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Prediction of pressure filtration characteristics of $CaCO_3$ suspensions ground in a vertical stirred media mill

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Abstract

Cake filtration is an important unit operation in the processing of minerals and pigments. The filtration characteristics are strongly dependent on the upstream processing of the ore, such as grinding parameters. This study aims at empirical investigation of the influence of key stirred media milling variables on the pressure filtration characteristics of calcium carbonate. Various regression models are presented and evaluated with respect to their statistical significance. The results show that all the grinding variables have a statistically significant influence on the average specific cake resistance, but the predictability of the models is not very good unless combined and polynomial terms are considered.

1. Introduction

The need for fine particles for various applications has increased recently in many industries, such as ceramics, minerals, pigments, cosmetics, electronics, and pharmaceutical industries (Breitung-Faes and Kwade, 2013; Inam et al., 2011; Choi and Choi, 2003). Grinding with stirred media mills has been proven to be an excellent alternative for the production of fine particles of the preferred size distribution.

In stirred media grinding, several factors have an influence on the obtained fineness of the product. These factors include, but are not limited to 1) the size of the mill, 2) the tip speed of the stirring element, 3) the rheology and solids concentration of the slurry, and 4) the size and density of the grinding beads (Becker et al., 2001; Inam et al., 2011; Garcia et al., 2002; He and Forssberg, 2007; Ohenoja et al., 2013a). High speed of the mill and the use of relatively small beads have been observed to produce narrower particle size distribution (Wang and Forssberg, 2000), but the optimal size of the beads depends on the feed size of the ground material (He and Forssberg, 2007). When the feed consists of fine particles, the grinding performance can be improved by the use of small beads (Ohenoja et al., 2013b), while the grinding of coarse particles is performed more efficiently by using coarse grinding media (Jankovic, 2003).

After the wet grinding stage, mechanical dewatering by cake filtration techniques is often performed in order to remove water from the product. A general trend in the mining industry has been that the flotation concentrates have become finer than they were before, which is mainly due to the increasing complexity and decreasing quality of the available ores (Townsend, 2003). This has increased the need to use great pressure differences to enable efficient separation. Cake filtration processes are affected by a great number of factors, including the solids concentration of the slurry, particle size distribution, particle shape, and surface charge of the particles, as well as the properties of the filter medium (Tarleton and Willmer, 1997; Wakeman, 2007). Due to these factors and the compressibility of the filtered materials, i.e. the increase of the average specific cake resistance with the applied pressure, the cake filtration processes are difficult to predict theoretically. Therefore, empirical modeling of cake filtration is often a more reasonable approach.

In this study, ground calcium carbonate (GCC) is used as the model substance. The aim is to increase awareness of the effect of grinding parameters on the pressure filtration properties of the ground suspensions. Various regression models are utilized as tools in this empirical evaluation.

2. Theory and calculations

The used regression models are introduced below. The theory of constant pressure filtration has been presented in earlier literature for instance by Svarovsky (1981).

The models described in Eqs. (1-4) were used for the prediction of the average specific cake resistance α_{av} (*Y*). In all the models presented below, X_1, X_2, X_3 and X_4 represent the bead size, grinding time, grinding speed and filtration pressure, respectively. The regression coefficients are represented by symbols β_1-β_{14}, while β_0 stands for the Y-intercept.

The simplest linear model used to describe the filtration properties was

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_4 X_4 \quad (1)$$

All possible combined effects were then added to improve the prediction as follows:

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_4 X_4 + \beta_5 X_1 X_2 + \beta_6 X_1 X_3 + \beta_7 X_2 X_3 + \beta_8 X_1 X_4 + \beta_9 X_2 X_4 + \beta_{10} X_3 X_4 \quad (2)$$

When also the quadratic terms were added, the model became

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_4 X_4 + \beta_5 X_1 X_2 + \beta_6 X_1 X_3 + \beta_7 X_2 X_3 + \beta_8 X_1 X_4 + \beta_9 X_2 X_4 + \beta_{10} X_3 X_4 + \beta_{11} X_1^2 + \beta_{12} X_2^2 + \beta_{13} X_3^2 + \beta_{14} X_4^2 \quad (3)$$

Evaluation of the statistical significance revealed that some terms in Eq. (3) were not statistically significant. After the deletion of the unnecessary terms, the model became

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_4 X_4 + \beta_5 X_1 X_2 + \beta_6 X_1 X_3 + \beta_7 X_2 X_3 + \beta_8 X_3 X_4 + \beta_9 X_3^2 \quad (4)$$

3. Materials and methods

A slurry with 20 w-% solids was prepared from ground calcium carbonate powder (Nordkalk Oy, Finland) and water. The density and the D_{10}, D_{50}, and D_{90} particle sizes of the solids were 2700 kg m^{-3}, 4.0 µm, 29 µm, and 83 µm, respectively. Malvern Mastersizer 3000 laser diffraction particle size analyzer was used for measuring the particle sizes of the original and ground samples.

A stirred media mill with an inner diameter of 152 mm and a height of 300 mm was used for grinding the calcium carbonate suspension. A three-level full-factorial experimental design was applied. The ground batches of slurry were poured in a baffled 3 dm^3 mixing tank. A slurry sample of 250 g was measured for each filtration test. The filter unit was a Nutsche pressure filter operating at pressures of 2, 4, and 6 bar. Homogeneous discs, made of cellulose, were used as the filter media. All experiments were conducted at room temperature (22 °C). The experimental details are presented in a previous paper by the authors (Kinnarinen et al., 2015).

The use of dimensionless values for the regression modeling enables comparison of the variables with relation to the direction and the strength of how the variables affect the output of the model. Therefore, each variable was converted to values ranging from -1 (minimum) to 1 (maximum). The coded values of the variables for regression models containing combined and quadratic terms are listed in the online supporting material (Appendix 1).

4. Results and discussion

4.1. Dependence of α_{av} on particle size distribution

The dependency of the average specific cake resistance α_{av} on the grinding result with the same material has been investigated previously by the authors (Kinnarinen et al., 2015), where it was observed that the fine end of the particle size distribution has the best correlation with α_{av}, and the correlation becomes weaker as the large end of particle size distribution is used for describing the size of particles. The great importance of the fine end of distribution in dewatering operations has been reported in the previous literature for instance by Mwale et al. (2005). The moisture content of the cakes increased from less than 20 w-% to about 30 w-% when α_{av} increased from $1 \cdot 10^{11}$ to $4 \cdot 10^{11}$ m/kg.

4.2. Regression modeling of α_{av}

The average specific cake resistance is an important measure of filterability and is therefore widely used as a main sizing parameter of industrial filters. The pore structure of the filter cake has a great influence on α_{av}, which means that α_{av} is affected by a great number of factors, such as the type of slurry and the properties of its solids, as well as the mechanism and rate of cake formation and growth (Dong et al., 2009; Iritani et al., 2012; Ni et al., 2006; Mahdi and Holdich, 2013). In practical filtration applications, the filtration pressure has a notable influence on α_{av}, because practically all mineral concentrates form more or less compressible cakes. The main results of the regression modeling with Eqs. (1-4) are presented in Table 1.

Table 1. Coefficients β_i of the regression models defined in Eqs. (1-4).

	Modeling of α_{av}: Coefficient β_i x 10^{10} (m kg^{-1})										
Eq. (#)	B_0	β_1	β_2	β_3	β_4	β_5	β_6	β_7	β_8	β_9	β_{10} - β_{14}
(1)	17.7	-3.33	6.65	8.74	2.21	-	-	-	-	-	-
(2)	17.7	-3.33	6.65	8.74	2.21	-2.13	-1.47	3.00	-0.49	0.69	1.05
(3)	17.5	-3.33	6.65	8.74	2.21	-2.13	-1.47	3.00	-0.49	0.69	1.05; -1.05; -0.56; 2.46; -0.44
(4)	16.5	-3.33	6.65	8.74	2.21	-2.13	-1.47	3.00	1.05	2.13	-

The information shown in Table 1 was utilized to evaluate which variables deserved to be included in the graphical illustration of the results (Fig. 1). It can be seen in Table 1 that the highest coefficients in Eqs. (1) and (4) are β_2 and β_3, which are the coefficients for grinding time and speed.

The linear regression models for the dependence of the average specific cake resistance α_{av} on the grinding speed and grinding time are visualized in Fig. 1 a-c, where the dimensionless values -1, 0 and 1 represent the minimum, midpoint and maximum values. As Fig. 1a shows, the agreement between the model and the measurement results is the best when the bead size (X_1) and the filtration pressure (X_4) are at the minimum. The inability of the model to predict the filterability at the maximum values of variables X_2 and X_3 becomes more apparent when the values of variables X_1 and X_4 are also increased (Fig. 1 b-c). The use of the nonlinear, statistically revised model (Eq. 4) is illustrated in Fig. 1 d-f. Comparison between Figs. 1 a-c and 1 d-f shows that the agreement between the measured and predicted values is improved by the inclusion of the statistically significant combined and quadratic terms. However, the model tends to overestimate α_{av} slightly when both X_2 and X_3 are at the maximum, and X_1 and X_4 are either high or low.

The measured and modeled values of α_{av}, according to Eqs. (1-4), are presented in Fig. 2 a-d. The first step to improve the basic case of the linear model (Fig. 2a), i.e. insertion of the combined effects of the variables, is presented in Fig. 2b. Based on Fig. 2b, the variables seem to have some important interactions, and taking them into account decreases the concavity of the graph to a great extent. The correlation coefficient is also improved greatly, to 0.91. Evaluation of the combination effect by using the information in Table 1 reveals that the interaction of the grinding time (X_2) and the grinding speed (X_3) have the most considerable influence on the increase of α_{av}. The combination of the bead size (X_1) and the grinding time (X_2), as well as the combination of the bead size (X_1) and the grinding speed (X_3), reduce α_{av}. However, these effects are not as strong as those caused by the combination of the grinding time and speed.

Further improvement of the model was performed by the addition of quadratic terms of the variables (Fig. 2c). As a result, the correlation coefficient was increased to 0.93. The main problem with this model (Eq. (3)) is that the statistical significance of some terms, evaluated by using the p-values, is poor. Omitting those insignificant terms in the modeling according to Eq. (4) resulted in a statistically improved model, the performance of which is illustrated in Fig. 2d. In spite of the improvement in the statistical significance, the correlation coefficient was not improved, which leads to the conclusion that various random effects have not had a very strong influence on the modeling in the case of Eq. (3).

5. Conclusions

The regression models indicated that there was a clear correlation between the grinding variables and the filtration properties of the slurry. According to the p-values obtained for the grinding variables, it was apparent that all the three variables had an impact on α_{av} at very high statistical significance ($p < 0.01$). Comparison between the measured and modeled values of α_{av} showed that the model had a slight tendency to underestimate the highest and lowest values of the average specific cake resistance. When the combined effects of the variables and also quadratic terms were included in the model, the prediction was improved and the correlation coefficient was increased from 0.84 to 0.93.

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