

The importance of temperature control in caustic cleaning routines in a Bayer plant

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Abstract

In Bayer process, extracted alumina is 50 % of dry bauxite weight. The remaining consists of impurities, such as, iron oxides, silicon, calcium, titanium and organic material. Some of these impurities are dissolved and they can precipitate in the form of complex compounds in different parts of the Bayer process. This study shows: 1) The importance of temperature control for the efficiency of caustic cleaning. If the temperature of the solution falls below 75 °C there will be formation of complex compounds ($C_4ACO_2H_{11}$) decreasing the efficiency of the solution; 2) Cleaning routines where the solution is prepared with mixing virgin caustic soda and water; the temperature control is essential because this crust in the tissue is formed predominantly by sodium tricalcium aluminate (TCA); 3) In caustic cleaning routines where the solution is prepared using a mixture of virgin soda and spent liquor dissolving capacity is significantly lower. The results of the studies show that increasing the caustic cleaning time will not be a compensation factor. In this case, the temperature control becomes even more important.

Keywords: Bayer process caustic cleaning; temperature control in caustic cleaning.

1. Introduction

Most alumina refineries clarifies bauxite digested pulp using a combination of sedimentation and filtration. More than 99 % of the sludge solids are removed in the decanter, while the remaining solids in the overflow of the decanter are removed by filtration under pressure. The study presents the characterization of mud a decanter, summarizes the process of crust formation in Bayer plant, comments the filtration process and caustic cleaning routines used in the refinery. Caustic cleaning processes were simulated in the laboratory and applied experimental design to measure the influences of variables.

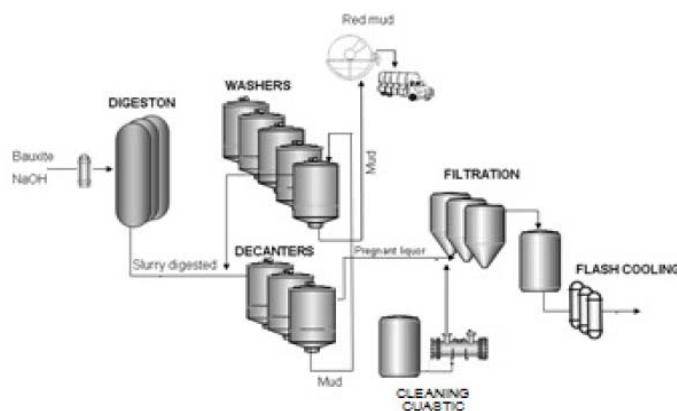


Figure 1. Summary of process.

2. Materials and methods

2.1. Mud characteristic of decanters

Analysis of the decanters overflow of mud were made using scanning electron microscope and a system of dispersion spectroscopy.

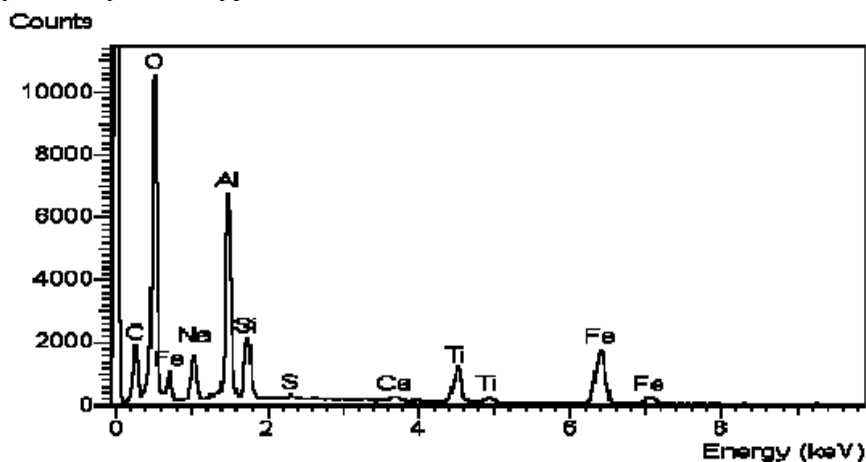


Figure 2. Analysis of the total area. C = carbon, O = oxygen, Na = sodium, Al = aluminum, silica Si =, S = sulfur, Ca = calcium, titanium and Ti = Fe = iron.

2.2. Crust formation in Bayer Plant

In the Bayer process extracting alumina is 50 % of the dry bauxite. The rest consists of impurities such as oxides of iron, silicon, calcium, titanium and organic matter. Some of these impurities are dissolved and can precipitate in the form of complex compounds in different parts of the Bayer process. These compounds and some salts contained in industrial water form different types of crusts in the equipment causing problems in pipes, heat exchangers and tanks.

2.3. Factors influencing the speed of crusting

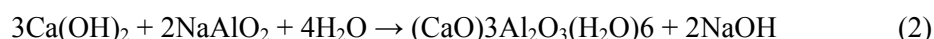
The operating parameters that determine the composition and rate of formation of crust in the Bayer process are: 1) the composition of bauxite and liquor; 2) temperature of the liquor streams and pulp; 3) stirring so the tanks and speed of liquor / pulp in the pipes; 4) the cooling water of the composition and the lime slurry.

2.4. Filtration

The principle of filtration is to cake formation where the two filtered flows through porous media in series (the cake and the filter medium). The cake grows continuously throughout the operation input by the suspension, and the pie properties depend on the position relative to the filter medium and the filtration time. Of the seven production lines of Hydro Alunorte five use of vertical pressure filters, which are simultaneously fed by the overflow of the decanter and flow of TCA (sodium tricalciumaluminato) system called body-feed. For a good filterability operates with an excess of TCA.

2.5. TCA (sodium tricalciumaluminato)

TCA is prepared by reaction of lime or lime quenched with sodium aluminate solution, usually with pregnant liquor prepared according to the following reactions:



Calcium aluminate hydrate compounds ($\text{C}_4\text{ACO}_2\text{H}_{11}/\text{C}_4\text{AH}_{13}$) form readily when $\text{Ca}(\text{OH})_2$ is added to sodium aluminate liquor at low temperatures, but decompose TCA when the reaction temperature exceeds 50 °C. These compounds have layers of hydroxyl ($\text{Ca}_2\text{Al}(\text{OH})_6$) that can incorporate various carbonate anions (CO_3^{2-}).

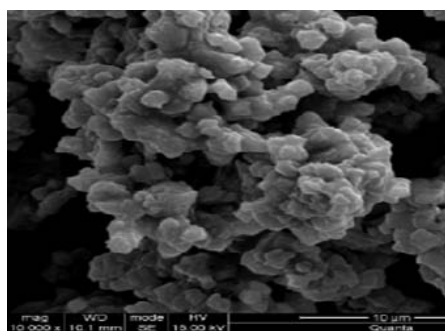


Figure 3. Electron micrograph of TCA.

2.6. Caustic cleaning routines

- 1) Filtration/Decanters and mud thickener -The solution is prepared with virgin soda and water and pumped into the heated interior of the filter. Heat exchangers guarantee the maintenance of the temperature in the specification. The quality of the cleaning solution is assessed by ratio alumina/soda. 2) Precipitation area - caustic solution is prepared by mixing virgin soda and spent liquor. The quality of cleaning solution is also evaluated by the ratio alumina/soda.

2.7. Laboratory tests

2.7.1. Laboratory tests with filter cloth using heater plate

Aims to assess removal of alumina and soda filter cloth.

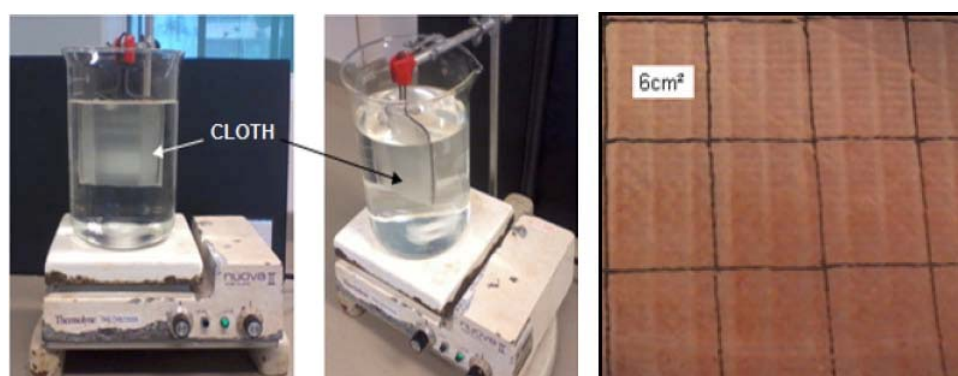


Figure 4. Laboratory apparatus and tissue samples.

2.7.2. Laboratory tests with filter cloth using oven

Aims to assess removal of alumina and soda filter cloth.

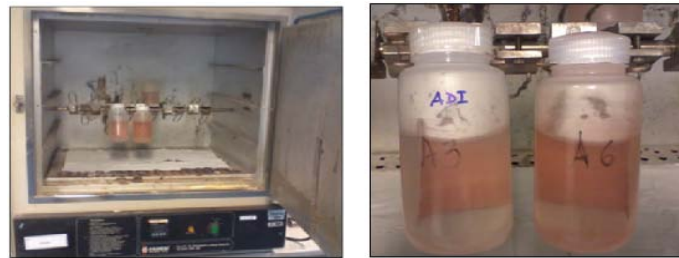


Figure 5. Laboratory apparatus and tissue samples.

2.7.3. Laboratory tests with hydrate crust using oven

Check the performance of two types of washing solutions results in the ratio $\text{Al}_2\text{O}_3/\text{NaOH}$ and dissolution rate.



Figure 6. Crusts hydrate of a thickener.

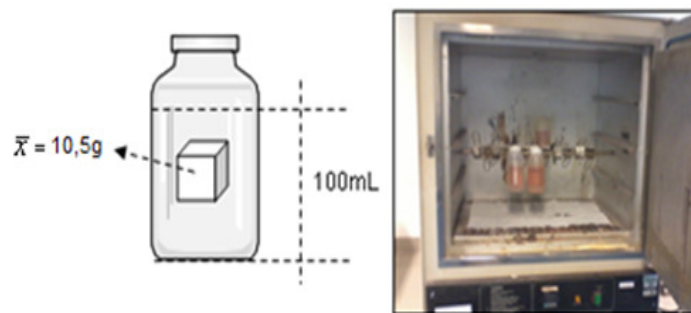


Figure 7. Laboratory apparatus.

Table 1. Chemical analysis of hydrate crust.

Al_2O_3	Fe_2O_3	SiO_2	V_2O_5	Na_2O	P_2O_5	MnO_2
(%)	(%)	(%)	(%)	(%)	(%)	(%)
97.320	0.051	0.509	0.003	2.37	0.0009	0.001

2.7.4. Studies of experiments (DOE)

The experimental design is a powerful tool to study the combined effect of several factors on the response variable of interest. One of the most popular techniques is the factorial design, in which are involved k factors (or variables) each present at different levels.

Table 2. Conditions of experiments to identify the influences in ratio $\text{Al}_2\text{O}_3/\text{NaOH}$ and dissolution rate of hydrate crust using wash solution prepared with virgin soda and water.

Factors	Variation level		
	-	Middle point	+
Caustic concentration (g/L)	300.0	389.6	484.0
Temperature ($^{\circ}\text{C}$)	60.0	75.0	90.0
Cleaning time (h)	1.0	3.5	6.0

Table 3 – Conditions of experiments to identify the influences in ratio $\text{Al}_2\text{O}_3/\text{NaOH}$ and dissolution rate of hydrate crust using wash solution prepared with virgin soda and spent liquor.

Factors	Variation level		
	-	Middle point	+
Caustic concentration (g/L)	301.0	395.2	487.0
Temperature ($^{\circ}\text{C}$)	60.0	75.0	90.0
Cleaning time (h)	1.0	3.5	6.0

2.7.5. Laboratory tests with metallic screen with crust using oven (VPF filter)

Check the capacity of the cleaning solution dissolving a very hard crust considered controlling temperature and concentration in the specification wash in 15 hours.



Figure 8. Metallic screen samples encrusted with mud crust.



Figure 9. Laboratory apparatus.

Table 4. Analysis of this mud crust in the sample of the metal screen.

Al_2O_3	Fe_2O_3	SiO_2	TiO_2	CaO	Na_2O	Na_2O
(%)	(%)	(%)	(%)	(%)	(%)	(%)
39.83	2.90	24.23	3.63	3.78	15.74	2.37

3. Results and Discussion

3.1. Caustic cleaning laboratory tests with filter cloth using sheet metal

Figure 10 shows the Pareto chart that ranks the effects on the ratio $\text{Al}_2\text{O}_3/\text{NaOH}$ in order of significance. The conditions which bars exceed the red line have significant effects. The big influence was the association between the variables caustic concentration and temperature. The isolated variables as well as other associations between variables did not have significant influence.

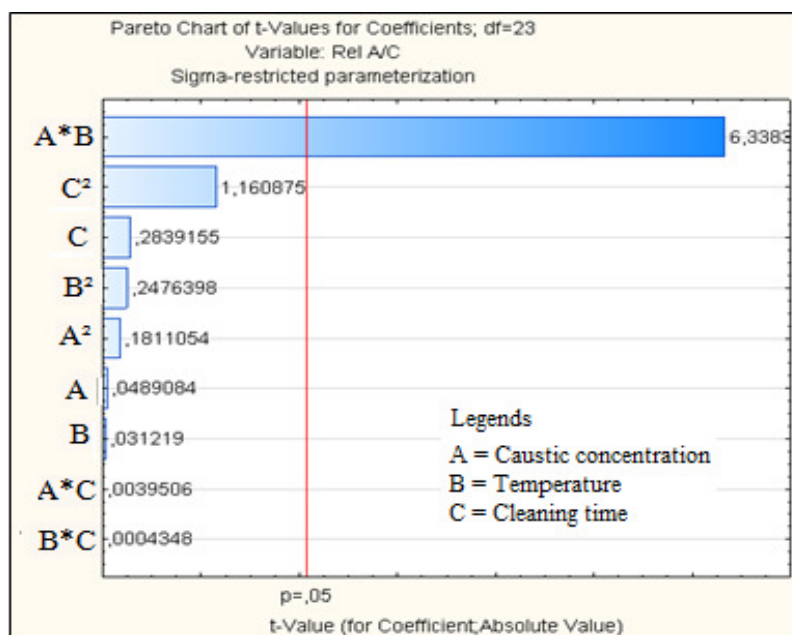


Figure 10. Significant effects on ratio alumina/caustic.

3.2. Caustic cleaning laboratory tests with filter cloth using oven

Figures 11 and 12 show the extraction of alumina and soda samples of the filter cloth. Results:

- Alumina extraction occurred regardless of variation in temperature, caustic concentration and cleaning time;
- An indication of soda extraction was negative with decreasing temperature. This behavior is due to a reaction between $\text{Ca}(\text{OH})_2$ and Bayer liquor at low temperatures forming $\text{C}_4\text{ACO}_2\text{H}_{11}$ compound which consumes NaOH. The lower the caustic concentrations the greater will be the loss of NaOH;
- Whereas the operational routine controls the solution by the ratio $\text{Al}_2\text{O}_3/\text{NaOH}$, the phenomenon observed in item ii) is committed to evaluating the solution.

Figure 13 shows the Pareto chart that ranks the effects on the ratio $\text{Al}_2\text{O}_3/\text{NaOH}$ in order of significance. The conditions which bars exceed the red line have significant effects. As the heated plate test, the major influence was the association between the variables caustic concentration and temperature. The isolated variables, as well as other associations between variables, did not exercise significant influence.

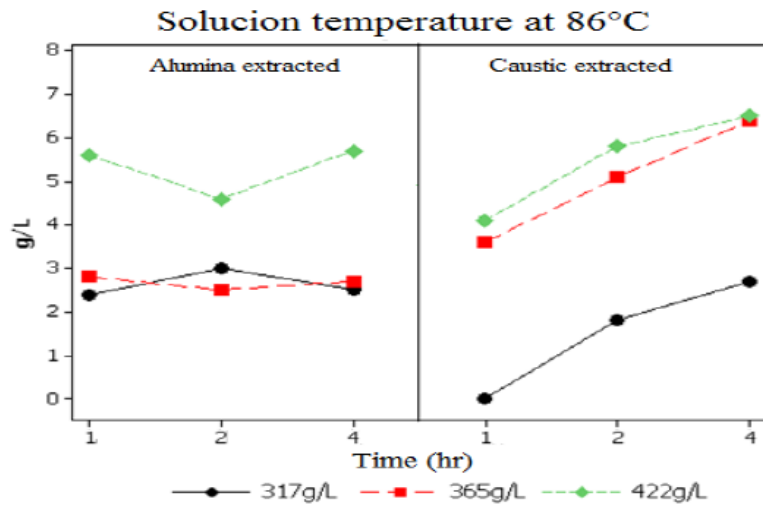


Figure 11. Effect of different conditions of caustic concentration and cleaning time in the alumina extraction and caustic extraction at 86 °C.

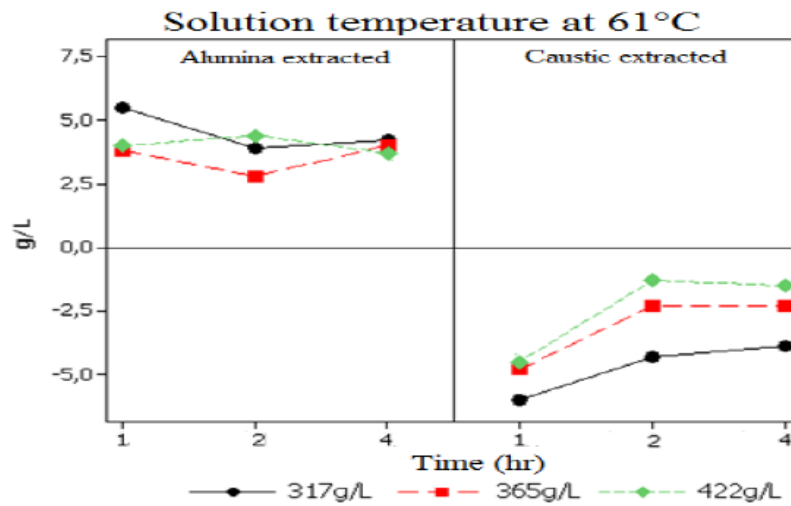


Figure 12. Effect of different conditions of caustic concentration and cleaning time in the alumina extraction and caustic extraction at 61 °C.

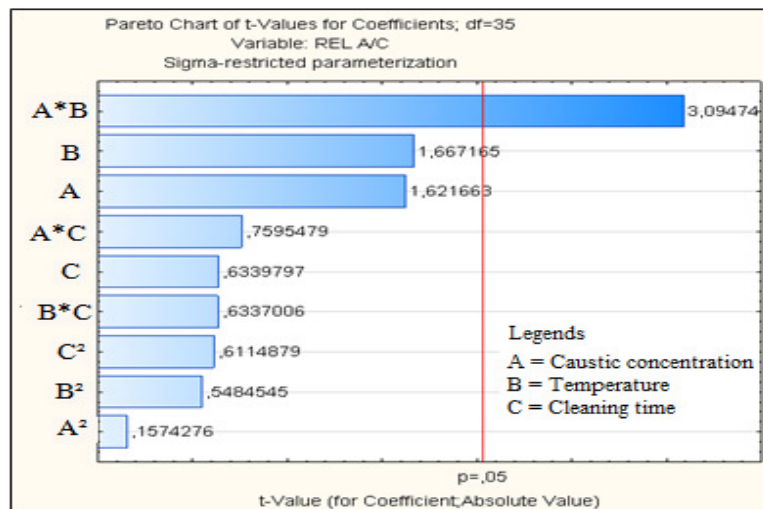


Figure 13. Significant effects on ratio alumina/caustic.

3.3. Caustic cleaning laboratory with hydrate crust using oven

Figure 14 shows the response surface graph for dissolution rate in the wash solution prepared with virgin soda and water, where the maximum response occurred in the region of maximum conditions: 484 g/L, 90 °C and 6 hours. The dissolution rate in the intermediate condition was 90.5 %, higher than the other conditions studied. This confirms the greater influence of the interaction weight caustic concentration and temperature, and the time weight function is to be dissolved and the solution quality. The Figure 15 shows the response surface plot for the ratio $\text{Al}_2\text{O}_3/\text{NaOH}$ with the washing solution prepared with virgin soda and water, where the maximum response region occurred in the conditions under which the solution parameters were maximum value: 484 g/L, 90 °C and 6 hours.

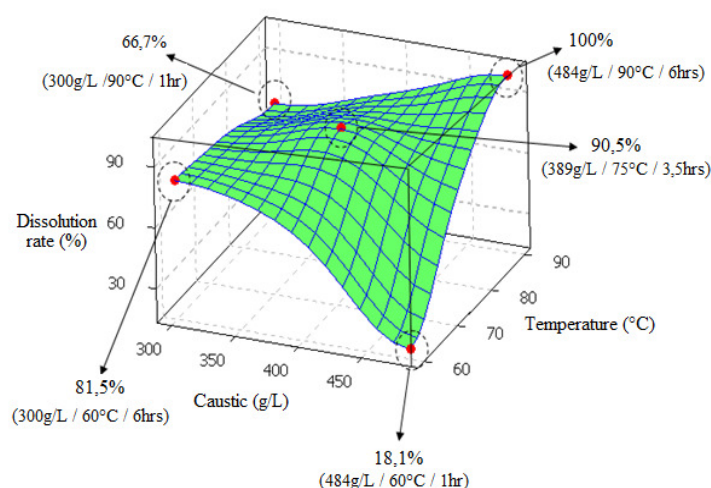


Figure 14. Response surface plot showing the effect of interaction between variables on the dissolution rate of the crust hydrate, with cleaning solution prepared with virgin soda and water.

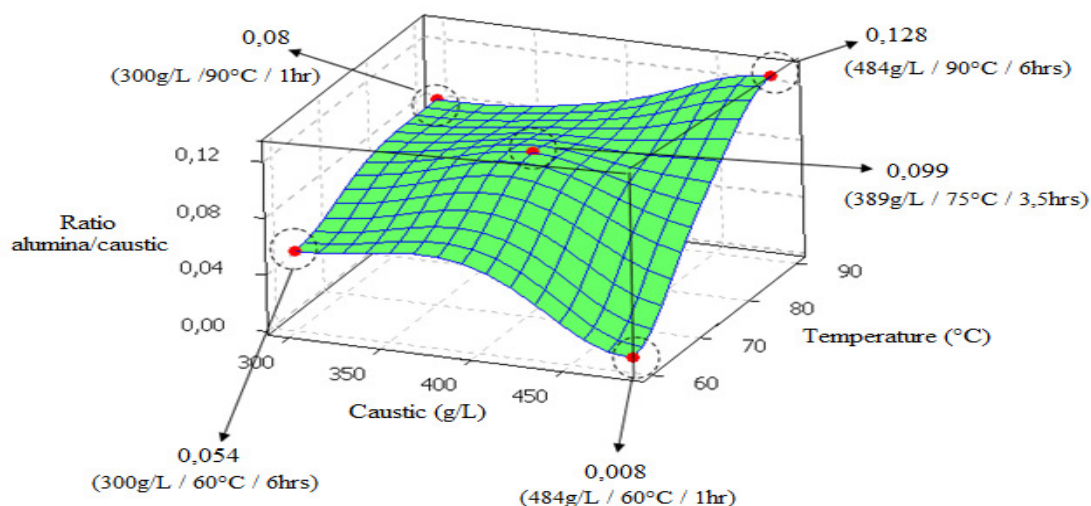


Figure 15. Response surface graph showing the interaction effect of the variables on the ratio alumina/caustic solution, with the cleaning solution prepared with virgin soda and water.

The Figure 16 shows the response surface plot for dissolution rate with the cleaning solution prepared with virgin soda and spent liquor, where the maximum response region occurred in the conditions under which the solution parameters were maximum value: 487 g/L, 90 °C and 6 hours. The dissolution rate in the intermediate condition was 90.8 %, also higher than the other studied conditions. This confirms the greater influence of the interaction weight caustic concentration and

temperature, and the time weight function is to be dissolved and the solution quality. Draw attention to the insignificant dissolution rates in caustic condition, at least when compared to the tests with the prepared solution with virgin soda and water. These results showed that for this type of solution is critical caustic control and temperature values over 395 g/L and 75 °C respectively. The results show that increasing the caustic cleaning time will not be a compensation factor.

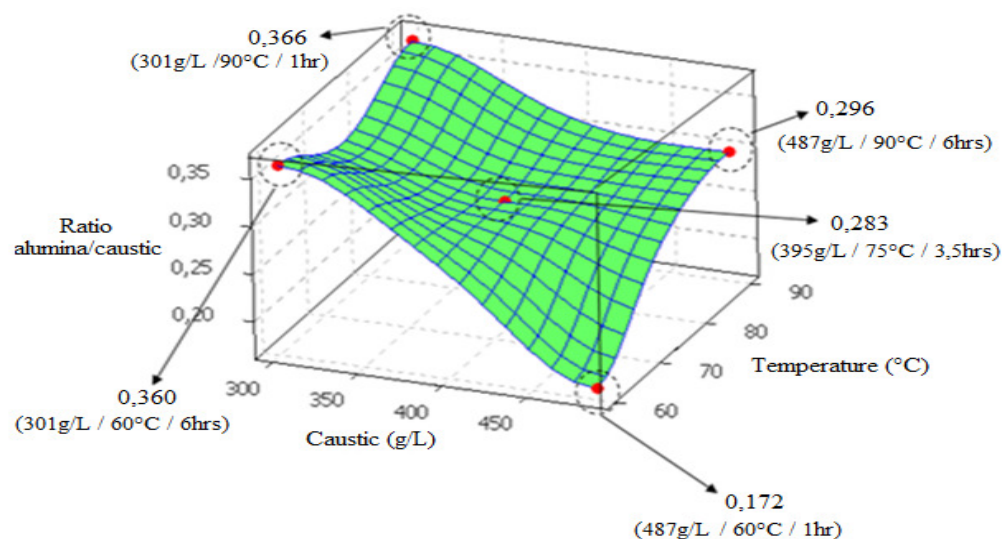


Figure 16. Response surface plot showing the effect of interaction between variables on the dissolution rate of the crust hydrate, with cleaning solution prepared with virgin soda and spent liquor.

Figure 17 shows the response surface plot for the ratio alumina/ caustic with the cleaning solution prepared with virgin soda and spent liquor, where the maximum response region in the conditions in which the solution parameters were maximum value: 301 g/L, 90°C and 1hour. In these conditions, the ratio $\text{Al}_2\text{O}_3/\text{NaOH}$ was 0.366, above the 0,200 specification limit. At intermediate point to the ratio alumina/caustic was 0.283. Only one result was below the 0.200 specification. This result was due to the formation of complex compounds.

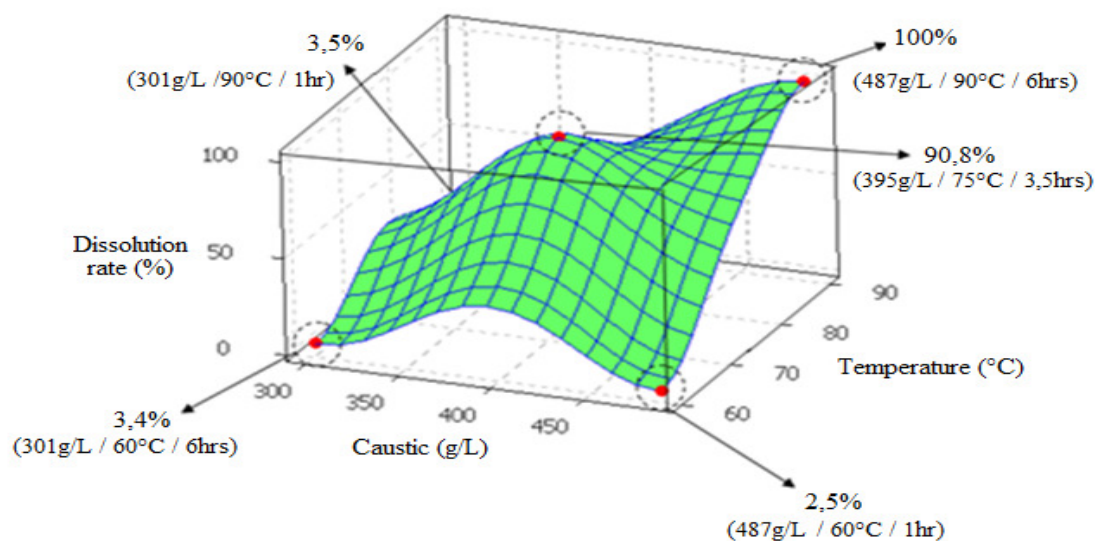


Figure 17. Response surface graph showing the interaction effect of the variables on the ratio $\text{Al}_2\text{O}_3/\text{NaOH}$ solution, with the cleaning solution prepared with virgin soda and spent liquor.

3.4. Caustic cleaning laboratory tests with metallic screen with crust using oven (VPF filter)

The laboratory apparatus does not reproduce in full the cleaning conditions of the area by the absence of flow. Tests showed that a large part of the crust was dissolved. After cleaning, a simple manual manipulation caused the crust gives off the screens. If there was the possibility to reproduce in the laboratory an incidence of flow possibly this crust on the screens would be removed almost entirely. Soda will be lost if the solution temperature is below 75 °C because the formation of compound $C_4ACO_2H_{11}$ reducing the efficiency of the solution. This phenomenon did not occur during cleaning due to strict control of temperature by 90 °C.



Figure 18. Aspects of the conditions of the sample after caustic cleaning wire mesh 15 hours with the total amount of crust removed.

4. Conclusion

Temperature control is fundamental to the caustic cleaning efficiency in a Bayer plant. If the temperature of the solution is much below 75 °C there will be a loss of control of the caustic concentration, it occurs the formation of complex compounds such as $C_4ACO_2H_{11}$ (calcium aluminate hydrate tricalciumaluminate), soda-consuming and greatly decreases the efficiency of the solution. In tissue rejuvenation routines where the cleaning solution is prepared by mixing virgin soda and water, the temperature control is essential because the crust is predominantly present in the tissue formed sodiumtricalciumaluminate (TCA). In cleaning routines where the solution is prepared by mixing virgin soda and spent liquor, the dissolution capacity is significantly lower. In this case, the temperature control becomes even more critical.

5. References

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