CONTROL OF THE CONTINUOUS MILLING OPERATION

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ABSTRACT

In the first phase of this study, conducted in the laboratory, the effect was established of solids and deflocculant content, as well as milling temperature on viscosity, oversize and particle size distribution of the milled suspension.

In the second phase, industrial tests were carried out in which mill input variables (initial solids content, deflocculant content and clay mass flow rate) were altered, and output variables (density, viscosity, oversize and suspension temperature) were analysed.

On the basis of the industrial results, input and output variables were correlated, which enabled determining how density, mill oversize and viscosity vary with time when changes occur in input variables. The study allows analysing the feasibility of automatic milling operation control.

1. INTRODUCTION

Continuous milling is becoming an ever more widespread technique in the ceramic tile industry. It has allowed eliminating downtime associated with mill charging and discharging and, in principle, enabled increasing resulting suspension solids content ^[1]; however, it is not free of problems: formation of crusts on the mill inner wall, formation of large conglomerates of clay and milling balls (a defect commonly known as "bolus"), accumulation inside the mill of hard-to-mill components, etc. Moreover, continuous milling demands greater process control, since if some deviation takes place (in suspension density, viscosity or milling oversize), it is necessary to make the correction without stopping the mill, and the flexibility in milling time (residence time) is much smaller than in a discontinuous mill.

All these greater control requirements are in sharp contrast with the fact that operation is still controlled (or supervised) manually. Corrective mill adjustments are based on manual measurement of density, oversize and viscosity determined by an operator. It is clear, therefore that it would be desirable to be able to fully automate the process, which is in fact the tendency in other sectors ^[2].

2. OBJETIVE

The objective of the study consisted basically of:

- Establishing the relation between the variables involved in milling, on a laboratory and industrial scale.
- Determining the rate at which density, viscosity and oversize change when feed changes (system dynamics).
- -The ultimate aim of the study consists of laying the groundwork for automation of the milling operation, achieving greater constancy in operating variables (density or solids content, viscosity and oversize), and even for preventing possible anomalies in the milling operation from arising.

3. MATERIALS AND EXPERIMENTAL PROCEDURE

To conduct both the industrial and laboratory tests, a red stoneware composition was used, made up of a mixture of Villar and Moró clays. The suspension was prepared using wastewater, to try and approach the industrial milling conditions. Suspensions made up in the laboratory (by planetary mills) and industrial suspensions (prepared with continuous mills) were used.

Suspension characterisation was done using the following equipment and procedures:

- Solids content: determined by oven drying and subsequent weighing of the dry oversize.
- Viscosity: capillary viscometer (Ford cup), torsion wire viscometer (Gallenkamp) and a rotational viscometer that allows characterising the viscosity at different shear rates.
- Particle size distribution: measurement using a laser dispersion instrument.
- Oversize: determined by the wet method with a 63 μ m mesh screen.

4. RESULTS AND DISCUSSION

4.1 TESTS CONDUCTED ON A LABORATORY SCALE

Three groups of tests were conducted. The first one consisted of studying the effect of the variation of solids mass content (ω_s) [kg dry solid/kg suspension] on the final properties of the suspension, holding deflocculant mass content (X_d) [kg deflocculant/kg dry solid]. The second group of tests consisted of varying the deflocculant fraction, holding the solids content. Finally the effect of milling temperature was studied.



Figure 1. Relation between milling time and mass oversize at 63 µm (laboratory test).

The composition of the clay mixture used was kept constant in all the tests and contained: 65% Villar clay and 35% Moró clay, working at a 63% solids content and 0.4%deflocculant percentage.

To determine the milling time required to reach an oversize close to the one obtained in the industrial suspension, the curve was determined that relates oversize to milling time (Figure 1). It is to be noted that keeping the oversize constant does not guarantee that milling will be similar in a laboratory mill and in an industrial mill, since it is necessary to keep in mind that geometric and operating characteristics (ball size, rotating speed, etc.) are quite different. This means that scaling up laboratory results to an industrial scale needs to be done with due caution.

4.1.1 Variation of solids content

Figure 2 plots viscosity and oversize versus solids content. It can be clearly observed that both variables are sensitive to changes in solids content. On increasing suspension solids content the oversize is larger, possibly due to the presence of a greater quantity of material during milling. There is a direct relation between viscosity and oversize.

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However, if a full particle size distribution is determined (Figure 3), a decrease in oversize is not found to be associated with a modification of particle distribution; i.e., milling, even on a laboratory scale, only reduces the size of the coarsest particles.



Figure 2. Variation of viscosity and oversize with solids content (laboratory tests).

4.1.2 Variation of deflocculant content

To study the effect of deflocculant content (X_d) on suspension properties, three millings were performed keeping the solids content close to that of the industrial suspension and varying the deflocculant content.

The quantity of initial deflocculant was the amount employed industrially. Based on this average value two tests were conducted, in one decreasing and in the other increasing the quantity of deflocculant present in the suspension by 0.1%. Figure 4 plots oversize and viscosity versus the percentage of deflocculant present in the suspension.



Figure 3. Variation of particle size distribution with solids content (laboratory tests).

As was to be expected, the variable that most changes with variations in deflocculant content is viscosity. The variation of viscosity with deflocculant content is not linear, just as occurred with solids content change. The first stretch of the curve has a very steep slope, which indicates that small variations in deflocculant content produce a great change in viscosity. As the deflocculant rises, the curve slope decreases.

The rest of the analysed variables (density and oversize) do not alter on varying deflocculant content. Density is obviously constant, as initial solids content was not modified.



Figure 4. Variation of viscosity and oversize with deflocculant content (laboratory tests).

4.1.3 Variation of milling temperature

To study the effect of temperature on suspension characteristics, milling was performed after previously heating the ball mill and raw materials to 40 °C. The mill charge corresponded to the average values tested before. Table 1 details the result, comparing it with the one obtained at ambient temperature. The temperature change produces a variation in viscosity; however, after the suspension cools, viscosity is the same in both cases. No change was observed in the other suspension properties, so that the only effect that temperature has on the milling is a reduction in viscosity owing to the rise in temperature, although it would seem to be, a priori, a reversible phenomenon.

	Test temperature (°C)		
-	24	40	
ω _s (%)	62.4	62.4	
X _d (%)	0.4	0.4	
ρ (g/cm³)	1.66	1.66	
μ (cp)	1850	1600	
R ₆₃ (%)	8.4	8.5	

Table 1. Results obtained on varying milling temperature (laboratory tests).

4.2 INDUSTRIAL SCALE TESTS CONDUCTED

4.2.1 Test approach and data processing

To relate the variations in feed to the changes in suspension properties, a series of alterations were effected in the industrial mill. In each action, only one variable of the feed was altered and its effect on the resulting suspension was analysed.

The actions were as follows:

1. Variation of solids content.

2. Variation of deflocculant content.

3. Variation of clay mass flow (production).

The solid, water and deflocculant mill input flow rates were varied in a range that was considered reasonable in standard operating conditions.

- Dry solid mass flow rate (m _{si}):	23,000 - 25,000 kg/h
- Water flow rate (Q _{wi}):	11,000 - 13,000 1/h
- Deflocculant flow rate (Q _{di}):	250 - 400 1/h

Each variable was modified, increasing and decreasing it, with regard to nominal operating conditions. This meant a total of 4 changes in the settings. Each of these changes was monitored for 30 minutes, taking suspension samples at the mill exit every 5 minutes. The initial operating conditions before each action were characterised for 15 minutes to make sure that before carrying out any action, the mill was working under steady conditions. All the suspension samples taken were analysed in the laboratory, determining their solids content, viscosity and oversize mass at 63 μ m.

With a view to quantifying mill behaviour when a modification takes place in the feed, it was attempted to relate each output variable with the modified input variables by an equation.

In control theory, the quantitative relation between inputs and outputs is determined by a mathematical function known as the "transfer function". This function allows parameterising two aspects of the response: on the one hand, it indicates the magnitude of change in an output variable on modifying an input variable (it indicates, for example, how much viscosity changes on modifying solids content in a given measure); and on the other hand, it indicates the velocity at which this change takes place (using the previous example, it quantifies the rate at which viscosity changes, until reaching a new steady value).

The form of the transfer function depends on the process being studied. However, in most cases a simple equation is used, which depends on three parameters, determined by fitting the experimental data. Simply expressed, the transfer function is the Laplace

transform of the function that relates inputs and outputs. This function is usually found from a differential equation ^[3], such as:

$$\tau_{p} \frac{dy'}{dt} + y' = K_{p} x'(t - \theta)$$
 (Ec. 1)

which has been adopted in this work. x' corresponds to the input variable (initial solids content, deflocculant content, etc.) expressed as the difference between the instant real value and the nominal one; y' represents the output variable (suspension solids content, temperature, etc.) also expressed as the difference with regard to the nominal value. The parameters of the pattern to be determined experimentally are $K_{p'} \tau_p$ and θ , whose physical meaning is as follows:

- K_p: Represents the relation between the change in the output variable and the change in the input variable.
- τ_p : Corresponds to mill response time. The larger τ_p is, the more slowly the system responds (mill) to input changes.
- θ: This is the time that elapses for the output variable to start presenting a change due to the alteration in the input variable.

Figure 5 graphically illustrates a disturbance step in an input variable (x') and the response of an output (y') as a function of time. Moreover, the physical meaning is indicated of the three parameters involved in the transfer function.



Figure 5. Physical meaning of the parameters involved in the dynamic model.

 τ_p and θ together quantify the velocity with which the mill responds. With a sharp fast change in the form of a step in the input variable, if θ was zero, the mill would reach 63% of the maximum response after a time equal to τ_p and 86% after a time equal to $2\tau_p$. If θ was not zero, these responses (63% and 86%) would be reached respectively after time $\theta + \tau_p$ and $\theta + 2\tau_p$.

4.2.2 Modification of feed solids content

Effect on suspension solids content at the mill exit

The input solids content, under standard mill working conditions, was determined starting from the nominal dry solid, water and deflocculant flow rate values. The actions consisted in altering this by 1% in both directions. Table 2 gives the nominal flow rates, solids content (ω_{si}), and deflocculant content (X_{di}) of the feed (the subscript *i* refers to it being an input variable). The solids and deflocculant content flow rates were held steady.

The points plotted in Figure 6 correspond to the experimental values found in the industrial test, and the solid lines to the values calculated theoretically from equation 1. The same graph shows the results of the two solids content modifications in the feed: that of ω_{si} increase \rightarrow reduction (action 1A) and that of ω_{si} reduction \rightarrow increase (action 1B). The figure also plots the instant at which the first change occurs in the solids content (rise in ω_{si} in action 1A and decrease in ω_{si} in 1B).

Situation	m _{si} (kg ds/h)	Q _{wi} (l/h)	Q _{di} (l/h)	ω _{si} (%)	X _{di} (%)
Initial	25000	12400	270	66.8	0.3
ω _{si} up	25000	13000	270	65.8	0.3
Initial	25000	12400	270	66.8	0.3
ω _{si} down	25000	11800	270	67.8	0.3
Initial	25000	12400	270	66.8	0.3

Table 2. Nominal mill input values on modifying solids content.

Table 3 shows the values of the model parameters. The values of the parameters found in the two actions (1A and 1B) should be the same, under ideal conditions; however, they are not partly because the mill does not behave exactly as equation (1) predicts, and partly also because the samples were taken every 5 minutes; there is therefore a certain lack of accuracy in the definition of τ_{0} and θ .

Parameter	Action 1A	Action 1B	
K _p	1.35	1.15	
τ _p (min)	13.0	10.0	
θ (min)	10.0	12.0	

Table 3. Parameters found in the theoretical fits.

These results are of great importance, since they allow characterising milling process dynamics from a control point of view, i.e., they enable quantifying how fast the mill responds on modifying the input variables. Considering that most of the response was reached after time $\theta+2\tau_p$, this gives an approximate value of 35 minutes. This is obviously whenever small departures from nominal operating conditions are considered.



Figure 6. Theoretical fit of solids content at the mill exit.

Effect on suspension oversize at the mill exit

The effect of input solids content on suspension oversize mass is plotted in Figure 7. It shows that neither alteration in solids content produces a defined effect on oversize; this always stays around the same value, albeit with certain swings.



Figura 7. Plot of oversize on raising input solids content.

This result differs from the one found in the laboratory tests, in which an increase in solids content produced a rise in oversize. This difference in behaviour can be due to the effect of ball size. In the laboratory mill, the smaller ball size more efficiently reduces particle size, while in the industrial mill, the minimum achievable particle size is possibly already close to being achieved with the ball distribution involved.

Effect on suspension viscosity at the mill exit

The effect of modifying solids content on suspension viscosity is shown in Figure 8. The decrease in input solids content by a percentage point leads to a drop in suspension viscosity of around 900 cp; in contrast, raising solids content by a point leads to a rise in viscosity of about 2500 cp. This quite non-linear behaviour is characteristic of viscosity. It is in fact this behaviour that limits the use of equation (1) in studying viscosity. Indeed, equation (1) is linear and therefore, independently of the values of K_p , τ_p and θ , this equation predicts that the magnitude of change in the output variable should be the same on raising or lowering the input variable. In Figure 8, the solid line shows the results obtained with equation 1, providing a poor fit, particularly in the action ω_{si} increase \rightarrow decrease.



Figure 8. Theoretical fit of viscosity.

Besides measuring viscosity, thixotropy was quantified as the measurement of viscosity after a 6 min rest of the suspension (μ (6 min)). The relation between both magnitudes (viscosity: μ (1 min) and μ (6 min)) is shown in Figure 9, where the relation is observed to be linear. This justifies concentrating on viscosity to characterise the variation in the rheological state with changes in input variables. Possibly, if the change in the input variables had been bigger, this linear relation might not have held.



Figure 9. Relation between measured viscosity after 1 and 6 min suspension rest on modifying solids content.

The viscosity of a suspension can be related to solids content by means of a semiempirical equation. For concentrated suspensions with Newtonian rheological behaviour, the equation used is the Krieger-Dougherty equation ^[4], which can be written as:

$$\frac{\mu}{\mu_s} = \left(1 - \frac{\phi}{\phi_m}\right)^{-[\mu]\phi_m}$$
(Ec. 2)

where:

- μ : suspension viscosity (Pa·s)
- μ_s : viscosity of the suspending phase (water, Pa·s)
- [µ]: intrinsic viscosity, related to particle shape and deflocculation state (dimensionless).
- φ: solids volume fraction (dimensionless)
- ϕ_m : maximum solids volume fraction (dimensionless)

On occasions a variant of the equation (2) is used, written as follows:

$$\frac{\mu}{\mu_s} = \left(1 - \frac{\phi}{\phi_m}\right)^{-[\mu]} \tag{(Ec. 3)}$$

This has been the expression used in this study, as it fitted the experimental results better.

The solids volume fraction in a suspension (ϕ) is related to solid density, suspension density and solids content. It can be calculated from the following expression:

$$\phi = \frac{\frac{\rho_{w}}{\rho_{s}}\omega_{s}}{1 - \left(1 - \frac{\rho_{w}}{\rho_{s}}\right)\omega_{s}}$$
(Ec. 4)

where:

- ρ_w : water density [kg/m³]
- ρ_s : solid density [kg/m³]
- ω_s : solids content [kg dry solid/kg suspension]

The maximum particle volume fraction in a suspension (ϕ_m) is related to particle size distribution (PSD) and to the suspension agglomeration state. In principle this relation is not known, but it can be assumed that, for a small range of variation in deflocculant (ω_d) and oversize (R_{63}) , given the relation between PSD and R_{63} , and between agglomeration state and X_d , it will approach the following linear expression:

$$\phi_m = \phi_{m0} + A_d X_d + A_R R_{63} \tag{Ec. 5}$$

where:

 ϕ_{m0} : maximum volume fraction, corresponding to $X_d = 0$ and $R_{63} = 0$.

- A_d: factor that relates viscosity to deflocculant content
- A_{R} : factor that relates viscosity to oversize
- X_d : suspension deflocculant content (%)
- R₆₃: milling oversize mass (%)

Substituting equations 4 and 5 in 3, an expression is found that relates viscosity to solids content, in which parameters $[\mu]$, $\phi_{m0'}$, A_d and A_R are not known

Using the experimental values, the foregoing parameters can be found by applying a least squares fit for viscosity. This fit is shown in Figure 10. The fit is observed to be quite good. The values obtained were as follows:

$$\phi_{m0} + A_d X_d = 0.613$$

[µ] = 8.5



Figure 10. Theoretical fit of viscosity with the Krieger- Dougherty equation.

As the deflocculant content remained constant, ϕ_{m0} and A_d could not be determined separately in equation (5). Furthermore, since A_R could not be calculated because the values of R_{63} were very similar to each other and had no clear effect on viscosity, the assumption was made that $A_R=0$.

4.2.3 Modification of deflocculant content

The variations in mill input deflocculant content were approximately $\pm 0.05\%$. The nominal values of the feed flow rates, as well as the solids and deflocculant content calculated from these flows are given in Table 4.

Situation	m _{si} (kg ds/h)	Q _{Wi} (l/h)	Q _{di} (l/h)	∞ _{si} (%)	X _{di} (%)
Initial	25000	12200	270	67.12	0.307
X _{di} down	25000	12220	250	67.12	0.260
Initial	25000	12200	270	67.12	0.307
X _{di} up	25000	12160	308	67.12	0.350
Initial	25000	12200	270	67.12	0.307

Table 4. Nominal mill input on modifying deflocculant content.

Effect on suspension solids content and oversize at the mill exit

Figure 11 shows that, on altering the deflocculant content, the solids content fluctuates by $\pm 0.3\%$; however, this fluctuation cannot be associated with the changes in deflocculant content. The presence of fluctuations could be related to material accumulations inside the mill, which would generate heterogeneities in the properties of the resulting suspension. Normally these variations are not important because the material ends up being homogenised in the storage tanks prior to spray drying; however, they should be kept in mind when designing the control system.



Figure 11. Evolution of solids content on modifying deflocculant content.

The actions on the deflocculant did not significantly affect suspension oversize. Figure 12 shows how the oversize swings, without exhibiting any clear tendency.



Figure 12. Effect of deflocculant modification on oversize.

Effect on suspension viscosity at the mill exit

Deflocculant content affects suspension viscosity, as shown in Figure 13. Decreasing the deflocculant content evidently raises viscosity. Together with this reasonable behaviour, viscosity is also observed to undergo short, abrupt variations (local peaks). These peaks (plotted in Figure 13 as crosses) are unrelated to anything that could happen at the mill input, since if this was the case, the peaks would be flatter (smaller magnitude, and last a longer time). They could be due, as before, to processes relating to what happens inside the mill (for example, the exit at a given moment of material accumulated inside the mill, etc.); in any event, the causes of these changes are not clear.



Figure 13. Theoretical fit with the Krieger-Dougherty equation.

Fitting the experimental data of this action to the Krieger-Dougherty equation is a little delicate. It is necessary to remember that as postulated, in this equation parameter ϕ_m depends on deflocculant content X_d according to equation (5). The difficulty of calculating ϕ_m is that the value of X_d is unknown (its concentration in a suspension cannot be analysed in a simple way). For this reason, it was decided to estimate it from the mill input deflocculant content, and use equation (1) to evaluate its elapsing time. The τ_p and θ values of this model were assumed to be similar to those used in fitting the solids content ($\tau_p = 13 \text{ min and } \theta = 10 \text{ min}$). The value of K_p was considered similar to the one set, as it is reasonable to assume that a variation of 1% in mill input deflocculant content will, when a new steady value is reached, lead to a variation of 1% in suspension deflocculant content. The estimated value of mill output deflocculant content is plotted in Figure 14.



Figure 14.Estimated elapsing time between mill input deflocculant content (X_{a}) and mill output deflocculant content (X_{a}) .

In this experiment the assumption was made that $A_R=0$ (equation (5)) as no correlation had been observed between oversize and viscosity. Due to the fact that X_d varied, the values of $\phi_{m0'}$ A_d and $[\mu]$ of the Krieger-Dougherty equation could be independently determined. The resulting fit is shown in Figure 13 (solid curve). The values found for the parameters were:

$$\phi_{m0} = 0.523$$

 $A_d = 0.34$
 $[\mu] = 8.5$

If these values are compared with those obtained on varying solids content, it is observed that $[\mu]$ is the same; however, the values of ϕ_{m0} and A_d are not consistent with those determined in the first action (modification of solids contents); in fact, if $\phi_{m0} + A_d X_d$ is calculated from the previous data, and considering the value of X_d in the first action, this would give $\phi_{m0} + A_d X_d = 0.627$ (compared with 0.613, which was the value found in the first action). This means that ϕ_{m0} (or A_d) is not constant, possibly owing to a variation in the deflocculation state or a modification in the solid input, in view of the fact that the first and second modifications were not carried out on the same day. Nevertheless, with the data available it is not possible to establish their relation with any other easily measurable variable.

4.2.4 Modification of clay mass flow rate

With a view to establishing the effect of clay mass flow rate on suspension properties, this flow was modified while holding solids and deflocculant content.

Since at the moment of performing the action, the solid mass flow input into the mill was the maximum allowed by the facility (25,000 kg/h), the two disturbances consisted of two decreases, then raising the result in two steps to the initial value. Table 5 presents the nominal values in each situation.

As was to be expected, no significant change occurred in solids content, oversize or viscosity on varying clay mass flow rate.

Situation	m _{si} (kg ds/h)	Q _{wi} (l/h)	Q _{di} (l/h)	ω _{si} (%)	X _{di} (%)
Initial	25000	12400	300	66.7	0.34
m _{si} down	24000	11900	288	66.7	0.34
m _{si} down	23000	11400	276	66.7	0.34
m _{si} up	24000	11900	288	66.7	0.34
m _{si} up	25000	12400	300	66.7	0.34

Table 5. Nominal mill input values on modifying clay mass flow rate.

Effect on suspension temperature at the mill exit

The temperature of the suspension at the mill exit was measured during the previous actions, no changes being observed either on varying the solids content of the feed or modifying the deflocculant content. However, during this last action, a variation in exit temperature was detected.

Suspension temperature increases on decreasing mill production, and drops on raising it again. This variation can be observed in Figure 15. The change can be simply due to the difference between the enthalpy of the suspension at the exit and that of the feed $(\Delta H = (m_{si}/\omega_{si})c_p(T-T_i))$ practically remaining constant when production varies (within certain limits). Therefore, on decreasing suspension mass flow rate (m_{si}) , the output temperature should rise (T).



Figure 15. Effect of solids feed flow rate on suspension temperature.

Using equation (1), it was attempted to find a relation between both variables. The fit is shown in Figure 15 (solid line), and is quite good. The parameters of the fitting equation are detailed in Table 6.

Value
-0.45
10.0
20

Table 6. Parameters of the equation fitting viscosity.

The value of K_p found means that a change of a ton per hour in the solids mass flow input into the mill produces a modification in suspension temperature at the exit of 0.45 °C. The value of K_p is negative because on increasing production, temperature decreases. It should be noted that the value of $\theta + 2\tau_p$ is similar to the one found in the other cases in which the equation was applied (1).

5. CONCLUSIONS

The study allows drawing the following conclusions:

- On a laboratory scale the effect has been studied of solids content, deflocculant content and milling temperature on viscosity and oversize of the resulting suspension. The most significant results are that the initial solids content affects oversize, that deflocculant content only affects viscosity (in particular it does not affect oversize), and that milling temperature does not affect any resulting suspension variable.
- It has been demonstrated that in the industrial mill, there is a direct relation between feed and suspension solids content. A simple model can be used to simulate the behaviour; the characteristic response time was about 35 minutes.
- The oversize on a 63 μ m screen (R₆₃) does not change with the change in solids content. This differs from the result found in the laboratory, where it was observed that increasing ω_s also raised R₆₃. The difference in behaviour between the laboratory mill and the industrial one could be due to the different operating conditions (ball size and rotating speed).
- Viscosity (μ) increases on raising solids content (ω_s). A slightly modified Krieger-Dougherty equation can be used to establish the relation between μ , ω_s and X_d (deflocculant mass content). The maximum volume fraction, under zero deflocculant concentration conditions (ϕ_{m0}), or the parameter that relates deflocculant content to volume fraction (A_d), varies over time, possibly as a result of slight modifications in raw materials or mill operating conditions (level of balls, material accumulation, etc.). In any event, the Krieger-Dougherty equation can be applied to study the variation in viscosity with solids content and with deflocculant content, if small corrections are made in ϕ_{m0} , with time.
- The modification of deflocculant content does not affect suspension oversize.
- When mill production increases, suspension output temperature decreases, but neither R₆₃ nor μ change (in the tested range of production variation).

The work conducted establishes how quickly the mill responds to changes in input variables, and shows the presence of sporadic fluctuations in suspension properties. This information will allow designing a control system that enables regulating suspension density and viscosity by acting on the mill feed variables.

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