Optimization of in-mill ball loading and slurry solids concentration in grinding of UG-2 ores: A statistical experimental design approach

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A R T I C L E   I N F O

Article history:
Received 15 January 2012
Accepted 29 May 2012
Available online 5 October 2012

Keywords:
Process optimization
Comminution
Design of experiments
Slurry

A B S T R A C T

The in-mill load volume and slurry solids concentration have significant influence on the ball mill product size and energy expenditure. Hence, better energy efficiency and quality grind can only be achieved with correct tuning of these influential operational factors to the desired optimum point. In view of the deficiencies of the classical “one-factor-at-a-time” methodology, statistical experimental design methodologies were applied in this study to optimize the slurry % solids and ball load volume during a batch ball milling process of UG-2 ore. The response surface methodology and central composite design were used to determine the best possible combination of ball load volume and slurry % solids for maximum size reduction index and minimum specific energy consumption (kW h/t). Second order response surface models were built to describe the relationship between the input factors and the response variables. Analysis of variance (ANOVA) tests and response surface plots were used to set the optimal level for each input factor. With compromise optimized values of 29% ball load volume and 75% slurry solids, the response surface models yielded specific energy consumption of 10.54 kW h/t and size reduction index of 3.93. Confirmatory experiments carried out in these optimized conditions resulted in specific energy consumption of 10.72 kW h/t and size reduction index of 3.91 thus corroborating the validity of the response surface models.

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1. Introduction

Milling is an important operation in mineral processing as it serves to reduce the size of mineral bearing ore particles to sufficiently finer size to ensure good liberation of the valuable minerals for subsequent processing. However, the process is inefficient and consumes an extensive amount of energy. The efficiency of a wet milling process is affected by both the rate of breakage and slurry transport through the mill; which in turn are dependent on design and operational factors such as liner profile, load volume, slurry properties and mill rotational speed (Austin et al., 1984; Songfack and Rajamani, 1999; Govender et al., 2010). In non-variable speed mills typical of conventional ball mills, slurry properties and load volume known to be the most influential operational factors (Makokha and Moys, 2011; Keshav et al., 2011). The properties of slurry are crucial in achieving efficient flow and distribution of slurry within the load and subsequent transport out of the mill. During milling process, the slurry is normally entrained in the load taking up the voids volume and thus directly influences the flow of fine particles out of the mill and the distribution of coarse particles to the breakage fields as well as the motion and behavior of the grinding media. On the other hand, the level of mill filling (load volume) defines the regime of load motion inside the mill and the intensity of milling. These events impact heavily on energy consumption and size reduction rate. A good understanding of the main and interaction effects of the two operational factors on mill performance is vital in establishing optimal factor settings for efficient milling. Thus the underlying objectives of this study are 2-fold: Firstly to assess the combined effects of ball loading and slurry % solids on the milling process of UG-2 ores and secondly to apply statistical experimental design approach to identify the conditions of ball loading and slurry % solids that optimizes ball milling process of UG-2 ores in terms of energy consumption inside the mill (kW h/t) in production of sub-75 μm particles and size reduction index (i.e. the amount of material less than 75 μm in the product stream relative to the amount in the feed stream). In deed with today's increasing focus by industrialists on expanding production capacities and minimizing costs (so as to gain and maintain a competitive edge), it is essential that all critical unit operations in industrial processes are optimized.

The two types of optimization commonly considered in mineral processing circuits are: (i) topological which concerns the arrangement of process equipments and (ii) parametric which deals with operational factors (Edgar and Himmelblau, 2001). While topological optimization has often been successfully
achieved by engineers, parametric optimization has remained challenging due to the tendency to apply the classical one-Factor-at-a-time (OFAT) approach where other variables are fixed thus relaxing the interaction effects between variables (Antony, 2003). This approach requires large resources to obtain only a limited amount of information of the process, hence it could easily lead to false optimum conditions for the process. Further, since the results are valid only under fixed experimental conditions, prediction based on them for other conditions is uncertain. In order to achieve valid, reliable and sound conclusions effectively, efficiently and economically, the statistically-based methods of design of experiments (DOEs) such as response surface methodology and central composite design are applied (Box et al., 1978). Due to the statistical balances in the design, a number of potential combinations of input factors at different levels can be evaluated for the best overall combination, in a small number of experiments. DOE was introduced in early 1920s by Ronald Fisher and has since been widely accepted and successfully applied in industrial process optimization.

In this work, parametric optimization of a wet ball milling process for UG-2 ores has been studied using response surface method (RSM) and central composite rotatable design (CCRD). The candidate process factors were ball loading and slurry % solids. The RSM was used to determine the optimum factor levels while the CCRD was utilized to properly distribute experiments within the factor space and collect the data for fitting the regression model that describes the response surface. The response surface is basically a map showing how the output variable changes at different level-combinations of the design factors. The optimum response could be a maximum or a minimum of a function of the design factors. For uniform comparison and computational ease, all factors were transformed from their actual values to codified values. The experimental results were statistically analyzed using ANOVA. The correlations between the design factors and the responses were adequately described by polynomial models. As will be seen later in this paper, a close match between the model and confirmatory experimental test results demonstrates that the statistical experimental design approach does indeed find the optimal level combination of input parameters that would result in efficient milling process.

2. Experimental

2.1. Equipment, materials and methods

The experimental work carried out for this project was accomplished using a 3 dimensional laboratory scale mill constructed from steel and mounted on a mill rig. The mill is driven by a 2.5 kW motor via a chain drive. The motor speed is controlled electronically using a speed controller (tachometer), which regulates the power supply to the motor by varying the current frequency. The milling chamber measures 550 mm in diameter by 400 mm long (inside liners) and is fixed to the mill axle on which the load beams are connected for torque measurements. The mill is lined with 18 pieces lifters that were suitably scaled down from industrial size. A 10 mm thick PVC disk is used to close the front side of the mill with a provision for feeding and discharging the mill contents during batch tests. The torque yielded by the load beam as a result of the tumbling load is transmitted to the computer as a voltage signal for processing and storage. A desktop computer which is interfaced to the data acquisition system is utilized for real time data processing using the Waveview® program from Eagles Technology.

A sample from the underflow of the primary cyclones at Anglo-Platinum UG-2 Concentrator was used as feed material. The size distribution of the feed material is presented in Fig. 1. All experimental tests were performed in batch-wise mode. The volume of slurry (slurry holdup) was kept constant (22 L) to mimic conditions of an overflow mill. The interstitial filling, U varied with the ball load volume as follows: $U = 1.5$ (J38%), $U = 1.7$ (J53%), $U = 2.1$ (J30%), $U = 2.6$ (J25%), $U = 2.8$ (J22%). Steel balls measuring 10 mm in diameter were used as grinding media. An analysis of the mass specific energy consumption in producing particles finer than 75 μm over a set period of grinding time was performed to study the relationship between two mill operating factors (i.e. slurry solids concentration and load volume) and energy utilization inside the mill. Since experimental tests were performed on a batch mill, then an index used to define specific energy consumption was calculated as in Eq. (1), where, $E_{75\mu m}$ is the specific energy consumption (kW h/t), $P$ is the mill power (kW), $P_0$ is the no-load power (kW), $m_i$ is the mass of slurry inside the mill (tons), $C_w$ is the weight fraction of solids in slurry, $t$ is the grinding time (mins), $S_i$ is the fraction of in-mill material <75 μm at time t = 0, $S_i$<75μm is the fraction of in-mill material <75 μm after grinding time, t.

$$E_{75\mu m} = \frac{(P - P_0)t}{60(C_w - S_i) - S_i}$$  \[1\]

The reduction index is defined here as the cumulative percentage of particles finer than 75 μm in the product relative to the percentage in the feed.

$$R_{75\mu m} = \frac{\% \text{Product}_{<75\mu m}}{\% \text{Feed}_{<75\mu m}}$$  \[2\]

The particle size distributions (PSD) were obtained by standard wet–dry sieving procedures using a vibratory sieve shaker. A small sample of about 400 g was removed from the mill after each test run and split to obtain a sample that is representative of the properties of both coarser and finer materials in the mill. A representative sample of about 80–90 g was then wet washed on the screen size (38 μm) to remove the fines. The wet samples were dried in an oven for 20 min followed by the sieving of each sample in separate runs but using the same sieves on the shaker machine. The reason for maintaining the same sieves in all the test runs is to ensure consistency in the results. This is given the fact that different sieves have different reliabilities despite having same nominal aperture size. During the successive sieving intervals, the screens were cleaned with compressed air to avoid the ‘blinding’ effect that would lower the reliability of the sieves. The duration of sieving in all the tests was 20 min which was considered sufficient for all the undersize to be separated. At the end of each sieving test, the material retained in each screen interval was weighed and its mass expressed as a percentage of the total mass after screening, including the mass washed out. Similar procedures were followed in all experimental runs.
pure error. Also, the contrast between the mean of the center studied was approximated using Eq. (4) while the axial values (\(n_c \times i\), augmented by 2 k axial points and rotatable design (CCRD). The CCRD comprises 2 k factorial points in influence on mill performance. Table 1 gives an outline of the factors, both of which have been shown to have a significant was carried out over a range of controlled conditions for the two composite design

2.2. Experimental design: response surface methodology and central composite design

Two candidate design factors for this test work were in-mill slurry solids concentration and ball load volume. The test work was carried out over a range of controlled conditions for the two factors, both of which have been shown to have a significant influence on mill performance. Table 1 gives an outline of the experimental layout and standard runs for the central composite rotatable design (CCRD). The CCRD comprises 2 k factorial points (coded \(\pm 1\)), augmented by 2 k axial points and 2 replicate points at the center. The terms k and \(k\) represent the number of factors studied and the distance of an axial point from the center respectively. Since the reason for using response surface analysis is to locate unknown optimal operational region, it is worthwhile to use a rotatable design that provides equal precision of estimation.

The transformation from actual to coded values is given by (Montgomery, 2005; Napier-Munn, 2000), where \(x_i\) is the coded value of variable \(x\) (=actual value of the variable) while \(x_{\text{max}}\) and \(x_{\text{min}}\) are the upper and lower limits of \(x\):

\[
x_i = \frac{2x - (x_{\text{max}} + x_{\text{min}})}{(x_{\text{max}} - x_{\text{min}})}
\]  

(3)

The number of center point replications for the two factors studied was approximated using Eq. (4) while the axial values (\(z\)) are given by \(2^{k/4}\) (Khuri and Cornell (1987)):

\[
n_c = 0.84(2^{3k/4} + 2)^2 - 2^k - 2k
\]  

(4)

Replicated points at center points can be used to calculate the pure error. Also, the contrast between the mean of the center points and the mean of the factorials points provides a test for lack-of-fit (LOF). The significance of LOF test can show if the curvature or higher order term of the factor is present or not.

The interaction plot (Fig. 2) is used to assess whether or not the two factors \((A \text{ and } B)\) have an interaction effect on the responses. As the lines are non-parallel, the effect of factor \(B\) (slurry solids concentration) on the two responses is dependent on the level of factor \(A\) (load volume). The crossing of lines suggests that the nature of interaction is antagonistic. We notice a higher degree of departure from parallelism in the graph for kW h/ton than that of reduction index, which clearly indicates the presence of a stronger interaction effect between factors \(A\) and \(B\) on the specific energy consumption (response variable).

A second order polynomial regression model (Eq. (5)) is proposed to predict the response surface based on the two operational factors under consideration:

\[
Y = \beta_0 + \sum_{j=1}^{k} \beta_j X_j + \sum_{j=1}^{k} \beta_{jj} X_j^2 + \sum_{i<j}^{k} \beta_{ij} X_i X_j + \varepsilon
\]  

(5)

In which \(Y\) is the predicted response, \(X_i, X_j\) are the coded independent variables, \(\beta_0\) is the intercept, \(\beta_j\) is the linear term coefficient, \(\beta_{jj}\) is the quadratic term coefficient, \(\beta_{ij}\) is the interaction term coefficient and \(\varepsilon\) represents other sources of variability un-accounted for by the model.

3. Results and discussion

3.1. Structure and derivation of the fitted model

The structure of the second order surface response models proposed in this study relating to specific energy consumption and the size reduction index is given by Eq. (5) in the previous section. The
values of the coefficients were obtained by least squares method as $\beta = (X'X)^{-1}X'Y$ using MATLAB R2007a and the fitted regression models are:

$$
Y_{(\text{kWh/ton})} = 11.83 + 0.83(A) - 0.48(B) + 0.47(A^2) - 0.25(B^2) + 0.99(AB) \tag{6}
$$

$$
Y_{(R)} = 3.68 + 0.23(A) + 0.37(B) - 0.29(A^2) - 0.13(AB) \tag{7}
$$

Notice that both the ball load volume and the slurry %solids hold an appreciable influence on specific energy consumption (kWh/ton) by the mill. The non-linear trend in variation of specific energy consumption with ball load volume and slurry % solids attests to the fact that, in a multivariate milling environment, better energy efficiency can only be attained with correct tuning of operational factors to the desired optimum point. Since the objective function is to minimize the kWh/ton and maximize the reduction index, then the positive coefficient of $A$ and negative coefficient of $B$ in Eq. (6) suggest that the response would be optimal at low and high level settings of $A$ and $B$ respectively. Equally, the positive coefficients of $A$ and $B$ in Eq. (7) suggest that an optimal response would exist when both $A$ and $B$ are at high level settings.

### 3.2. Model adequacy checking

Each of the fitted models was checked to determine if it provides an adequate estimation of the true response surface. This was accomplished using Fisher's variance ratio test ($F$-test). Student's $t$-test and coefficient of determination ($R^2$) and the results are presented in Tables 2 and 3. For both model 1 (Eq. (6)) and model 2 (Eq. (7)), the observed $F$-value of the regression was higher than the critical $F$-value, implying that the regression in both cases is significant at the 5% significance level. Further, the $F$-value for lack-of-fit in both cases was observed to be lower than the critical $F$-value, implying that the two models present no evidence of lack-of-fit. The Student's $t$-test was employed to assess the significance of individual regression coefficients. The $t$-statistics ($t_0$) was compared with the critical value at 10% significance level (i.e. $t_{0.10} = 1.84$ and $t_{0.05} = 1.89$). It was found that for model 1, the constant term, the linear terms, the $A$ quadratic term and the interaction term are all significant. For model 2, all the terms except the $AB$ interaction term were found to be statistically significant.

Presented in Fig. 3 are comparisons of experimental results of specific energy consumption and reduction index versus predictions by the second order regression models. It can be seen that the regression models described the data adequately with the linear correlation coefficient (i.e. the amount of sample variation explained by independent variables) of $R^2 = 79\%$ and 82\% for models 1 and 2 respectively.

### 3.3. Response surfaces

Figs. 4 and 5 show 3D surfaces as well as contour plots of the relationships between the two control variables ($A$ and $B$) and the responses. Fig. 4 shows the effects of slurry solids concentration and load volume on the specific energy consumption by the mill in producing new particles finer than 75 $\mu$m. The results indicate a reduction in energy consumption as slurry solids concentration increases for levels of load volume below the center point. This trend partly agrees with the observations by Clermont et al. (2008). For load volumes above the center point, the energy consumption appears to increase with increase in both load volume and slurry solids concentration which is consistent with the experience in milling practice. It may sound logical that the additional ball load would tend to shift the center of gravity of the now increased load volume towards the mill center which reduces the length of the torque-arm. However, the torque does not reduce since this effect is counteracted by the increased mass of the load. Again, increased volume of the load results in high dynamic pressure exerted by the load on the mill walls. This helps to lock the load to the mill rotary motion subsequent to which a higher lifting action is experienced and the mill torque and power increase accordingly. Similarly, increased solids concentration improves the friction coefficient between the load and the mill wall which in-turn promotes the lifting action. This in effect would increase the impact energy but reduce the abrasion energy where the latter is largely responsible for production of finer particles (sub-75 $\mu$m). These events would subsequently lead to an increase in the specific energy consumption in production of new sub-75 $\mu$m particles. The unsteady trend in specific energy consumption clearly demonstrates the complex nature of the relationship between mill energy efficiency and operational factors whose understanding hitherto remains obscure.

Fig. 5 shows the effect of ball load volume (factor $A$) and slurry solids concentration (factor $B$) on the reduction index of particles. The generation of new particles below 75 $\mu$m appears to be influenced significantly by both factors. The reduction index can be observed to follow an increasing trend with increase in slurry
solids concentration. However, the reduction index is lower at both low and high load volumes but higher at center point region (actual load volumes of between 26% and 32%). These results suggest that the choice of desirable level of slurry solids concentration and load volume is paramount to achieving optimal reduction index and energy efficiency.

3.4. Determining the optimum conditions

The interest here is to determine conditions that maximize the particle reduction index and minimize the specific energy consumption (kW h/ton) in production of new particles <75 μm. Having established the adequacy of the fitted models (Eqs. (6) and (7)) within the design space, the coordinates of the stationary points can then be located using the fitted models. The stationary point is a combination of design factors where the surface is either at minimum, maximum, saddle or ridge. The objective functions can be expressed in matrix form as:

\[ \mathbf{y} = \mathbf{b}_0 + \mathbf{x}^T \mathbf{b} + \mathbf{x}^T \mathbf{B} \mathbf{x} \]  

The stationary point \((x_s)\) exists if the derivative of Eq. (8) with respect to the elements of the vector \(x\) is equal to zero. The point \(x_s\) can be found using matrix algebra (Nuran, 2007) as \(x_s = -0.5\mathbf{B}^{-1}\mathbf{b}\) and the estimated response at the stationary point is given by, \(\hat{y}_s = \mathbf{b}_0 + 0.5\mathbf{x}_s^T \mathbf{b}\), where \(\mathbf{b}\) is \((k \times 1)\) vectors of first order coefficients while \(\mathbf{B}\) is \((k \times k)\) symmetric matrix with quadratic coefficients as main diagonal elements and half-interaction coefficients as off-diagonal elements. The conditions at the stationary point for model

![Fig. 3. Relationship between measured and predicted results of \(E_{75\mu m}\) and \(R_{75\mu m}\).](image)

![Fig. 4. (a, b) Response surface and contour plots of the effect of load volume (A) and slurry % solids (B) on the specific energy consumption (kW h/ton) in production of new particles <75 μm.](image)
1 were obtained in coded values as $x_1 = -0.0415$ and $x_2 = 0.8778$ along the path of steepest descent, giving a response of 11.15 kW h/ton. The stationary point for model 2 was located at conditions of $x_1 = 0.0415$ and $x_2 = 0.8778$ lying along the path of steepest ascent, yielding a reduction index of 3.89. The stationary points lie within the design bounds of the region of exploration in this study, i.e. $-1.414 < x_1 < +1.414$; $i = 1, 2$; where $x_1 = A$ and $x_2 = B$ (coded levels of process variables).

To determine the nature of the stationary points, the method of canonical analysis was applied. The models were first transformed into a new coordinate system with the origin at the stationary point and the axes of the system rotated to be parallel to the principal axes of the fitted response surface. Details of the canonical analysis technique can be reviewed in the work by (Khuri and Cornell (1987); Myers and Montgomery, 2002). The eigen values were obtained as $(0.72, 0.50)$ for model 1 and $(0.30, 0.01)$ for model 2, which results in the following canonical form of the fitted models:

\[
\hat{y}_{(\text{kWh/ton})} = 11.15 + 0.72Z_1^2 - 0.50Z_2^2 \quad ; \quad \hat{y}_R = 3.89 - 0.32Z_1^2 + 0.01Z_2^2
\]

where $Z_1$, $Z_2$ are transformed independent variables (canonical axes). The mixed signs of the canonical coefficients imply that the stationary points are saddle points and not the optimal solution. Thus, the method of ridge analysis (Myers and Montgomery, 2002) has been used to determine the potential optimum factor levels of the response surfaces at fixed distances from the center of the experimental region. Since this study involves two factorial points, the boundary of the experimental design is assumed to be at a radius of $\sqrt{2}$ from the design center point. The optimal levels of the variables were obtained as, $x_1$ or $A = (-0.546, 0.125)$ and $x_2$ or $B = (1.144, 0.793)$ in coded values yielding responses of $Y = (10.02, 3.98)$ using Eqs. (6) and (7) respectively. The respective actual un-coded values of variables were: Load volume (%) = (27.3, 30.6) and Slurry % solids = (76.4, 72.9).

The results suggest that with other factors held constant, better energy efficiency in grinding could be achieved at lower ball loading of 27.3% (~27%) and high slurry concentration of 76.4% (~76%) while higher reduction index could be attainable at a relatively higher ball loading of 30.6% (~31%) and lower slurry concentration of 72.9% (~73%) solids. In essence, it implies that grinding is likely to proceed more efficiently at lower ball loading and high slurry % solids or higher ball loading and lower slurry % solids. However, one would argue that the cost associated with capacity losses at lower ball loading or media wear at lower slurry solids concentration will offset the potential gain in milling efficiency. Hence in practice, it is important to find a compromise optimum in setting the operating points to ensure more profits are realized (Oehlert, 2000).

3.5. Confirmatory tests

Confirmatory tests were performed at a compromise optimum setting of 29% ball load volume and 75% solids concentration in slurry to test the validity of the conditions as suggested by the models. The specific energy consumption was found to be 10.72 kW h/t while the reduction index was 3.91 both of which
compare closely with the compromise optimal values (see Table 4). Therefore in these compromise experimental conditions, the two formulated models are considered acceptably valid as they fit experimental data reliably well with maximum error margin of 1.7%.

4. Conclusions

This study forms part of the efforts to effectively optimize wet ball-milling of UG-2 Platinum ores. Statistical experimental design methods were employed to determine optimum conditions of load volume and slurry percent solids with respect to two response variables: specific energy consumption and size reduction index. Each response variable was adequately described as a function of the operating factors by a second order model. The validity and adequacy of the two models were also assessed using ANOVA tests (F-tests, Student’s t-tests and coefficient of determination–R²) and the tests confirmed achievement of convergence at the optimum experimental conditions.

Response surface plots and contour maps were drawn to show how the responses varied with changes in level settings of the operating factors. They showed that specific energy consumption is more dependent on ball load volume while the reduction index is more or less equally influenced by the ball load volume and slurry solids concentration. Also, the results suggest that better energy efficiency could be achieved at lower level setting of ball load volume and high level setting of slurry % solids whilst higher reduction index could be attainable at a higher level setting of ball load volume and lower level setting of slurry % solids. Therefore, the two operating factors need to be controlled precisely. Within the space of the experimental conditions considered, the factor levels that yield the compromise optimum specific energy consumption (10.54 kW h/ton) and reduction index (3.93) were found to be at ball load volume of 29% and slurry solids concentration of 75%. This was verified using confirmatory experimental tests under these optimum conditions producing specific energy consumption of 10.72 kW h/t and reduction index of 3.91; which clearly shows that the models fit the experimental data reliably well within 5% error margin.

Overall, this study has shown that application of statistically-based methods of experimental design could help to obtain substantial information of the process effectively and reliably (not by guess work or intuition) from only few experimental tests; which presents savings on time and cost. This information would be helpful for optimization of the process conditions.

Acknowledgements

We are grateful to Anglo-Platinum South Africa for the financial support and supply of the feed samples. Additional funding by National Research Foundation (NRF) is also acknowledged. Special thanks are due to Mr. Francois Katubilwa for his help with experimental work and Dr. Geoffrey Simate for providing many useful suggestions which helped to enhance the quality of this paper.

References


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