.62 \pm 0.10\%$, respectively.

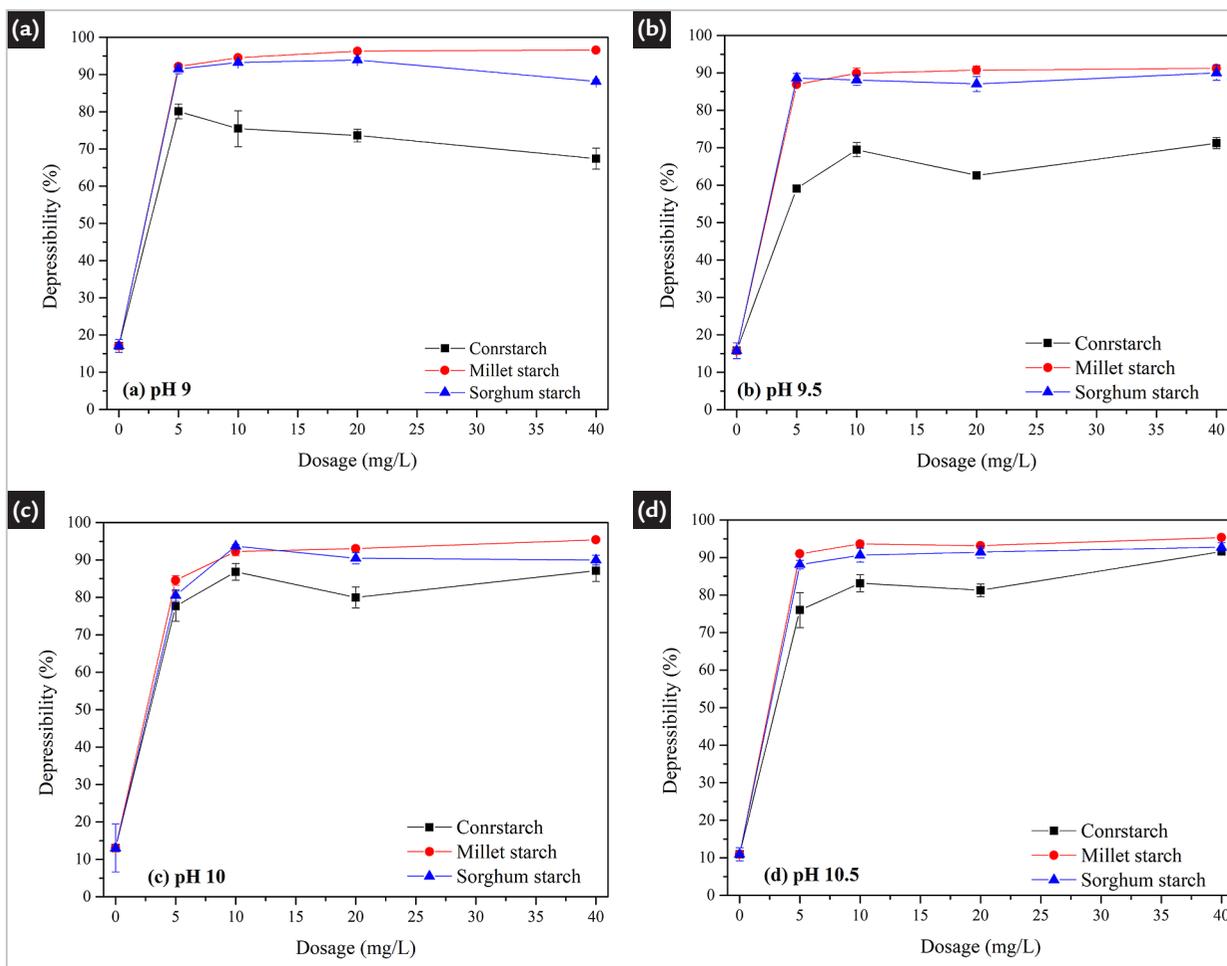


Figure 5 - Average hematite depressability as a function of the depressant dosage at four different pHs: (a) 9, (b) 9.5, (c) 10, and (d) 10.5.

Figure 6 shows the 3D model for hematite flotation. Table 5 presents the ANOVA analysis for the hematite results. The model F-value of 122.59 implies the model is significant. There is only a 0.01% chance that an F-value this large could occur due to noise. However, the adoption of a cubic equation to model the experimental data could lead to the appearance of an inflection point and local minimum and maximum points. In

this particular case, the local minimum point was observed for all tested starches around the dosage of 30 mg/L. For the best knowledge of the authors, this point should not be taken into consideration, since this dosage was not tested. Abaka-Wood *et al.* (2018) performed flotation tests with Australian hematite and quartz in a standard 250 cm³ Denver microflotation cell. The authors suggested that the hematite flotation response is

pH dependent. Unexpectedly, for the found results, the pH was not significant, suggesting that the pH variation did not affect the hematite depressability for all tested starches. The first order interactions were significant for all three parameters, which indicates that the hematite depressability is not sensible to the pH variation alone, but it is sensible to pH variations associated with variations in any of the other two parameters.

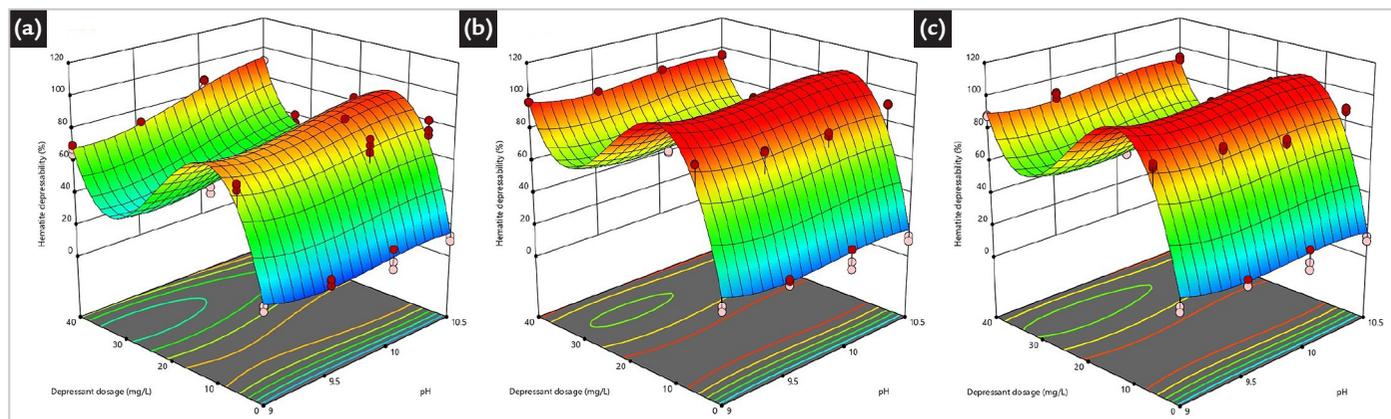


Figure 6 - Hematite response surface for (a) cornstarch, (b) millet starch, and (c) sorghum starch.

Table 4 - Models fit summary for hematite flotation results.

Model	Sequential p-value	Lack of Fit p-value	Adjusted R ²	Predicted R ²	Conclusions
Linear	< 0.0001	< 0.0001	0.3151	0.2915	
2FI	0.7297	< 0.0001	0.3064	0.2612	
Quadratic	< 0.0001	< 0.0001	0.6626	0.6421	
Cubic	< 0.0001	< 0.0001	0.9345	0.9297	Suggested
Quartic	< 0.0001	< 0.0001	0.9876	0.9856	Aliased

Table 5 - ANOVA for Cubic model for the hematite flotation. Insignificant model terms are.

Source	Sum of squares	Degrees of freedom	Mean square	F-value	p-value
Model	1.523E+05	21	7254.38	122.59	< 0.0001
A-pH	76.81	1	76.81	1.30	0.2563
B-Depressant dosage	47721.68	1	47721.68	806.46	< 0.0001
C-Starch type	5622.75	2	2811.37	47.51	< 0.0001
AB	668.14	1	668.14	11.29	0.0010
AC	700.80	2	350.40	5.92	0.0033
BC	388.91	2	194.46	3.29	0.0400
A ²	341.99	1	341.99	5.78	0.0174
B ²	54970.31	1	54970.31	928.95	< 0.0001
ABC	367.93	2	183.97	3.11	0.0474
AB ²	31.82	1	31.82	0.5376	0.4645
B ² C	819.39	2	409.69	6.92	0.0013
A ³	393.63	1	393.63	6.65	0.0108
B ³	40220.53	1	40220.53	679.69	< 0.0001
Residual	9349.56	158	59.17		
Lack of Fit	8737.72	38	229.94	45.10	< 0.0001
Pure Error	611.85	120	5.10		
Cor. Total	1.617E+05	179			

The found results suggest that starch adsorption occurs onto the surface of both minerals. However, at the tested pH range, the interaction between quartz and amine is stronger, making it difficult (maybe even preventing) the starch adsorption onto this mineral surface (Shrimali and

Miller, 2016). According to Lima *et al.* (2013), high amine dosages lead to the formation of clathrates, a compound in which molecules of one species occupy the empty spaces in the lattice of the other species, therefore resulting in the depression of hydrophobic minerals. The authors showed that high dosages

of monoetheramine changed the quartz surface properties, resulting in an interaction between CS and the collector, reducing the quartz contact angle from 62° to 43°, leading to quartz depression. This interaction between CS and amine could partially explain the results found with high dosages of this depressant.

4. Conclusions

Millet starch showed high quartz floatability and hematite depressability, which could be explained due to its high amylopectin content ($67.10 \pm 0.62\%$). Millet starch shows a great potential to replace cornstarch as the standard hematite

depressant. With higher extraction yield ($38.6 \pm 0.45\%$) and high depressability results, the industrial adoption of millet starch could lead to operational costs reduction. On top of that, millet starch is less sensitive to pH variations, which

could lead to better results when facing industrial fluctuations in the process. This was the first attempt to introduce millet starch as a novel, sustainable depressant in the mineral industry, in order to move away from corn.

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