Polyacrylamides in reverse cationic iron ore flotation: bench scale study

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Abstract

A bench scale study was carried out to investigate the influence of polyacrylamides as co-depressant in a blend with starch depressant on the reverse cationic iron ore flotation. The results indicated that the use of polyacrylamides as dual function polymer should be an alternative to improve the flotation process. These polymers act as a depressant and flocculant reagent, decreasing the mechanical entrainment of hydrophilic iron minerals, forming hydrophobic aggregates. Also, the use of polymers as a co-depressant could prevent the possibility of clathrate formation caused by starch and amine overdose. The results obtained showed that cationic and non-ionic polyacrylamides improve the selectivity when applied at low dosages. Only the slightly charged anionic polyacrylamide yielded good results in flotation tests. The design of experiments carried out with non-ionic polyacrylamides showed that flotation results was were affected by the physico-chemical conditions of the system, such as pH, flocculant molecular weight and amine type.

keywords: iron ore, flotation reagents, polyacrylamides, bench scale.

1. Introduction

The iron content in the final tailings of industrial reverse cationic circuits is consistently high. Typical $Fe_{tailings}/SiO_{2concentrate}$ ratios reach the value of five or even higher.

Silva *et al.* (2017) reported the results of the revaluation of a mechanical cells circuit of reverse iron ore flotation. Silica content in the final concentrate stayed at 2.1% and the iron content in the tailings dropped from 11.0% to 9.8%. The result was economically significant and other benefits, such as decrease in amine consumption and reduction in the circulating load, were achieved. Nevertheless, when one considers that the requisites for concentration are (i) liberation, (ii) differentiability, (iii) and dynamic separability (Peres and Araujo, 2009), similar grades of silica in the concentrate and iron in the tailings could be expected, considering the high degree of the liberation characteristic of itabirite ores. High iron loss in the tailings represents economic and environmental impacts.

The differential property in flotation is the hydrophobic/hydrophilic character of the mineral surfaces induced or modified by the adsorption of reagents. The dynamic separability is the composition of forces in the equipment capable to impart different paths for the particles as a response to the differential property. Flotation columns, large volume cells (tank cells) and pneumatic cells improved the concentrators performance to a certain degree. Novel reagents and the combination of traditional reagents represent a wide field open for investigation (Zhang *et al.* 2019).

Several polymers have been used as depressants and flocculants in iron ore concentration, such as starch, guar, dextrin, and carboxymethylcellulose (Nakhaei and Irannajad, 2017). Turrer

and Peres (2010) performed laboratory flotation tests and examined several depressants, including 6 CMCs, 3 lignosulphonates, 1 guar gum and 4 humic acids. It was observed that only two polymers (one carboxymethylcellulose and guar gum) yielded similar performance in comparison with starch. A novel hematite depressant, the macromolecule polymaleic anhydride-triethylenetetramine (PMTA), was reported by Zhang *et al.* (2017). Multiple carboxyl as chelating groups and multiple amines as hydrophilic groups were introduced in a molecule structure. Using dodecylamine as collector at pH 11, the flotation recovery achieved was 98.29% while starch yielded recovery of 95.35%.

The water drainage from flotation froths results in the entrainment of fine hydrophilic particles. Polymers that play the dual function of depressant and flocculant can reduce the entrainment by particle aggregation, reduction of flotation retention time and decrease of water recovery in the froth as discussed in Liu *et al.* (2006).

The formation of non-selective flocs will prevent the concentration and Liu *et al.* (2006) affirm that, if the polymer depressant acts as a dual function by selectively turning gangue minerals hydrophilic and selectively flocculating them, it will be a better depressant than the one which only acts as a depressant.

According to Weisseborn (1995), the advantages of starch with respect to polymers are higher selectivity and stronger depressant action. Nevertheless, high dosages of starch and collector may favor clathrate formation within the amylose helical structure (Lima *et al.*, 2013). Friend and Kitchener (1973) suggested that the previous addition of a selective reagent, such as corn starch, would flocculate the hematite particles, and consequently, facilitate the subsequent polyacrilamide adsorption.

Many studies addressing applications of dual polymers in flocculating system were carried out. The action of dual polymer systems is more efficient than the application of a single flocculant, according to Gregory and Barany (2011). The sequence of addition of the polymers is important and the pre-adsorbed polymer can produce two important effects: providing adsorption sites for the second polymer or causing a more extended adsorbed conformation as a result of 'site blocking'. Yang *et al.* (2019) tested a two-step flocculation process on fine iron ore tailings. Using an anionic or nonionic flocculant as primary flocculant and cationic flocculant as secondary improved the water recovery rate of the slurry and produced a full re-growth of flocs. Lu *et al.* (2016) using also two-step flocculation system improved the dewatering of fine coal tailings. The performance depends on the various flocculant combinations and their order of addition to the slurry.

Lapointe and Barbeau (2017) studied the dual polymer system to improve the flocculation of water samples. They examined the synergic effect of the combination of an activated starch-based polymer with polyacrylamide. The tests showed that the polyacrylamide dosage was strongly reduced, and the injection sequence was not a critical parameter so a blend solution was proposed to simplify the preparation and injection of the polymers.

The first report of the use of a dual polymer system acting as depressant in bench scale iron ore cationic flotation experiments came from Turrer *et al.* (2007). The author used anionic, cationic and nonionic polyacrylamide as partial replacement for starch. Significant increases in the metallurgical recovery of 5.5% and 7.8% were achieved with cationic and non-ionic polymers, respectively. So it represents a wide field open for investigation.

So, the objective of this investigation was to improve the flotation indexes by applying high molecular weight flocculants as co-depressant in the reverse cationic iron ore flotation.

2. Materials and methods

An Itabirite ore sample collected at Casa de Pedra mine, in the Iron Quadrangle, Brazil, was utilized in the experiments. X-ray fluorescence was used for the determination of: Fe, SiO_2, P, Al_2O_3, Mn, TiO_2, Na_2O, K_2O, CaO and MgO. The LOI (loss on ignition) content was determined by calcination. Wet sieving was carried out in a Rotap apparatus with screen openings of 0.150 mm, 0.106mm, 0.074 mm, 0.053mm, 0.044 mm, 0.038 mm and 0.023 mm, 0.017 mm, 0.012 mm, 0.008 mm and 0.006 mm by a Malvern particle analyzer. The products were dried and weighed on an analytical scale.

The flotation tests were conducted in a Denver laboratory cell, model 5202, in a 2 L vessel. The solid content in the pulp was 60%. After agitation for homogenization the reagents were added according to the sequence starch, co-depressant, collector with conditioning times of 5 min, 3 min, and 1 min, respectively. The pH was adjusted during the conditioning and flotation was carried out at pH = 10.5 as per indication of exploratory tests. The solid concentration was adjusted to 40% just prior to flotation. The amine dosage was 40 g/t and starch dosage was 600 g/t.

A complete design of experiments was performed to provide information on the influence of selected variables on the flotation response: (i) pH (ii) molecular weight of co-depressant, (iii) type of starch (usual or low amylose), (iv) type of etheramine (mono or di). Each variable was evaluated at two levels. The selected responses were: $\%Fe_{tailings}$, $\%SiO_{2concentrate}$, metallurgical recovery, and Gaudin selectivity index.

3. Results and discussion

The result of the chemical analysis of the ore are presented in Table 1.

Table 1 - Chemical analysis of the iron ore.

Fe	SiO_2	Al_2O_3	P	PPC	Mn	CaO	MgO	TiO_2
43.7	32.6	0.8	0.03	1.2	0.2	0.099	0.089	0.06

The result of the size analysis is presented in Figure 1. The analysis indicates that 38% of the material is < 38 µm and the coarser fraction (> 150 µm) is 6.91%.

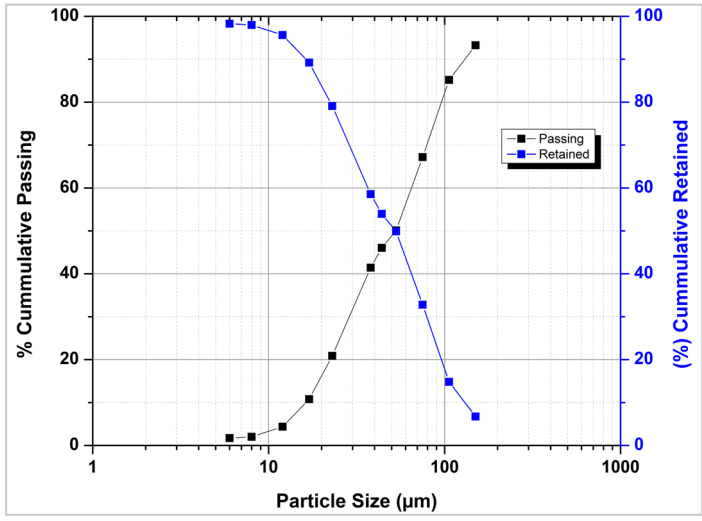

Figure 1 – Particle size analysis of iron ore sample.

Figure 2 shows the results of Gaudin selectivity index, metallurgical recovery, $\%Fe_{tailings}$, $\%SiO_{2concentrate}$ achieved with anionic polyacrylamides. Reagent A110 was the only polyacrylamide which yielded slightly better results than the standard test. The highest value obtained for Gaudin selectivity index were 4.19, for the dosage of 10 g/t.

Regarding metallurgical recovery, the best results were obtained with the dosage 10 g/t. Values of 90.64% and 90.48% were achieved with polyacrylamides A110 and A130, respectively. All the results for polyacrylamide A130V were inferior to those of the standard test (85.77 %).

The $\%Fe_{tailings}$ achieved for the standard test was 16.34%. All the results obtained for polyacrylamide A130 were below this value. The best result was 13.43%, for the dosage of 10 g/t. Polyacrylamide A110, at the dosage of 10 g/t yielded 11.91%. Polyacrylamide A130V did not achieve good results.

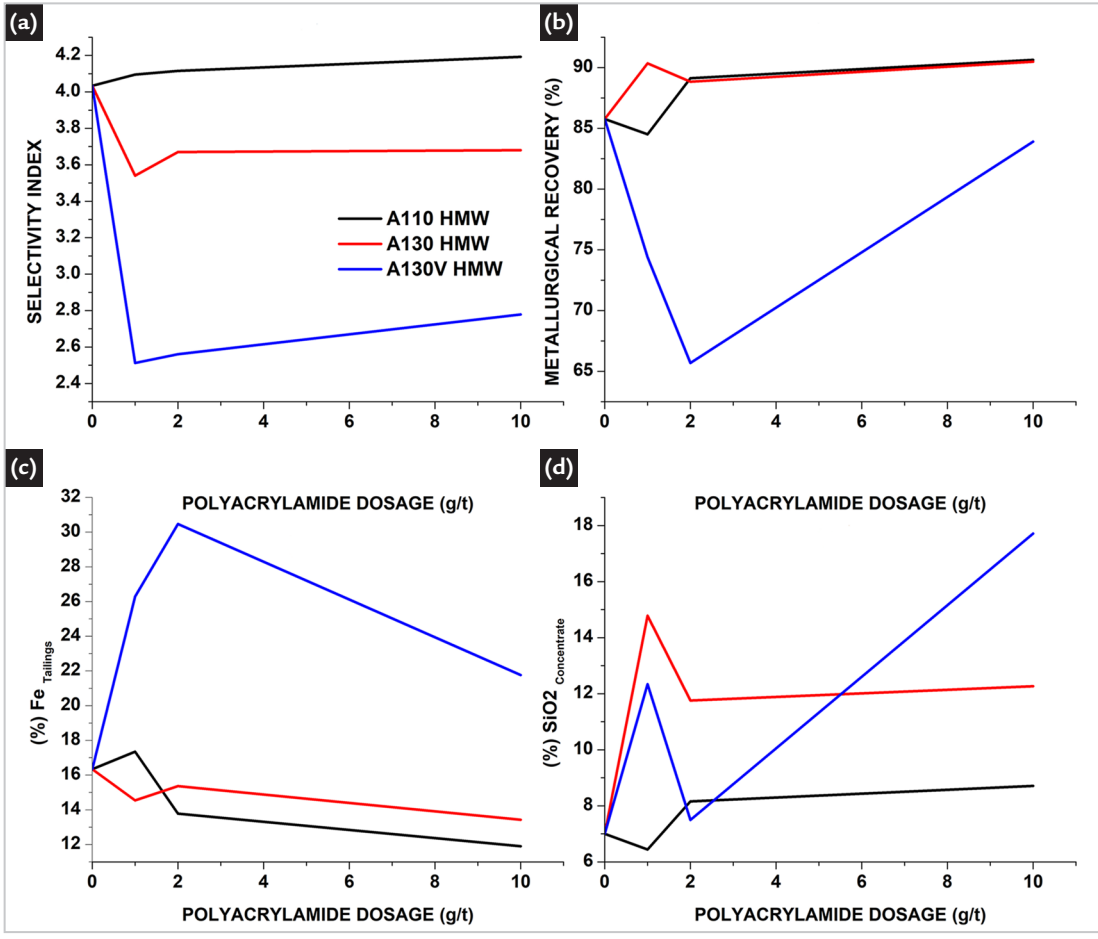

Figure 2 – Flotation results of anionic polyacrylamides (a) selectivity index (b) metallurgical recovery (c) $\%Fe_{tailings}$ (d) $\%SiO_{2concentrate}$.

For $\%SiO_{2concentrate}$, only polyacrylamide A110 yielded a value (6.44% at 1 g/t) below that of the standard test (7.0%).

The results obtained are in accordance with Ding and Laskowski (2007), who carried out batch flotation tests and confirmed that some polyacrylamides performed very well in the coal reverse flotation process. They concluded that polyacrylamides with a different degree of anionicity responded differently and gangue flotation could only be possible with the addition of the polymers with lower degree of anionicity. Also Dwari *et al.* (2018) reached similar results in hematite flocculation. With the increase in ionicity, the negatively charged particles were electrostatically repelled by the anionic polymer.

Figure 3 shows the results of Gaudin selectivity index, metallurgical recovery, $\%Fe_{tailings}$, $\%SiO_{2concentrate}$ using cationic polyacrylamides.

The different reagents present similar trends concerning the Gaudin selectivity index. The best result was obtained at polyacrylamide dosage of 2 g/t, and the worst at 10 g/t. The highest values were 5.29, 6.64 and 4.44 for C492 HMW, C498 HMW and C492 superfloc, respectively. The result for the standard test is 4.03.

The increase in cationic charge and in molecular weight caused an increase in the Gaudin selectivity. The higher cationic charged polymer, C498 HMW, provided the best result in selectivity. Liu *et al.* (2007) achieved a similar trend in diaspore depression. The depressant abilities of the cationic polyacrylamides followed the order of cationicity; the higher the cationic charge, the higher the depressant effect.

Comparing the molecular weight, C492 HMW achieving better results than C492 Superfloc, which has lower molecular weight. The use of C492 superfloc provided a slight increase in the Gaudin selectivity index compared with the standard value. An unexpected result was observed in the case of the C492 superfloc at the dosage 1 g/t.

The values of the indeces for metallurgical recovery, $\%Fe_{tailings}$, $\%SiO_{2concentrate}$ were superior than those of the standard test for almost all polymer dosages. The best results were obtained at low dosages.

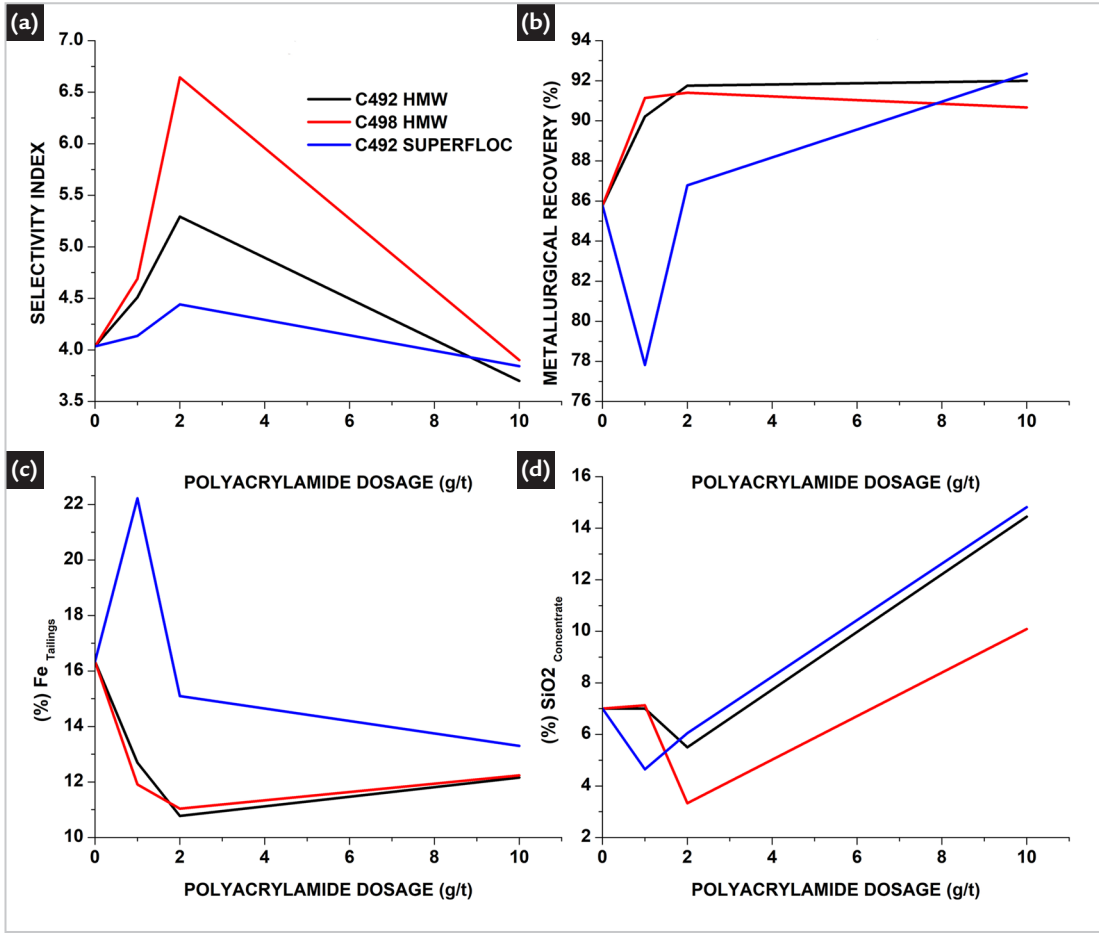

Figure 3 – Flotation results of cationic polyacrylamides (a) selectivity index (b) metallurgical recovery (c) $\%Fe_{tailings}$ (d) $\%SiO_{2concentrate}$.

Figure 4 shows the results of Gaudin selectivity index, for metallurgical recovery, $\%Fe_{tailings}$, $\%SiO_{2concentrate}$ using non-ionic polyacrylamides.

Both tested polyacrylamides showed similar action regarding the Gaudin selectivity index. The best results obtained were 7.25 and 8.42 for Magnafloc 351 and Magnafloc 333, respectively, for the dosage 2 g/t. The increase in the molecular weight provided an increase in the Gaudin selectivity index.

The increase in molecular weight promoted an increase in the Gaudin selectivity. Comparing the molecular weight, Magnafloc 333 achieving better results than Magnafloc 351, which has lower molecular weight.

Both non-ionic polyacrylamides showed similar effects on metallurgical recovery and $\%Fe_{tailings}$, but Magnafloc 351 yielded the worst $\%SiO_{2concentrate}$ index.

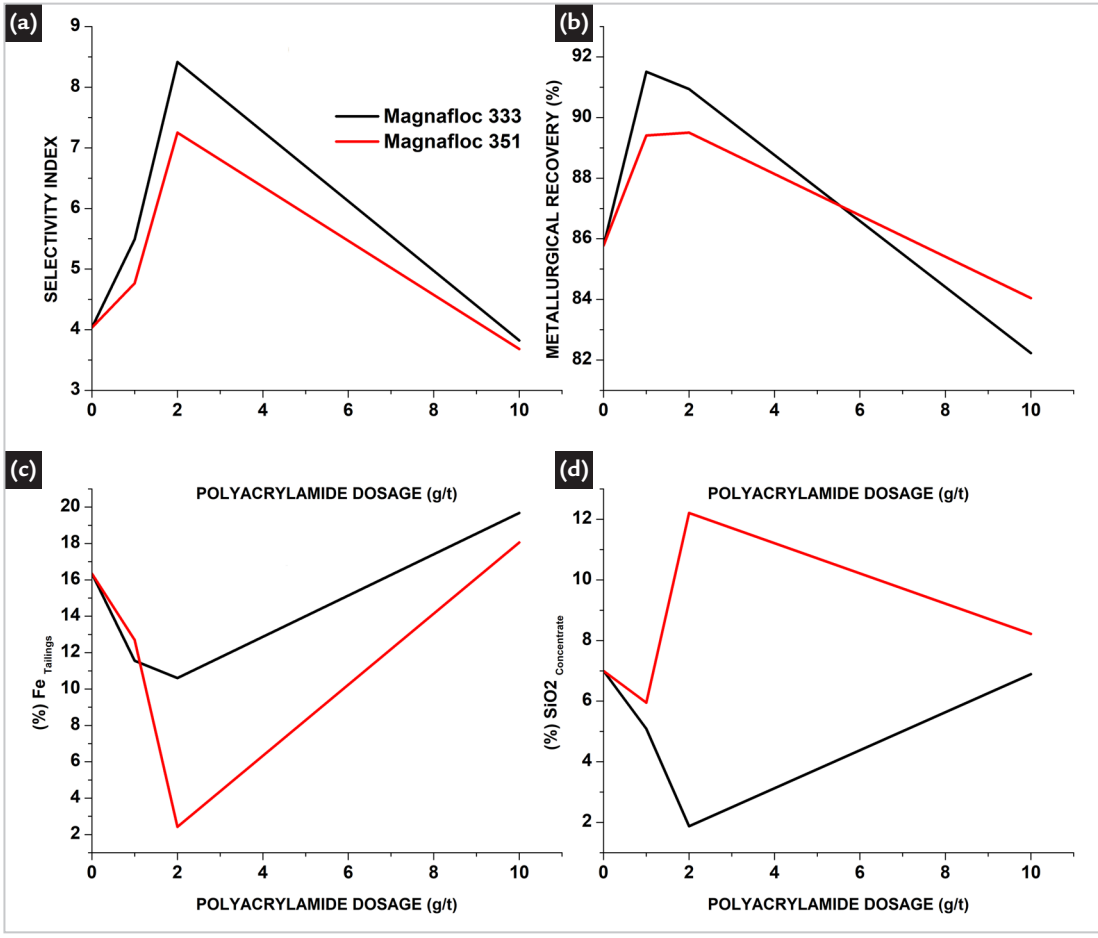

Figure 4 – Flotation results of non-ionic polyacrylamides (a) selectivity index (b) metallurgical recovery (c) $\%Fe_{tailings}$ (d) $\%SiO_{2concentrate}$.

A design of experiments (DoE) was applied to investigate the influence of some independent variables on the flotation response. According to Matos *et al.* (2019), the design of experiments provides a structured way to conduct flotation tests, with a small number of tests. The change of independent variable is under control providing a controlled evaluation of their influence on the response. Tudu and Mandre (2017) successfully used this technique to evaluate the response of selective flocculation of different variables as pulp density, pH and flocculant dosage.

The investigation aimed to provide preliminary information of variation of pH, amine type, starch type, and polyacrylamide molecular weight on the flotation results. Non-ionic polyacrylamides were selected for this stage, since they provided the better results than those reported in previous studies (Turrer, 2010; Oliveira, 2016).

The evaluated factors and levels tested are presented in Table 2.

Table 2 - Evaluated variables and levels tested.

Variable	Levels	
	+	-
pH	9.5	10.5
Molecular weight	MAG 333	MAG 351
Amine type	Mono-amine	Di-amine
Starch type	100% amilopectin	75% amilopectin

Figures 5 and 6 present the main effects and the interaction among factors pH, molecular weight, amine type, starch type for selectivity index.

The main effect plots also supply information on the relative significance of the parameters on the flotation response. In the main effect plots, if the line is approximately horizontal, then the parameter has no significant effect. The parameter with the highest inclination line will have the most significant effect. So, the analysis of the main effects considering adjusted means shows that high pH levels yield higher floatability levels in comparison with low pH levels and that this floatability increases with the high molecular weight, using diamine type and for low amylopectin content in starch as observed in Figure 5.

The best results were obtained at pH=10.5, using Magnafloc 333, di-amine and starch with 75% amylopectin.

Interactions occur where the impact of a parameter is dependent on the setting of a second parameter. DoE experiments can identify interactions as many parameters are changed simultaneously in the design. The interactions plot in Figure 6 suggests that there might be significant interactions due to pH*amine type, molecular weight*amine

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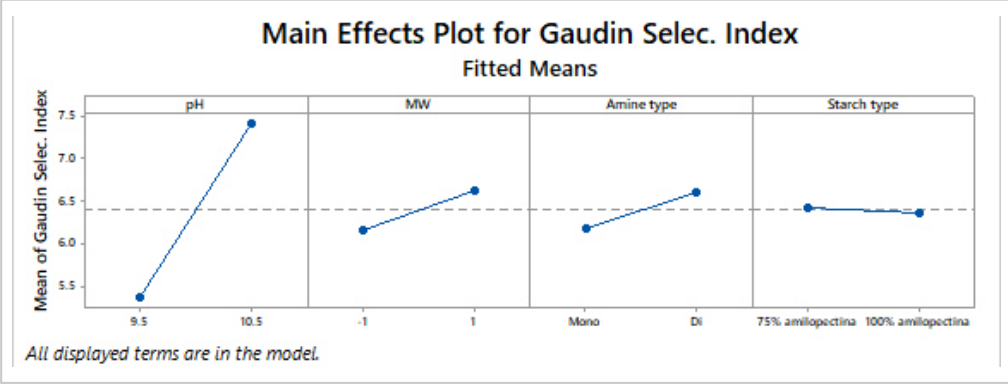

Figure 5 – Main effects in selectivity index in relation of pH, molecular weight, amine type and starch type.

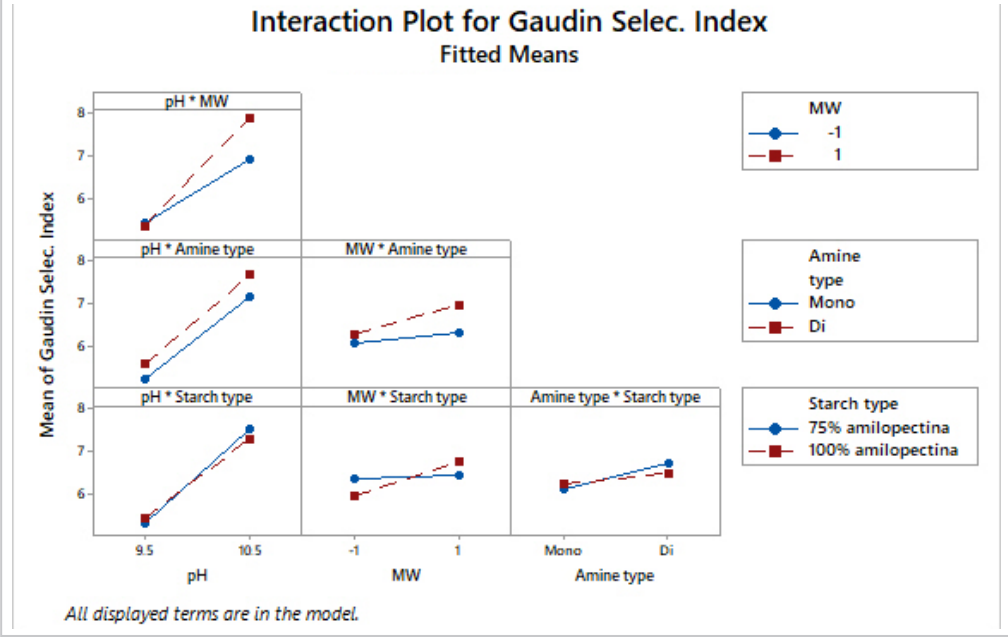

Figure 6 – Interaction of Gaudin selectivity index between pH, molecular weight, amine type and starch type.

4. Conclusions

Bench scale tests of reverse cationic flotation of iron ore have been conducted to study the influence of polyacrylamides as co-depressant in a blend with starch depressant and the following conclusions can be drawn under the conditions used in this study:

• It was observed that the best flotation results were obtained with non-ionic and cationic polyacrylamides.

• Among the cationic polyacrylamides, higher charge and higher molecular weight yielded better flotation results.

• Anionic polyacrylamides did not promote good results in flotation, only the polymer with the lowest charge promotes slight improvement in the results

• The non-ionic polyacrylamides provide the best flotation results in bench scale flotation

• Among non-ionic polyacrylamides, higher molecular weight yielded better results, which was confirmed in the design of experiments section

• Considering the design of experiments, the results were significantly affected by pH, amine type and polyacrylamide molecular weight and the best flotation results were obtained with pH=10.5, polyacrylamide Magnafloc 333, di-amine and starch with 75% amilopectin content.

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