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IMPROVEMENT OF ACTIVE INGREDIENTS IN GRIND STAGE ON OPERATING COSTS REDUCTION OF A INDUSTRIAL CHEMICAL FACILITY

Authors:

Nelson Lisboa da Cruz

UNISAL – Campus de Lorena/SP

Henrique César Sampaio

UNISAL – Campus de Lorena/SP

ABSTRACT

The milling process requires resources that are mostly quite expensive as high energy consumption, equipment, grinding media, spending from devices exchanges, among others. And so a deep analysis of the circuit fragmentation operation type is necessary to make possible to choose the type of equipment and operation more appropriate to the purpose for which it is intended. Therefore, this work aims to assist in directing for choosing the most appropriate type of operation in the fine grinding process employed in the industrial sector. For the chemical industry sector, its main feature is the production of high value compounds, making the operating cost factor even more important. The energy consumed in comminution processes and mechanical deterioration to which the equipment is subjected, is directly related to operating costs and investment in comminution circuits. A better conformation of the comminution circuit is not only capital savings, as well as energy resources, to increase profitability, reduced operating time and better use of materials and energy resources. Through optimization methods and process control, seeks to achieve operating and optimal process control, so mathematical models can be built from Design of Experiments (DOE) methodology.

Keywords: Costs Reduction. Milling. Chemical Facility.

1 THE COMMINUTION PROCESS IN AN AGRICULTURAL PRODUCTION DEFENSIVE

1.1 Definition of the experiment goals

This work evaluated which parameters were being controlled and what levels were adjusted to a grinding process of a crop protection company. The experiment goal consists on the milling process and how to increase the grinding flow for this base to grind, because it consists on the "bottleneck stage" of the manufacturing process of this agricultural defensive. For milling in sphere mills will be necessary a review of its operation and its parameters related to this circuit and change different concentrations of ground base.

It was verified that the specific plant milling process was short of the expected results (considering the current demand for the product), it is necessary an improvement in the order of 30%.

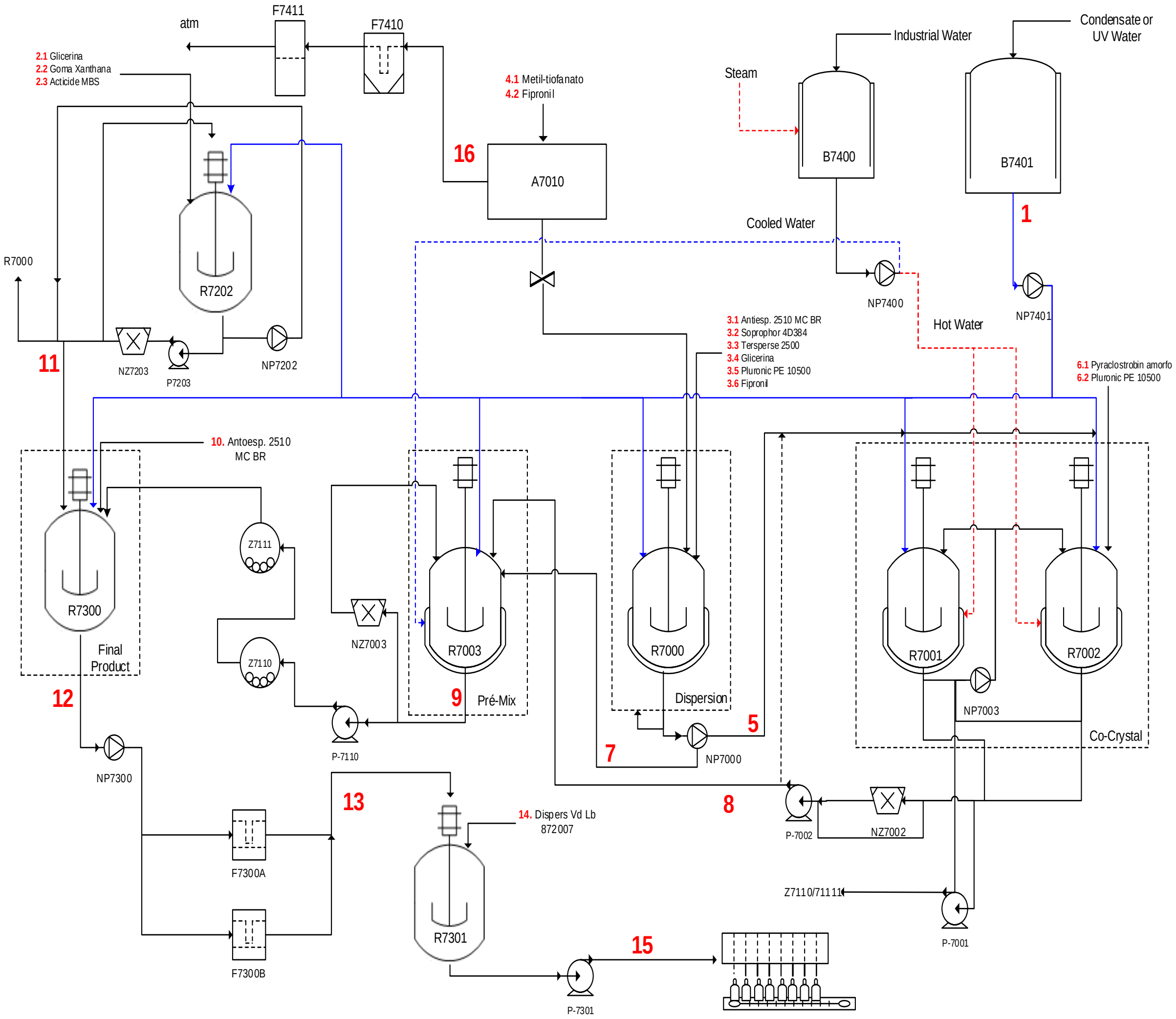
As a second potential point, it was also evidenced the need to improve the co-crystallization (stage prior to the milling step) to support possible increases in future demand.

1.2 The Production Process

The pesticide production plant is installed in a chemical complex in Guaratinguetá/Brazil since 2008 and it is dedicated exclusively to the formulation of such products, there is no process of chemical synthesis. It produces 78 different products. The method DOE was selected due to repeated non-conformities in particle size function. The product is a concentrated suspension type insecticide for seed treatment, the chemical group pyrazol classified as toxic medium containing 250 g / l of Fipronil and 850 g / l of inert ingredients. It is initially issued an operating instruction, called ‘March Sheet’ for product formulation. This document contains the exact amounts of each raw material, specific to the formulation of that lot, plus the addition of order. The final product is thickened from a mixture of gel and dispersion. The dispersion is formulated separately from the gel, but simultaneously in two different production lines. The function of the gel is to enhance the suspensibility of active ingredient dispersion in the final product. After the issue of 'March Sheet' starts the simultaneous formulation of the gel and dispersion. After added all of the raw materials, the weight of the dispersion should undergo a pre-grinding process in a colloid mill, in order to make all particles to obtain a uniform size, removing its lumps and stabilizing the mixture.

The parameters must be within the specified analysis, afterwards the product may be moved to the buffer tank. Its displacement occurs via pump, through a filter to contain possible lumps. The production process flowchart is shown in Figure 1.

Figure 1 – Production flowchart a crop protection process



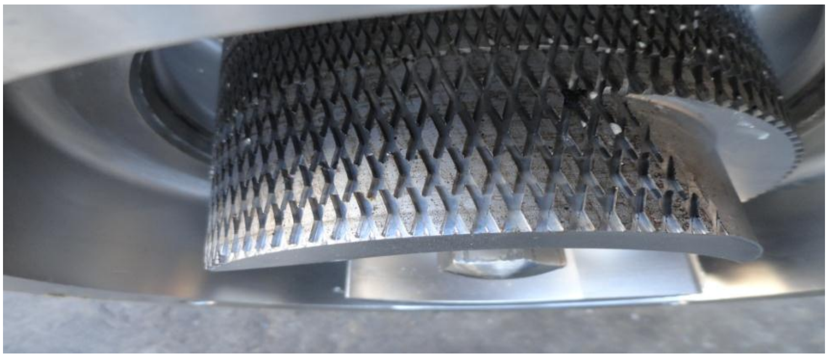
1.3 The Milling Process

The product undergoes a process of pre-grinding in the colloidal mill, Figure 2, in order to make uniform particle size and eliminating lumping, obtaining an uniform mass. The colloid mill consists in a main shear mechanism according causing breakage of larger particles. This process have a power generation, thus reducing the particle and disrupts their clusters. As the particles of the disperse phase of the emulsion are smaller, less energy will be required to overcome the surface tension while maintaining the stability on this step.

Figure 2: Colloid mill

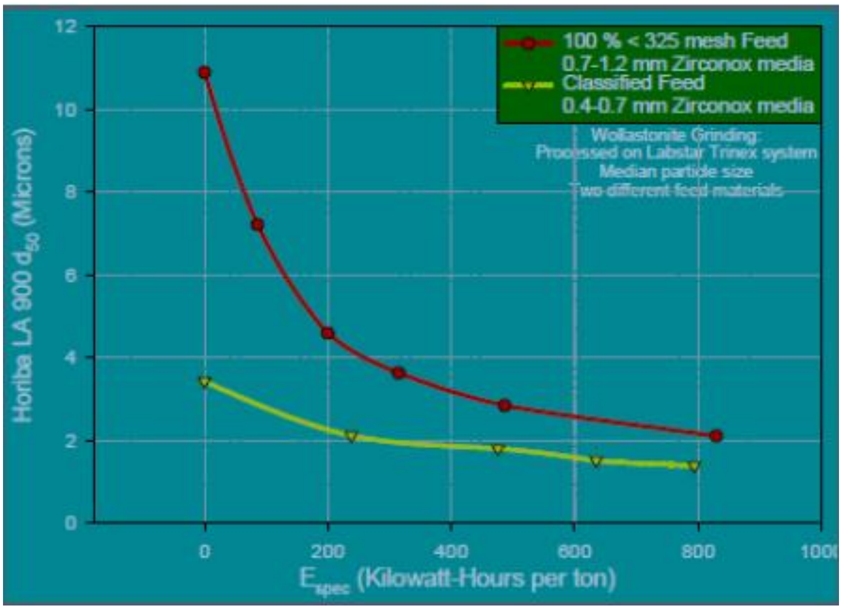
The product to be emulsified is fed by gravity and comes into contact with a high-speed rotation rotor. This rotor is adjusted to a stator, so that the space between them is minimal, according to Figure 3. The distance between them is called ‘grinding gap’.

Figure 3: The grinding rotor-stator system



Once the material comes into contact with the rotor is thrown to the edges by the action of centripetal force. This force pushes the material through the narrow passage between the rotor and the stator, which suffers breaking shear. The mill has a system for cooling the product, which increases temperature during operation. The cooling is obtained through a chiller type equipment with a mixture of water and monoethylene glycol at 5°C. The temperature is controlled, not surpass 38°C for no degradation of the active ingredient and increase energy consumption, as showin in Figure 4.

Figure 4 - Correlation between particle size and energy consumption



All milling process was developed with two LME type mills operating in series. Conventional grinding is operated in open circuit, that is the product passes through the mill once, which is possible only because of the pre- grinding process. Although the circuit has a rating of equipment. After densification, the material retained on the filter does not return to the process for further milling. According to Metso (2006), this type of operation is more suitable when there is another step to further comminution and the feed material is already fine, otherwise may have disadvantages such as the absence of control system. The mills are the LME 200, as shown in Figure 5.

Figure 5: Mill LMZ type balls with agitator shaft system with discs.

The mills comprise grinding a camera with 580 mm diameter and 1600 mm of length and volume of 250 liters. As calculation defined by engineering team, all mills in series should be supplied with diameter zirconium oxide spheres ranging from 0.8 to 1.0 mm. The first mill receives a load of 85 % of its volume and the second windmill 95% to be performed a pre reduction in particle size and it does not happen in the second high pressure mill. The product residence time in the mill varies with the pump power. If the particle size distribution is out of specification, the rotation of the pump is reduced and thus there is an increased product residence time in the grinding chamber. Currently, the milling process is the critical point of the operation due to the high time required for the specification attainment. The operation usually lasts 3 to 4 hours.

1.4 Process variables analysis

Through supplier catalog of the grinding lines of equipment (mills and pumps), it was conducted a brainstorm with experts (supervisors and engineers) with extensive experience to select the variables and noise. The factors and leves consideraded are shown in Table 1.

- Variables:
    - Inlet temperature in the mill.
    - Ball mill load in chamber (considered a difference of 10% lower in the second mill).
    - Size (diameter) of the average beads (both the first and to the second mill).
    - Concentration of millbase.

  - RPM (revolutions per minute) of the engine mills.
  - Engine RPM screw pump feeding the mills.
  - RPM Base vessel agitator grinding during grinding.
  - Time Pre-Grinding Step colloid mill.
  - GAP adjustment or flow passage restriction in step pre-grinding process colloid mill.
  - Colloidal mill engine RPM, in step pre-grinding.

Noises are the variables that can be controlled in a laboratory, but they are difficult to control during manufacturing (Miro-Quesada; Del Castillo, 2004).

- Noises:
  - Mill outlet temperature.
  - Inlet pressure in the mills.
  - Average size of solids granules in the grinding base (variation of the active ingredient synthesis process, for example).
  - Deterioration of the equipment used in the process.
  - Deterioration of the balls due to the use.
  - Variation in particle size analysis.

Afterwards there was a selection, where the study was restricted to the following variables (also set the levels):

- Variables:
  - Inlet temperature in the mill.
  - Ball mill load in camera (always considered a difference of 10% lower in the second mill).
  - Size (diameter) of the average beads (both the first and to the second mill).
  - Concentration of millbase.

Table 7 – Factors and levels

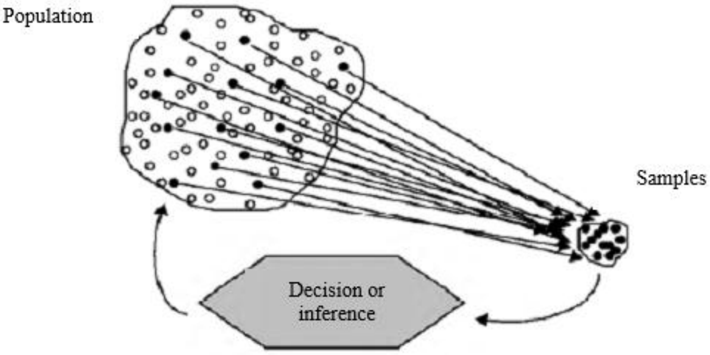
FACTOR	**FATOR LEVEL**		**PROCESS INFLUENCE**
	-1	**1**	
A - Load ball grinding body	85% v/v	95% v/v	Quantity recommended by the manufacturer ball mill (considering the first mill)
B - size balls	0,6 - 0,8 mm	1,0 -1,2 mm	Improves efficiency process

C - Concentration millbase	25% m/m	30% m/m	Improve efficiency process - avoids grinding liquid
D - Temperature input mill	10ºC	12ºC	Increase less final temperature process.

1.5 The response variable selection

According to the requirements of the grinding sheet (profitability), the response variable should be the flow rate of the base multiplied by the concentration (indicates the flow total solids). The restriction is the particle size (D50 % < 3.0 µ and D90 % < 4.0 µ) already established for the ground base, so that the final product (ready for sale) reaches the medium size specifications particle and also wet sieve. Thus, it was monitored the output of the second mill, measured by laser diffraction wet, sampling variations can occur, since it is not being analyzed the entire population, according to Figure 6.

Figure 6 – Illustration of a sampling procedure



Source: Manson, Gunst, Hess (2003)

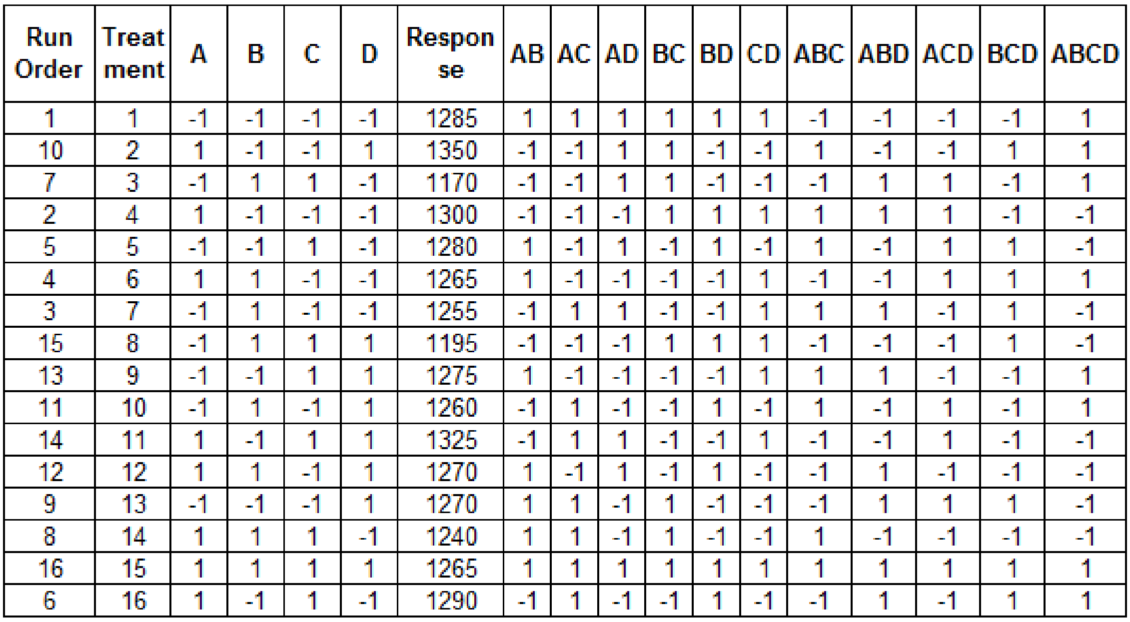
1.6 Selection of the experimental matrix

It is necessary tools to analyze the data collected during the project and the results of statistical tests should be interpreted. In interpreting these data, the researcher moves from the empirical domain for theoretical. This involves inference and generalization of considerations (MEREDITH, 1998). Tools are necessary to obtain a clear focus on utility and restricted application.

Examples of tools: cause and effect diagrams, control charts, histograms, among others. Techniques can be defined as a collection of tools; as such techniques are: Design of Experiments (DOE), Statistical Process Control (SPC), Benchmarking, Effect Analysis and Failure Mode (FMEA), and others (MCQUATER et al., 1995). Tools and techniques require special attention to a number of critical factors aiming to use and efficient and effective implementation.

For performing the tests in an array with only 4 input variables (factors) and a variable response, it decided on a complete matrix or full factorial. However, to get an idea of experimental error, it was chosen a combination in the experimental matrix and repeated 2 times. The experimental matrix was generated via Minitab 17.1.0, so the software "randomized" the order of experiments execution.

Table 2: Experimental matrix

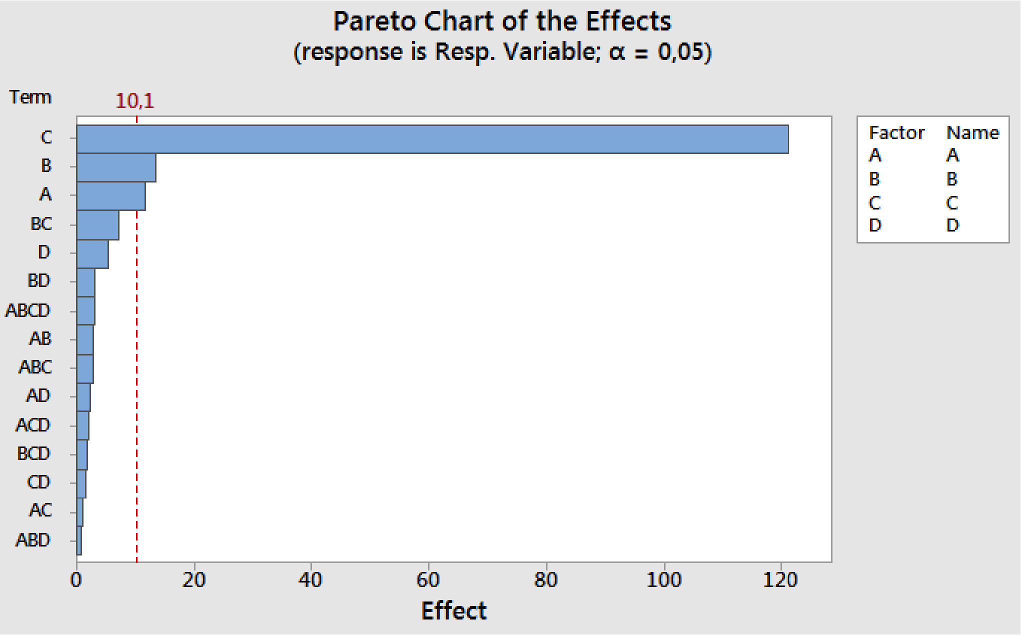


Run Order	Treatment	A	B	C	D	Response	AB	AC	AD	BC	BD	CD	ABC	ABD	ACD	BCD	ABCD
1	1	-1	-1	-1	-1	1285	1	1	1	1	1	1	-1	-1	-1	-1	1
10	2	1	-1	-1	1	1350	-1	-1	1	1	-1	-1	1	-1	-1	1	1
7	3	-1	1	1	-1	1170	-1	-1	1	1	-1	-1	-1	1	1	-1	1
2	4	1	-1	-1	-1	1300	-1	-1	-1	1	1	1	1	1	1	-1	-1
5	5	-1	-1	1	-1	1280	1	-1	1	-1	1	-1	1	-1	1	1	-1
4	6	1	1	-1	-1	1265	1	-1	-1	-1	-1	1	-1	-1	1	1	1
3	7	-1	1	-1	-1	1255	-1	1	1	-1	-1	1	1	1	-1	1	-1
15	8	-1	1	1	1	1195	-1	-1	-1	1	1	1	-1	-1	-1	1	-1
13	9	-1	-1	1	1	1275	1	-1	-1	-1	-1	1	1	1	-1	-1	1
11	10	-1	1	-1	1	1260	-1	1	-1	-1	1	-1	1	-1	1	-1	1
14	11	1	-1	1	1	1325	-1	1	1	-1	-1	1	-1	-1	1	-1	-1
12	12	1	1	-1	1	1270	1	-1	1	-1	1	-1	-1	1	-1	-1	-1
9	13	-1	-1	-1	1	1270	1	1	-1	1	-1	-1	-1	1	1	1	-1
8	14	1	1	1	-1	1240	1	1	-1	1	-1	-1	1	-1	-1	-1	-1
16	15	1	1	1	1	1265	1	1	1	1	1	1	1	1	1	1	1
6	16	1	-1	1	-1	1290	-1	1	-1	-1	1	-1	-1	1	-1	1	1

2. RESULTS ANALYSIS

The results present in the graphs shown in the Figures 7 and 8 is to determine which factors or interactions and obtained greater effect than the Fcrítico (cutoff for significance).

Figure 7: Factor interactions and effects - Pareto Chart



Source: Minitab® 17.1.0

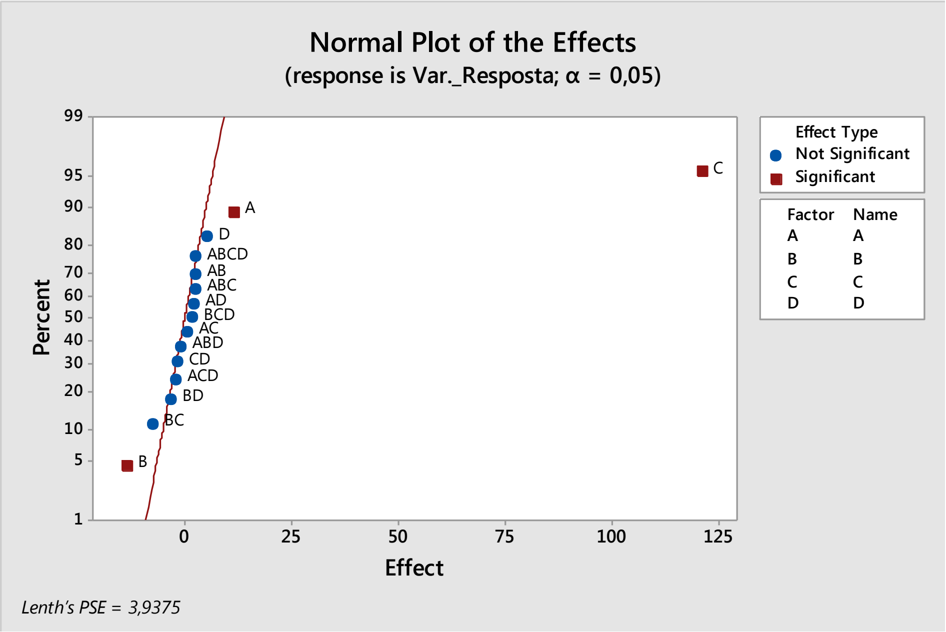
In this case, a value of 10.1. If this happens, it means that, statistically, no difference between the means is the effects or interactions, or failed in accepting the following hypotheses H0:

Factors: **H01:** μA = μB = μC = μD
H11: at least one medium differs

Interactions: **H02:** μAB = μAC = μAC = μBC = ... = μABCD
H12: at least one medium differs

The graph interpretation of the Figure 7 is to determine which factors and interactions or considerably distanced from the normal straight line (straight adjusted, where it is expected that point if there were no significant effect), thus indicating that they are significant. Basically it moves the way to show the factor or interaction is significant or not.

Figure 8: Factor and interaction effect - normal curve



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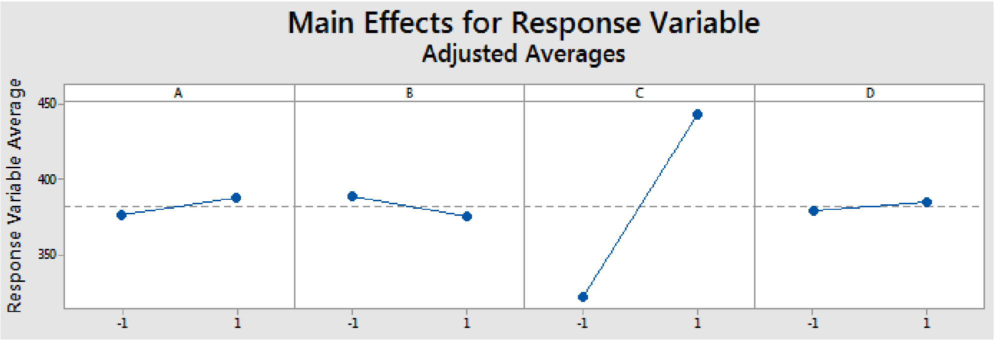
The interpretation of the graph in Figure 8 is to check for factors which distance is greater when this factor is adjusted in level "-1" and "1" . The greater distance, the higher factor of significance level for the study, it means that a higher concentration of basic grinding causes a great effect on the dependent variable.

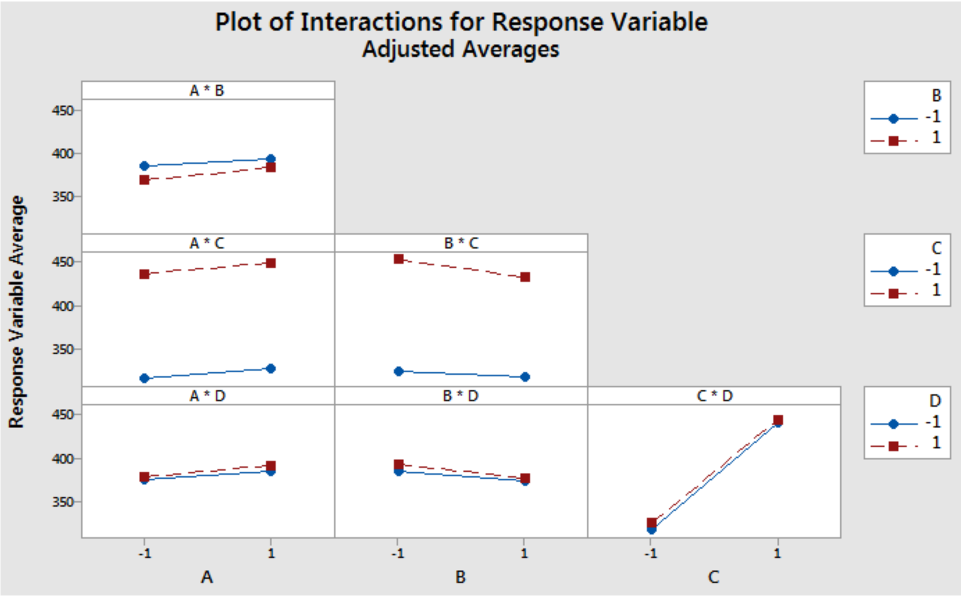
Figure 9: Factor effect on variable response



Source: Minitab® 17.1.0

The interpretation of the graph in Figure 10 , consists of verifying in pairs, if there are lines crossing the effects of second order interactions when adjusted levels "-1" and "1". This indicates that there are significant interactions for the study.

Figure 10 – Effect of Interactions



Source: Minitab® 17.1.0

If the lines are "parallel", as is the case, shows that there are no significant interactions. The regression equation for the variable response is given by the following expression:

Var._Resposta = 382,3 + 5,828 A - 6,703 B + 60,70 C + 2,703 D + 1,422 A*B + 0,5156 A*C + 1,203 A*D - 3,578 B*C - 1,578 B*D - 0,7344 C*D + 1,422 A*B*C - 0,3906 A*B*D - 0,9844 A*C*D + 0,9219 B*C*D + 1,484 A*B*C*D

The relevants terms are: A, B and C. Adjusting all terms, as predicted response by one Model Milling flow of 444.53 kg/h of total solids. The experiment # 16 results 455 kg/h. The objective is the optimization processes and it was conducted a replicate of this experiment. The replicate results 448.7 kg/h and 452.4 kg/h. Thus, the estimated Experimental Error (standard deviation) was estimated at 3.16 kg/h, value much lower than the $F_{critical}$, which confirms the decision of the significant factors.

3. CONCLUSION

This article presents parameters which are essential for the comminution circuit analysis, and the best way to analyze properly the comminution circuit to be implemented, as the operation mode is associated to energy consumption and a good sizing processes based on the particle size distribution became also visible, the effectiveness of the increased specific weight of grinding bodies in the process. The correct configuration of a comminution circuit shows not only capital economy, but also energy resources.

So the energy absorbed in the comminution process is directly related to operating costs and investment in comminution circuits, as well as mechanical deterioration which the equipment is subjected. To achieve a high level of profitability with low operating time and better utilization of physical resources and energy it is necessary a careful definition and based on research, since all types of equipment and operations provide advantages and disadvantages. From now on it is up to choose the best fit to the type of product and facilities, taking into account that each process contains peculiarities and research found in the area, for the most part are still empirical.

Compared to the initial level, which was 330.5 kg/h of total solids, an increase process efficiency of 34.5%, reflecting increased capacity for the Company, may thus absorb the increase in demand which was requested (original problem). It was verified that, other than the operation had a paradigm, simply increasing the concentration of the product, resulting in a significant gain in grinding flow in terms of total solids, unlike the professionals involved (engineers and supervisors) believed.

Another interesting point observed was the effect of Factor B (average size of the beads used), which had an effect on the response variable 10 times smaller than the factor C also quite different from what was expected for this type of process.

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