

Study of Influences on the Productivity of Bauxite Slurry Hyperbaric Filtration through Box Behnken Design

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Abstract

The Hydro Alunorte refinery receives bauxite slurry used in the alumina production from Mineração Paragominas, both sites located in Pará – Brazil. This bauxite slurry is transported via pipeline and dewatered in hyperbaric filters located in Alunorte. This study was carried out to evaluate the influence of the addition of milk of lime in bauxite slurry on the productivity of the hyperbaric filters considering ranges of pH, densities, and particle size (top size) of the bauxite through the application of the statistical technique Box Behnken Design in order to decrease pipeline wearing rates. The simulation of the filtrations was carried out on a laboratory scale, and the cake formation time was the main outcome, which, in turn, enabled the calculation of the filters' productivity. The analysis of variance applied to the laboratory results allowed a regression model to develop a relationship between the control factors and the response variable productivity with $R^2(\text{adj})$ of 80.56 %. The slurry density had a positive quadratic effect on the model equation, being the factor with the most significant impact on filter productivity. The increase in pH with the addition of milk of lime into the bauxite slurry and higher bauxite top sizes also positively impacted the response variable. On the other hand, more basic pH levels enhanced the negative effect of the top size reduction on productivity. The impact of this factor is nearly negligible at pH close to neutrality. This study will support future experiments on an industrial scale for the decision-making of the most economically viable slurry modification alternative and optimized operational parameters that meet both the pipeline and hyperbaric filters needs.

Keywords: Bauxite slurry, Design of Experiment, Milk of lime, Pressure filtration, Top size.

1. Introduction

Most of the bauxite processed at the Alunorte refinery comes from Mineração Paragominas (MPSA), both sites located in Pará - Brazil. Bauxite is pumped from the mine to the refinery in the form of a slurry with 50 % solids content through a 244 km long pipeline. Once at Alunorte, the slurry is filtered in a plant with 13 hyperbaric filters to raise the bauxite solids content to 86 %, and then directed to the Bayer process digestion stage. The hyperbaric filters are composed of a set of twelve rotating discs inside a cylindrical pressure vessel, where the pressure difference through the cloths is produced with compressed air. The bauxite's filtrate is treated to reduce its solids content and recycled to incorporate the refinery's industrial water supply.

Pipeline integrity studies carried out previously showed an accelerated metal thickness loss, mainly due to erosion and internal corrosion problems caused by bauxite slurry transportation. These problems put impact on decreasing useful lifetime and increasing the frequency of pipeline maintenance. For this reason, it became necessary to evaluate the increase in slurry pH through the addition of milk of lime, as well as to change the particle size (top size) and slurry density, with a high probability of impact on the Alunorte's hyperbaric filters performance.

Widely used in industrial applications, milk of lime was one of the options considered for the slurry pH raising test mainly because it is a product with low cost and high alkaline content. In addition, milk of lime is already commonly used in alumina production through the Bayer process, having benefits in the alumina extraction efficiency, controlling impurities formation from ore, aiding in the pregnant liquor filtering process (TCA coating), and caustic soda loss minimization by bauxite residue [1]. Preliminary studies indicated a consumption of approximately 700 to 1.000 g CaO per tonne of bauxite in order to raise slurry pH from 7.0 to 10.5. This prior CaO addition in slurry would probably replace or reduce the 800 g/t milk of lime dosage currently used for bauxite's digestion depending on the quantity of CaO that remains in the solid phase after bauxite slurry filtration. This issue will be approached in parallel studies along with the review of possible impacts that industrial water containing milk of lime may offer to its users, such as cooling tower make up and flocculant preparation.

Bauxite filtration is a relatively rare applied process all around the world. Thus, the approach to this topic is scarce in the literature. MPSA's peculiar logistics to transport the ore to Alunorte via pipeline requires it to be in the form of slurry, meeting a certain range of particle size and density. For this reason, Alunorte develops many studies involving dewatering performance. Alex Pinheiro et al. [2] conducted an industrial-scale test with Alunorte's hyperbaric filters using Design of Experiments (DOE). Results indicated that rotation speed, basin level, and pressure significantly influence the productivity (solids throughput), cake moisture, and solids in the filtrate. This work indicated no significant correlation between the bauxite's particle size and the filtration performance.

Focusing on concept analysis, Selomulya et al. [3] developed hyperbaric filtration studies to investigate the implications of micro-properties (size, structure, and strength) of coal aggregates for coal dewatering. The authors indicate that low cake permeability due to smaller capillaries, related to fine particles, can be overcome by higher filtration pressures. However, high pressure may also have the opposite effect, altering the interconnected pores by collapsing the cake structure, resulting in low filtration rates.

Thus, this work aimed to study the influence of the three control variables (pH, density, and top size) on the filtration process productivity (response variable). It will support the decision-making of the most economically viable slurry modification alternative that meets both the pipeline and hyperbaric filters needs. The study was conducted on a laboratory scale using Box Behnken Design (BBD) statistical technique.

2. Methodology

2.1 Box Behnken Design

A Box Behnken Design (BBD) was developed with three factors and a central point to understand the influence of top size, density, and presence of milk of lime (pH) in bauxite slurry on hyperbaric filter productivity. Currently, up to 12 % in mass of particles exceed 300 μm , and typical values for d90, d50, and d10 are 197, 75, and 4 μm , respectively. Higher pH levels above the current value of 7.0 were tested by adding milk of lime with 176 g/L solids content and d90, d50, and d10 equal to 75, 15, and 5 μm , respectively. The levels for each factor are specified in Table 1.

Table 1. Design factors and levels of BBD.

Design Factor	Levels			Response Variable
	-1	Center point	+1	
Top size	100 % < 355 μm	100 % < 425 μm	100 % < 500 μm	Productivity
Density	1.44 g/cm^3	1.46 g/cm^3	1.49 g/cm^3	
pH	7.0	9.25	11.5	

The BBD technique was introduced by Box and Behnken in 1960, fitting into a Response Surface Methodology (RSM) and being one of the best known and applied in the experimental process optimization to establish the best analysis conditions. This technique is composed of rotating plans where each variable has three levels, and the experimental points are distributed equidistantly from the central point [4]. Figure 1 presents the experimental points geometric view for this design for three factors with two levels and a central point.

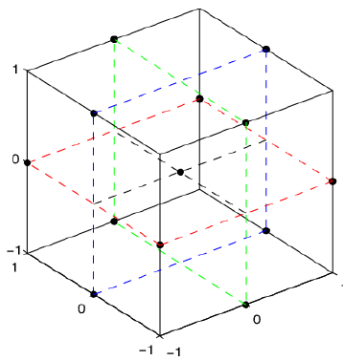


Figure 1. Box Behnken design of experiments geometric view.

The experiments were carried out using benchtop equipment capable of reproducing the hyperbaric filter operation on a laboratory scale. Table 2 presents the non-randomized experiment matrix. The runs were performed in triplicate, totalling 39 filtration rounds.

Table 2. Non-randomized experiment matrix.

Experiment	Top size (μm)	Density (g/cm^3)	pH
1	355	1.46	7.00
2	500	1.46	7.00
3	355	1.46	11.50
4	500	1.46	11.50
5	355	1.44	9.25
6	500	1.44	9.25
7	355	1.49	9.25
8	500	1.49	9.25
9	425	1.44	7.00
10	425	1.44	11.50
11	425	1.49	7.00
12	425	1.49	11.50
13	425	1.46	9.25

It is important to emphasize that the benchtop equipment used to carry out the filtrations is useful to evaluate the behaviour of the control variables only during the cake formation process. Thus, this study on filter productivity does not consider other impacts arising from the change of these variables on the operation of the industrial filter, such as problems with cake unloading and cloth blinding.

2.2 Experimental Procedure

A sufficient volume of bauxite slurry was collected using an automatic sampler in the ore pipeline and divided into three equal parts for adjusting the desired top size levels. For this task, each of the three samples was wet sieved with the corresponding specified mesh (500, 425, and 350 μm). Then, each adjusted slurry to its top size was quartered into three equal parts to adjust the density levels: 1.44, 1.46, and 1.49 g/cm^3 . Density adjustment was performed by removing the supernatant water after three hours of standing from the bauxite slurry and confirming the final density of the slurry by analysing the solids content on a halogen moisture analyser.

The experiments in Table 2 were randomly executed in triplicate with the following constant and equal parameters for all rounds: pressure, filter cloth, bauxite slurry feed mass in the equipment, and dewatering and cake formation rate (t_2/t_1) equal to 1. The 5.8 barg pressure that was applied in the experiments and the t_2/t_1 ratio were the same as those used in the industrial filters' operation. Similarly, the same filter cloth used in the field was also used in the laboratory, and for each round of filtration, a new piece of cloth was used.

The first step of the laboratory procedure consisted of adding milk of lime to the bauxite slurry under agitation and pH reading until the desired pH was reached. As the bauxite slurry originally had a pH of 7.0, experiments with this pH specification did not need this step, only those with a pH of 9.25 and 11.5. Then, 40 grams of bauxite slurry with or without milk of lime was weighed in a beaker with analytical balance and fed into the filtration equipment. The feed mass of 40 grams, chosen after preliminary tests, was determined so that the cake formed after filtration presented thickness and moisture values close to those verified in the operational area. After feeding the bauxite slurry into the equipment, the beaker was weighed again to obtain the effective mass. Then, the feeding compartment was closed, the pressure supplied, and the slurry began to be filtered, forming the cake. Cake formation was visually monitored by the same analyst in all experiments through a translucent window. The time from the beginning of filtration until the end of cake formation (t_1) was timed, which was characterized by the cake surface becoming completely opaque. Immediately afterward, the dewatering time (t_2) began, which should be equal to the time computed in t_1 , that is, the complete filtration lasted twice t_1 .

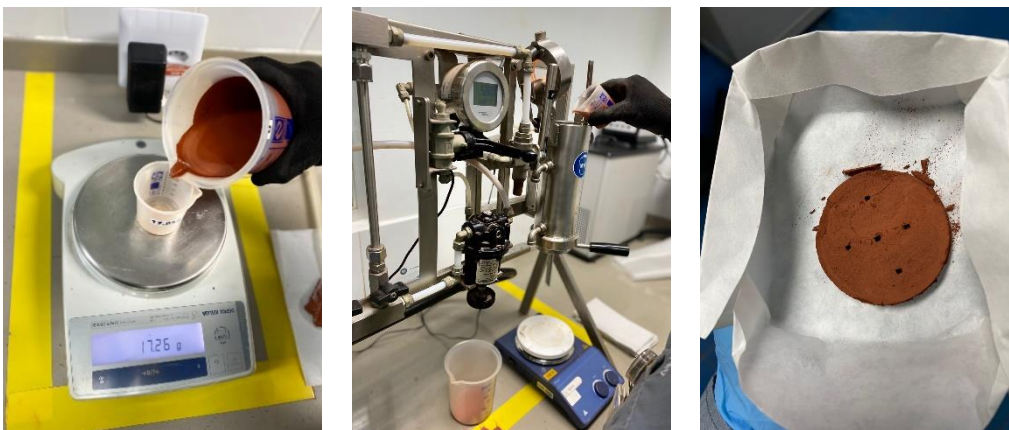


Figure 2. Experimental procedure for the slurry bauxite hyperbaric filtration.

Soon after the end of t_2 , filtration was discontinued by closing the pressure valves. Cake thickness was measured with a caliper at five different points and always in the same positions for all experiments, and then the cake was weighed on an analytical balance, put into an oven at 100 $^{\circ}\text{C}$ for 12 hours, and weighed again for solids content analysis.

2.3 Hyperbaric Filtration Productivity Calculation

With the cake formation time obtained through filtration experiments and the knowledge of the cake formation angle of the industrial filter equal to 122 ° (disc design parameter), it is possible to calculate the rotation speed of the industrial filter and, subsequently, its specific and hourly productivity [5].

$$n = \alpha_1 / (t_1 \times 360^\circ) \quad (1)$$

where:

n Rotation filter speed, h⁻¹
 α_1 Cake formation angle, °
 t_1 Cake formation time, h.

$$P_s = (m_s \times n) / A_b \quad (2)$$

where:

P_s Specific productivity, kg/m².h
 m_s Mass of solids, kg
 n Rotation filter speed, h⁻¹
 A_b Benchtop equipment filtration area, m².

$$P_h = (P_s \times A_f) / 1\,000 \quad (3)$$

where:

P_h Hourly productivity, t/h
 P_s Specific productivity, kg/m².h
 A_f Industrial filter filtration area, m².

While the specific productivity considers the filtration area of the laboratory equipment ($A_b = 0.001963 \text{ m}^2$), the hourly productivity is calculated with the industrial filter's filtration area ($A_f = 168 \text{ m}^2$).

3. Results and Discussion

3.1 Productivity Model

After the analysis of variance in the hourly productivity results obtained, the adjusted model described by Equation (4) was generated with R²(adj) of 80.56 % to describe the relationship between the control factors top size, density, and pH and the productivity response variable. The significance level α chosen was 0.05.

$$P = 18668 - 0.1322TS - 26\,532D + 128.5pH + 9\,527D^2 + 1.214pH^2 + 0.02099TS \times pH - 106.9D \times pH \quad (4)$$

where:

P Productivity predicted by model, t/h
 TS Top size, μm
 D Density, g/cm³
 pH pH

To assess how the variation of factors impacts filter productivity, Table 3 presents scenarios of interest to the MPSA and Alunorte's technical team with the respective productivity values predicted by the model (Equation 4). It is worth mentioning that the model is applicable only within the levels on which it was built.

Table 3. Evaluated scenarios with predicted productivities using the model.

Case	Top size (μm)	Density (g/cm^3)	pH	Productivity (t/h)
1	500	1.44	7.0	105.9
2	500	1.49	7.0	137.6
3	425	1.44	7.0	104.8
4	425	1.49	7.0	136.5
5	500	1.44	10.5	128.0
6	500	1.49	10.5	141.0
7	425	1.44	10.5	121.6
8	425	1.49	10.5	134.6

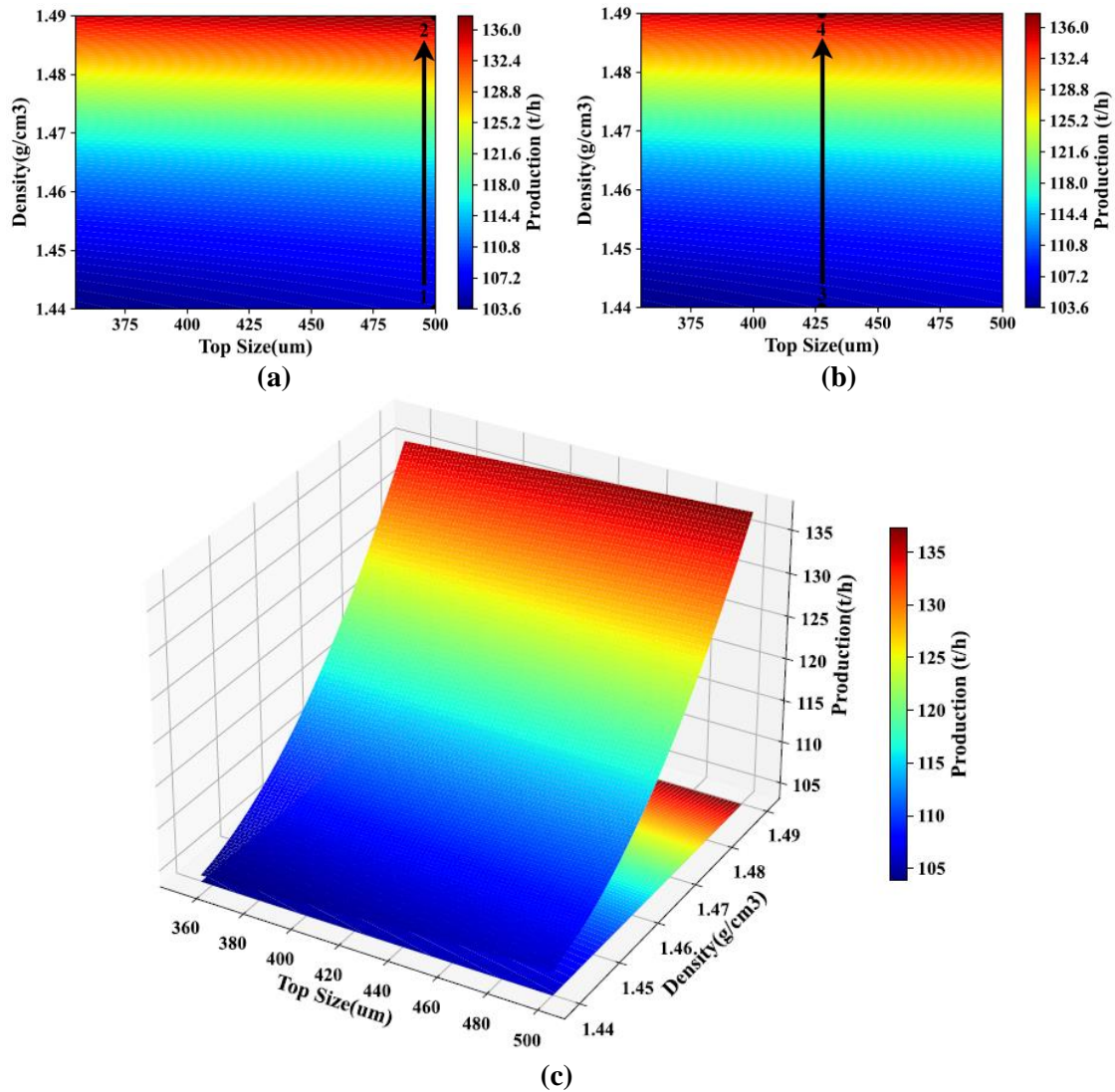


Figure 3. Contour plots and response surface for top size and density with pH 7.0. a – Contour plot with cases 1 and 2, b – Contour plot with cases 3 and 4, c – Response surface.

Figures 3 (a), (b), and (c), respectively, present the contour plots for scenarios 1-2, 3-4, and the response surface with different values of top size and density, keeping pH equal to 7.0. From the analysis of Figures 3 (a) and (b) with Table 3, it is observed that, for pH values of 7.0 and constant densities, there are no significant changes in productivity with the change in top size (from scenario 1 to 3 and from scenario 2 to 4).

From the analysis of the response surface, it is possible to notice more clearly the significant effect of the density change on the filter's production capacity when top size is constant (scenario 1 to 2 and 3 to 4 in Table 3).

A similar analysis was performed in scenarios 5-8 of Table 3, changing the pH from 7.0 to 10.5 to explore the model's capability and understand how density and top size behave at this new pH level. The analyses of Figures 4 (a) and (b) with Table 3 indicate that pH values equal to 10.5 increase the curvature of the response (productivity) and allow significant transitions in production capacity.

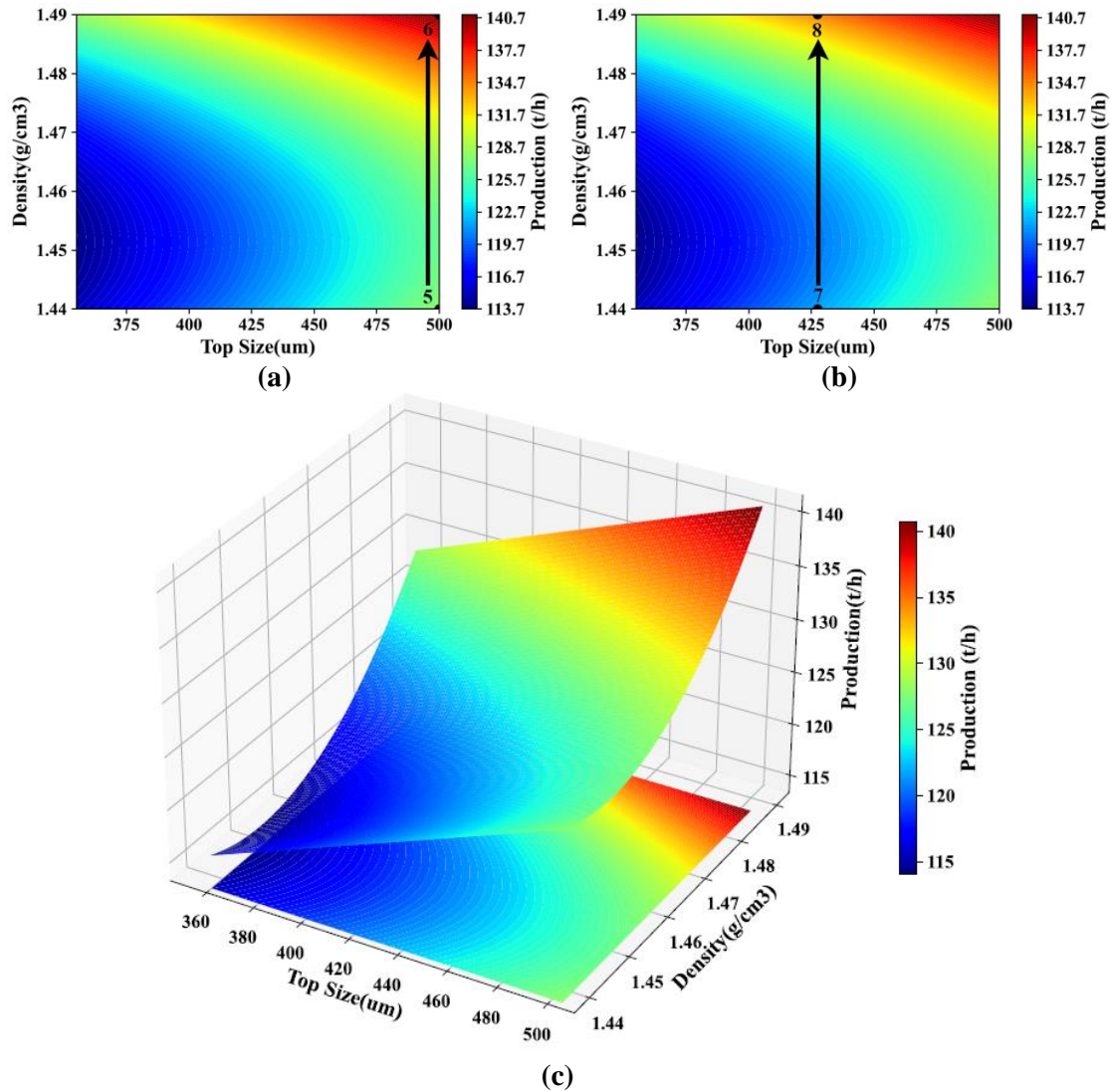


Figure 4. Contour plots and response surface for top size and density with pH 10.5. a – Contour plot with cases 5 and 6, b – Contour plot with cases 7 and 8, c – Response surface.

At pH equal to 10.5 (Figure 4), it is possible to see that, if the density is constant, there is a significant productivity reduction with the top size reduction (from scenario 5 to 7 and from 6 to 8). It is different from what occurs from scenario 1 to 3 and from 2 to 4 with pH 7.0, in which there is no relevant change in the response variable by changing the top size.

Density is the factor that causes greater sensitivity to filter's productivity. From a statistical point of view, it is due to the quadratic term in the model. In Figure 4 (a), for instance, the increase in

density with top size fixed at 500 μm gives an increase in productivity from 128.0 t/h to 141.0 t/h when the density increases from 1.44 g/cm³ to 1.49 g/cm³ (scenarios 5 and 6, respectively).

As mentioned before, the top size reduction at high pH values (10.5) causes a decrease in filter production capacity. In this sense, the initial productivity value for scenario 7 with a top size of 425 μm and pH 10.5 is lower (121.6 t/h) compared to scenario 5 of 500 μm and pH 10.5 (128.0 t/h). The density increase from 1.44 g/cm³ to 1.49 g/cm³ contributes positively, raising productivity from 121.6 t/h (scenario 7) to 134.6 t/h (scenario 8).

For all scenarios evaluated, initial productivity values related to density 1.44 g/cm³ are much higher in cases at pH of 10.5 than at pH 7.0, indicating an increase in this curvature in the response variable with increasing pH.

3.2 Model Adequacy Checking

The validity of the analysis of variance that generated the model of Equation 4 requires that certain assumptions are satisfied: residues are normally and independently distributed with mean zero and constant but unknown variance [6]. Violations of these assumptions and model adequacy can be studied by graphical analysis of residuals, as shown in Figure 5.

Figure 5 (a) presents the normal probability plot of residuals. The p-value of 0.891 obtained denotes the normality of the residuals since it is above the significance value $\alpha = 0.05$. Figures 5 (b) and (d) show the plots of residuals versus fitted values and observation order, respectively. As observed, there is no anomalous behaviour or tendency in the distribution of residuals, indicating a uniform and random disposition of variances, a criterion linked to homoscedasticity. Furthermore, the histogram of Figure 5 (c) shows the residuals with mean zero.

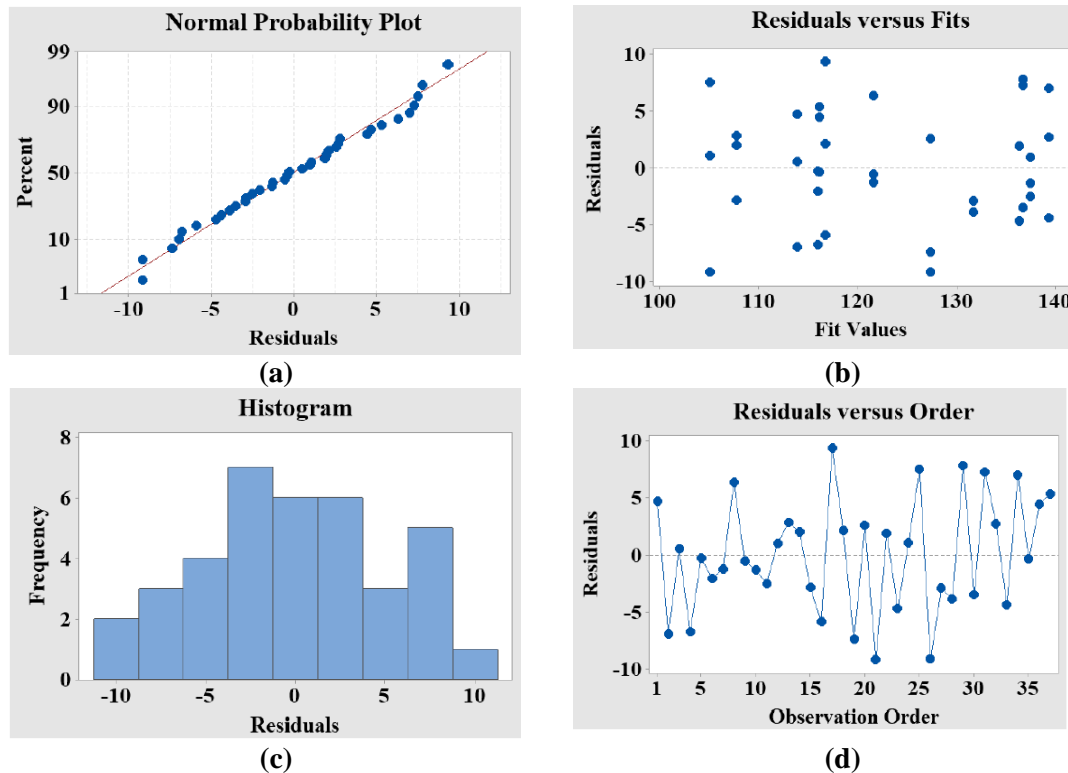


Figure 5. Graphical analysis of residuals. a – Normal probability plot of residuals, b – Plot of residuals versus fitted values, c – Histogram of residuals, d – Plot of residuals versus observation order.

4. Conclusions

This paper aimed to evaluate the impacts of the top size and density of the bauxite slurry and the addition of milk of lime on the filtration process productivity of Alunorte's hyperbaric filters that dewater the bauxite slurry from MPSA.

Based on a Box Behnken Design with filtration equipment on a laboratory scale, it was possible to satisfactorily obtain the necessary data to evaluate the influence of the input variables (top size, density, and pH) on the output variable (hyperbaric filtration productivity).

It was possible to obtain an adjusted model with $R^2(\text{adj})$ of 80.56 %. For all scenarios evaluated, the increase in density positively impacted productivity. This fact is related to the positive quadratic effect of density in the equation obtained for the model. This factor proved to be the most significant for changing the filter's production capacity.

It was observed that the profile for productivity at pH values close to 7.0 does not show significant influence from the change in top size. On the other hand, it is possible to achieve high productivity by changing the density to values above 1.46 g/cm³.

In addition, pH and top size increase positively impacted productivity. Higher pH levels (around 10.5), make productivity more sensitive to the top size control variable when compared to lower pH values (around 7.0).

This study will be useful to define future experiments on an industrial scale to compose the complete analysis of the best parameters of the bauxite slurry and optimized operational parameters that meet both MPSA pipeline and Alunorte's hyperbaric filters productivity needs. Furthermore, future experiments will be performed to optimize bauxite cake moisture and study the impacts on keeping using the dewatered filtrate with milk of lime as industrial water supply to Bayer process.

5. References

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