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Quantifying the effect of the grinding aids in a batch stirred mill by a modelling approach

Modelleme yaklaşımıyla kesikli karıştırmalı bir değirmende öğütme yardımcılarının etkisinin ölçülmesi

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A B S T R A C T

Grinding aids are commonly used in dry grinding to disperse the material in the system effectively and to improve grinding performance. Most of the time, different grinding aids with varying dosages are tested in laboratory conditions by trial and error. A simple methodology is required to compare the results of different grinding aids and dosages in batch milling. In this study, the utilization of the modelling approach in comparing the effect of grinding aid types and dosages were aimed. In order to achieve this, an experimental program has been conducted using a laboratory-scale batch stirred ball mill. The empirical model developed using the Farazdaghi-Harris model offers a new approach to determine the effect of grinding aids on grinding performance.

Keywords: Stirred ball mill, Fine grinding, Grinding aids, Modelling, Farazdaghi-Harris model

Introduction

Efficient fine grinding becomes increasingly essential due to the increasing industrial demand for finely ground products. This demand has led to the development of the new fine grinding mills, i.e., stirred ball mills (Valery and Jankoviç, 2002). Stirred ball mills provide higher energy efficiency compared to conventional ball mills in fine and ultrafine grinding operations due to their operational characteristics and particle breakage mechanisms. The energy efficiency of the stirred mills is mainly ensured with the use of smaller grinding media that is mixed at high stirrer speeds and operated at high filling ratios. It can also produce fine-grained products with narrow particle size distributions after the grinding process by stirred mills (Gao and Forssberg, 1995, Zheng et al., 1996, Kwade and Schwedes, 1997, Kwade, 1999). In recent years, stirred ball mills are widely used for fine and ultrafine grinding processing in the ceramics, chemical, food, cement, paint, pharmaceutical, plastics and cosmetic industries (Jankovic et al., 2004, Altun et al., 2013, Ouattara and Frances, 2014, Toprak et al., 2014, Gokcen et al., 2015). Demand for stirred ball mills is increasing because of their ease of use, simple construction, high size reduction ratio, low energy consumption compared to other fine grinding machines and its suitability for modelling studies (Gao and Forssberg, 1995, Zheng et al., 1996).

Various mathematical models have been developed, aiming to identify events occurring in the mill to assist in the design and optimization of the ball mill operations. The developed models also aim to describe the events occurring in the stirred ball mills. These mathematical models can be used to estimate product size, mill performance, and process simulation as well (Ferrara et al., 1987, Gao and Forssberg, 1995).

Empirical modelling is one of the most widely used analytical methods to produce practical solutions in engineering and industrial studies and is extremely rich in terms of problem-solving

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potential. The empirical approach combines measurable process variables, energy use and product size so that it can be used to estimate a single size such as p_{80}, 50 etc. or size distribution of a mill discharge (Mannheim, 2007). Empirical models are generally developed using experimental data for a specific mill application.

The empirical model developed in this study presents a new approach for modelling the vertical stirred ball mill by employing the Farazdaghi-Harris model to determine the effect of grinding aids on grinding performance.

Grinding aids (GAs) are generally organic compounds added to the mill during the grinding, and their principal purposes are to reduce the energy consumed in grinding and increase the efficiency of the mill. During the grinding process, fine particles produced may agglomerate. The particles that agglomerated reduce the fluidity of the material to be ground. Furthermore, the cushioning effect caused by the coating of the grinding medium and the liner with fine particles reduces the dry grinding efficiency (Wang and Forssberg, 1995). Grinding aids commonly used in dry milling significantly increase milling efficiency by preventing particle agglomeration, effectively dispersing particles, and improving material fluidity (Paramasivam and Vedaraman, 1992, Fuerstenau, 1995, Altun et al., 2015, Prziwara et al., 2018, Toprak et al., 2018). In addition, they also increase grain liberation by promoting breakage along grain boundaries. They are added to the mill during the grinding in proportions calculated depending on the material weight to reduce particle agglomeration. Grinding aids promote crack propagation when they enter the existing crack in the particle, causing the particle to break more easily (Choi et al., 2009). GAs are generally organic compounds that contain glycols and amines. They are categorized into three groups based on their structure: aliphatic amines-base, glycols-base and phenol-base. The high polarity of their chemical functioning groups reduces the surface energy forces, which cause agglomeration of the newly produced particles (Jeknavorian et al., 1998, Hashem et al., 2019)

Most of the time, different grinding aids with varying dosages are tested in laboratory conditions by trial and error. A simple methodology is required to compare the results of different grinding aids and dosages in batch milling. In this study, the utilization of the modelling approach in comparing the effect of grinding aid types and dosages was aimed. For this purpose, an experimental program was carried out using a stirred ball mill.

1. Material and methods

1.1. Material

Potassium feldspar, which was used as the test material in grinding experiments, was supplied by Kütahya Porcelain Company. After density analyses, the mean density of the test material was calculated as 2.65 g/cm^3. The particle size distributions of the feed and ground products were characterized using Malvern Mastersizer 2000 Particle Size Analyzer. The average values of three measurements were used for evaluating the experimental results.

1.2. Method

The HD-01 model laboratory scale vertical stirred ball mill, used in both dry and wet milling modes, was developed by Union Process (USA) (Figure 1). This model ball mill is designed for using at stirring speeds ranging from 100 to 600 rpm. The HD-01 model ball mills use alumina balls in diameters ranging from 3 to 6 mm as grinding media. The typical feed size is less than 100 microns. The HD-01 series mills have water-cooled jackets for cooling (or heating) the grinding tank. The grinding system has a data acquisition software developed by the manufacturer company. The software monitors the parameters used in the experiments and records the observed experimental data in an Excel worksheet for future references.

Figure 1. *The Model HD-01 stirred ball mill used in the experiments*

The grinding system consists of a grinding chamber and a centrally positioned rotating stirrer. A constant gap of 6.35 mm was left between the bottom end of the centrally positioned stirrer and the bottom of the tank. The material to be ground is placed in the tank with the grinding media and then is stirred by the shaft rotating at high speed. When the stirrer starts to rotate, the experimental parameters begin to be recorded with online data acquisition software. The high-speed stirring process causes the grinding media to exert both shearing and impact forces on the material. The ultimate result of the grinding process is an extremely fine-grained product with narrow particle size distributions, measured in micron or micron fractions. It is also possible to add chemicals or additives to the mill at any time during milling or to take samples from the mill.

Table 1 shows the technical specifications of the stirred ball mill used in the grinding experiments.

Table 1. *Technical data of the stirring ball mill*

Mill Diameter, mm	80
Mill length, mm	120
The volume of the mill, cm^3	592
The total weight of the ball, g	997.4
The diameters of alumina balls used as grinding media, mm	3 and 5
The fraction of mill volume filled by the ball bed at rest (J)	0.72
The fraction of mill volume filled by powder bed (f_c)	0.216
The fraction of the spaces between the balls at rest which is filled with powder (U)	0.75
Sample weight, g	203.3

2. Results and discussions

2.1. Grinding tests

The tests were initially conducted in conditions where no grinding aids were used. As a result of breaking the ionic bonds of the material due to a mechanical process, quite reactive positive and negative charges are created on the newly broken surfaces (Assaad et al., 2009). These charges enable the particles to agglomerate. As a result, these particles adhere to the mill surface and balls more easily. Following that, the tests were carried out at various dosages with two different types of grinding aids. Grinding aids can be classified into three types based on their structure: aliphatic amine-based, glycols-based and phenol-based (Hashem et al., 2019) The amine group Triethanolamine (TEA) and glycol group Ethylene glycol (EG) are used in this study (as given in Table 2).

Ethylene glycol is absorbed by the particles and the mill surface via hydroxyl groups, which neutralizes this electrostatic surface charge. Furthermore, the alkyl part of EG shields the surface charge of the particles, reducing adhesive forces and preventing powder aggregation and coating. This effect increases the mill's ability to produce finer particles. The increase in grinding index values caused by increasing EG dosage could be attributed to the solid surface's monolayer coverage. Unlike ethylene glycol, triethanolamine causes the formation of multimolecular layers on rigid surfaces, chiefly when used in high doses. The absence of any change in the grinding index values indicates the presence of this formation. Grinding aids, which tend to form multimolecular layers, should be used with caution as they can create capillary forces that favor agglomeration (Hashem et al., 2019).

The kinetic test program was implemented to observe the effect of grinding aids at different grinding times. Therefore, at each grinding aid dosages, the grinding was carried out for 30, 60, 90, and 120 minutes.

Table 2. *Test conditions at different grinding aid dosages*

Dosage (g/t)	TEA	EG
0	x	
1000	x	x
2000	x	x
4000	x	x

The size distributions of ground products with TEA and EG are presented in Figures 2 and 3, respectively. Size distribution data were utilized in the mathematical modelling of the mill.

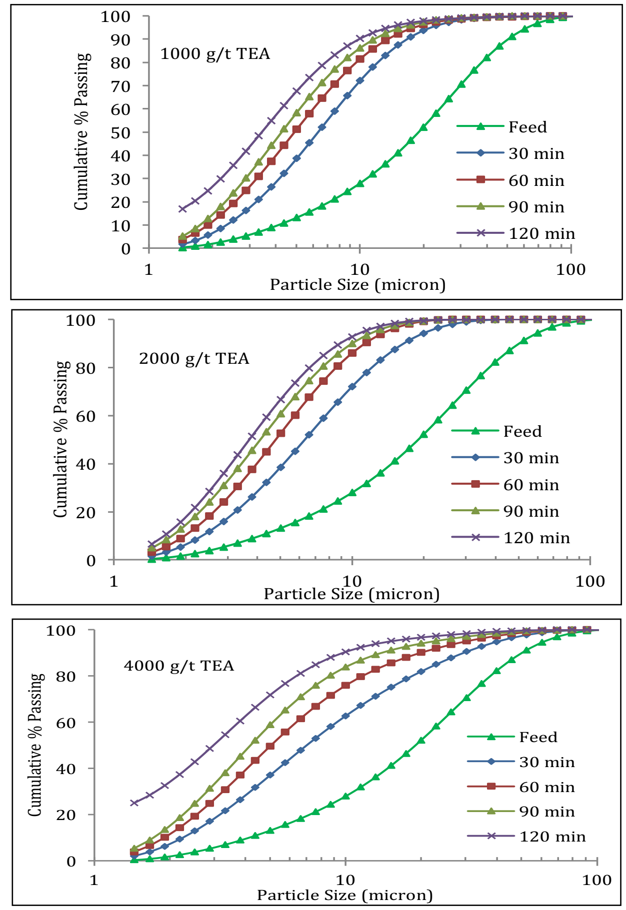


Figure 2. *Particle size distribution data with TEA grinding*

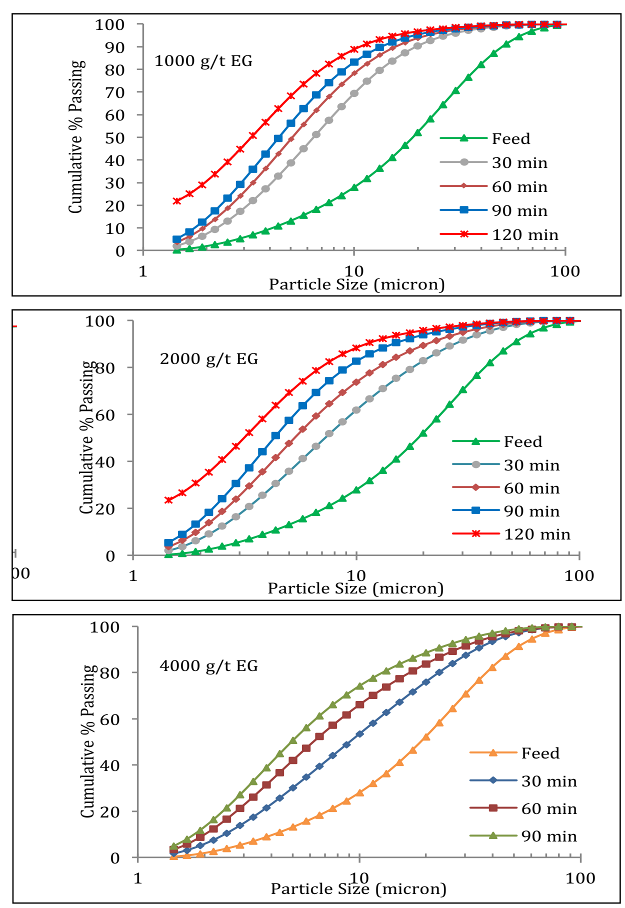


Figure 3. *Particle size distribution data with EG grinding*

2.2. Mathematical Modelling of Batch Stirred Milling

The size reduction in a comminution system is achieved by breaking the particles into smaller fragments. Reduction ratio in each size class is given by (Pi/Fi) and defined as the cumulative disappearance rate factor (Size vs ln(Pi/Fi)) and this behaviour can be mathematically expressed which can be used for modelling. The data obtained from the batch grinding tests performed without using grinding aids are used for modelling purposes. In order to figure out the disappearance rate of particles in each grinding test, the cumulative ratio change of the feed and product for each size fraction was sketched by size (Figure 4). Especially at coarser size ranges, the ratio was almost indicated similar behaviour for varying grinding times. The major difference was in the fine particle sizes as it could be expected.

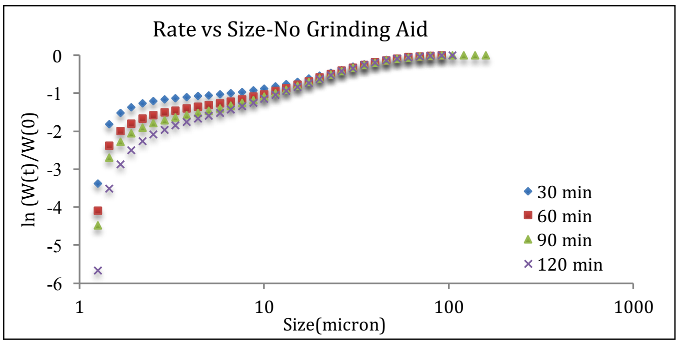


Figure 4. *Particle disappearance rate when no grinding aid was used*

The mathematical model of the batch grinding process is defined by deriving the mathematical expression for particle size versus the cumulative disappearance rate factor $\ln(W_i(t)/W_i(0))$. Farazdaghi-Harris approach (Eq.1) gave the best mathematical expression in terms of the data fitting (Farazdaghi and Harris, 1968).

$$y(x) = \sum_{i=0}^{N-1} \frac{1}{a_i + b_i x^{c_i}} \tag{1}$$

By using nonlinear Marquardt-Levenberg optimization technique or algorithm, the model parameters could be back-calculated (Marquardt, 1963). The model parameters *a, b* and *c* are constants, which reflect the grinding conditions and material properties. Specifically, b is related to the mill operating parameters such as ball size, tip speed and stirrer type and c is related to material properties. Parameter a is reflecting the mill rheology which is the grinding time (depending on fines generation), grinding aid type and dosage.

Model fitting studies have been conducted for each grinding time. As can be seen from the graphs given in Figure 5, there is a good agreement between the fitted and experimental results indicating that model structure represents the behaviour accurately.

For each data series, as mill operating conditions and the material are kept constant the calculated values of the Parameters b and c as constant numbers are -8.07E-04 and 2.362, respectively. The effect of grinding time is reflected in the Parameter *a*, which is presented in Figure 6. The change of Parameter a by grinding time indicates that as the grinding time increases, the increase in Parameter a reduces significantly. This behaviour is interpreted as the slowing down effect of grinding by time. As a result, the kinetic behaviour of the grinding action can be reflected on a single parameter while the other parameters are kept constant. These parameters are thought to be the controlling parameters for the grinding system and material properties. As the tests are conducted at the same system with the same material, no change has been observed on the related parameters.

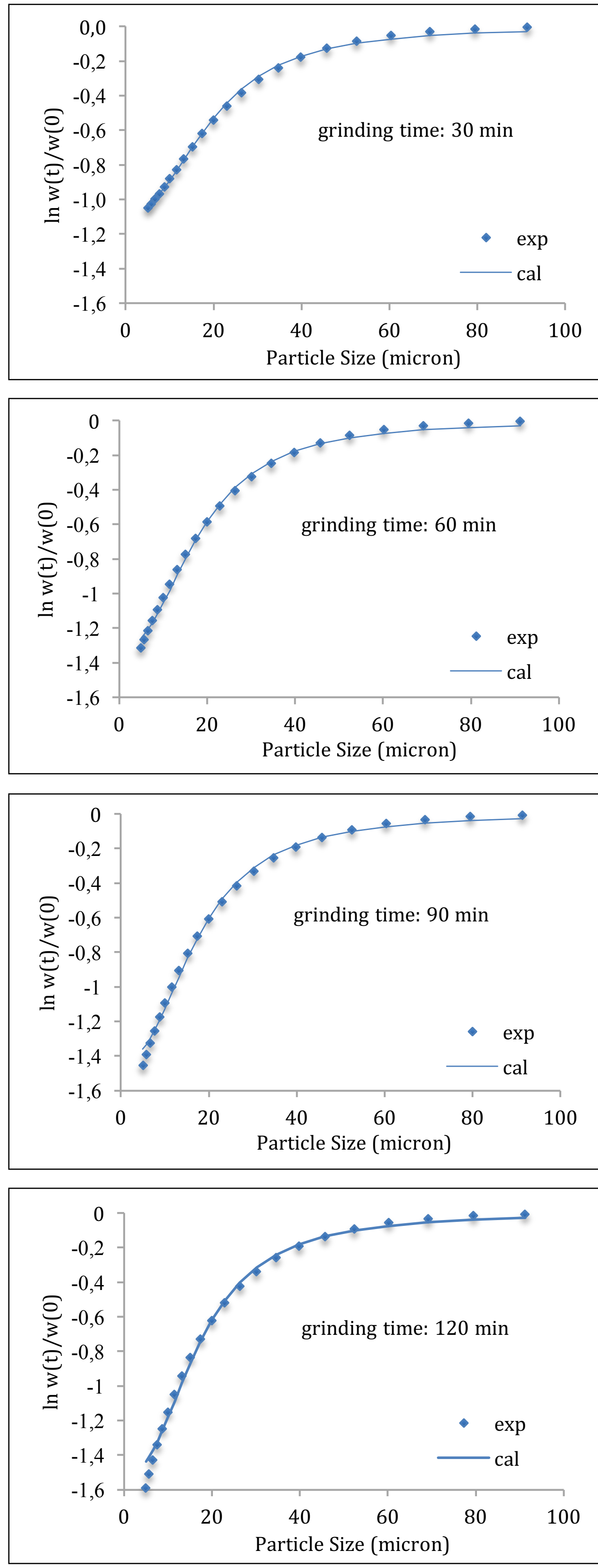


Figure 5. *Graphical representation of the fitted and experimental results for each grinding time*

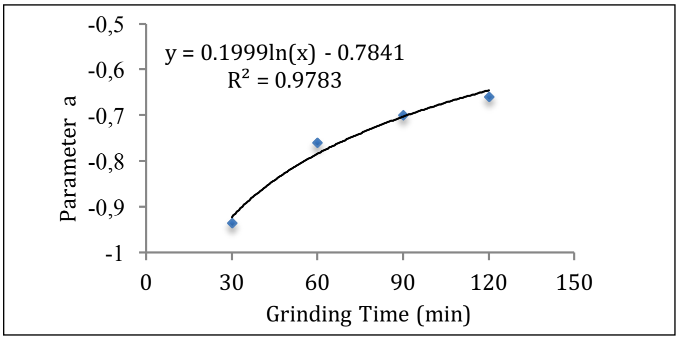


Figure 6. *Variation of "Parameter a" by time*

2.3. Comparing the grinding aid types and dosages during batch stirred milling

The structure of the model presented above can be used to provide a quantitative comparison of grinding aid types and dosages. The test results of TEA (Triethanolamine) and EG (ethylene glycol) have been analysed with the suggested model structure in order to prove this hypothesis.

The disappearance rate of material with varying dosages of TEA and EG at different grinding times are presented in Figure 7 and 8, respectively.

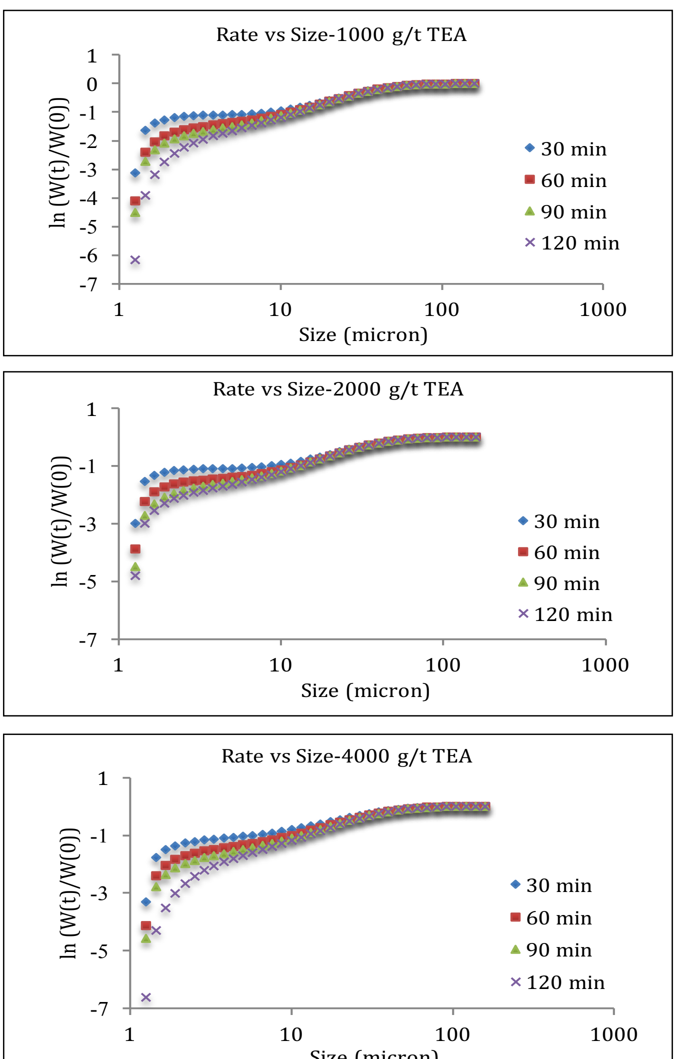


Figure 7. *Disappearance rate of material with TEA at varying dosages*

As can be seen from the graphs, a similar trend could be followed for varying grinding times, grinding aid types and dosages. In this experimental study the findings indicated that below 10 micron size the disappearance rate was increased. It is known that during grinding the ground particles may agglomerate, for that reason the dispersion effect of grinding aids on material is effective at very fine size ranges. Normally in a continuous milling operation this can be reflected on the discharge rate of the mill. But in a batch milling environment there is no transport from the mill. Therefore, at fixed milling operation conditions (ball size, ball load, tip speed) the dispersed fine particles can be ground more effectively. At shorter grinding times (in a batch grinding condition it is typically below 30 minutes), the higher the aid dosage, the more effective the grinding is. As the grinding times increase the fines generation increases but the increased grinding aid dosages does not improve the grinding efficiency. It can also be pointed out from the graphs for extended grinding time that the effect of grinding aid dosages on the grinding performance became stable.

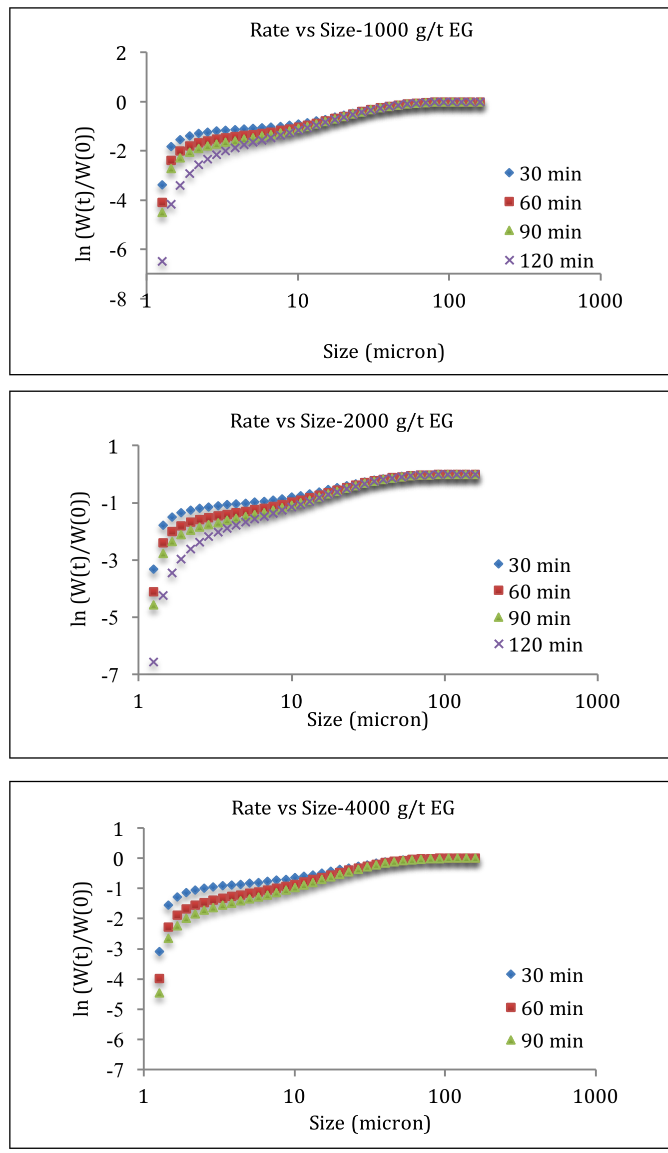
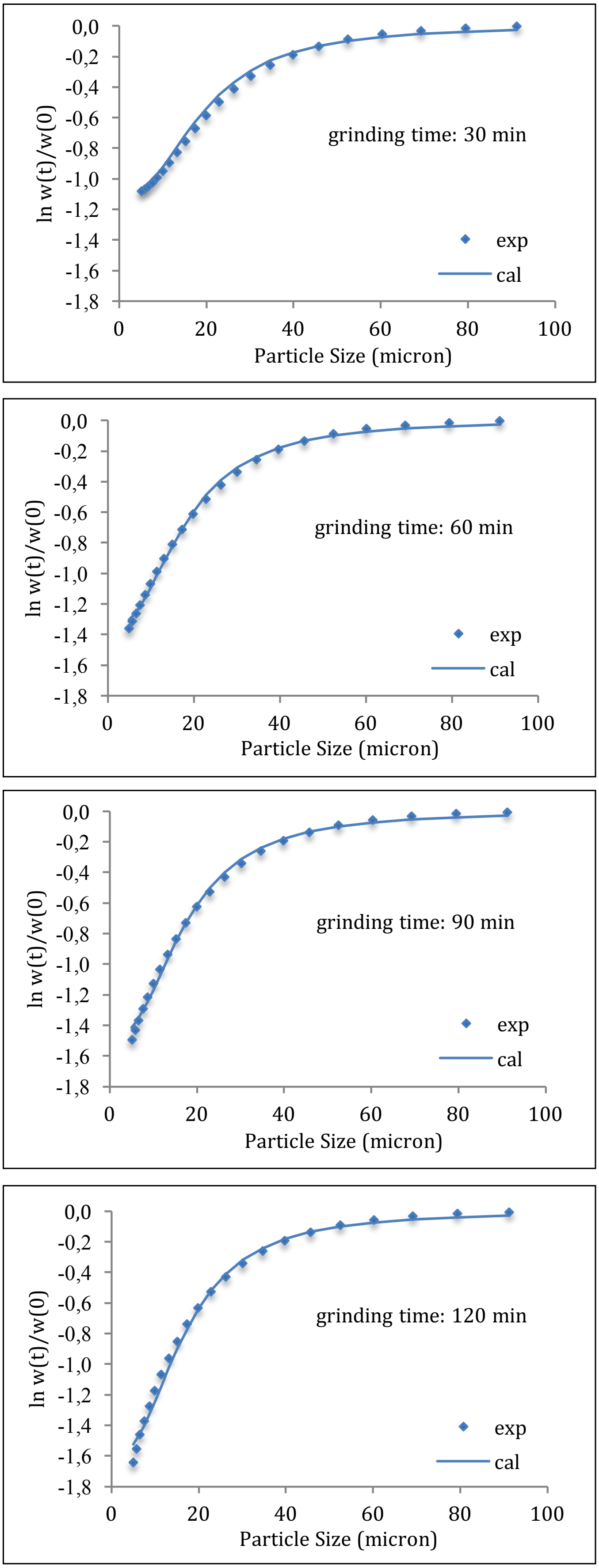
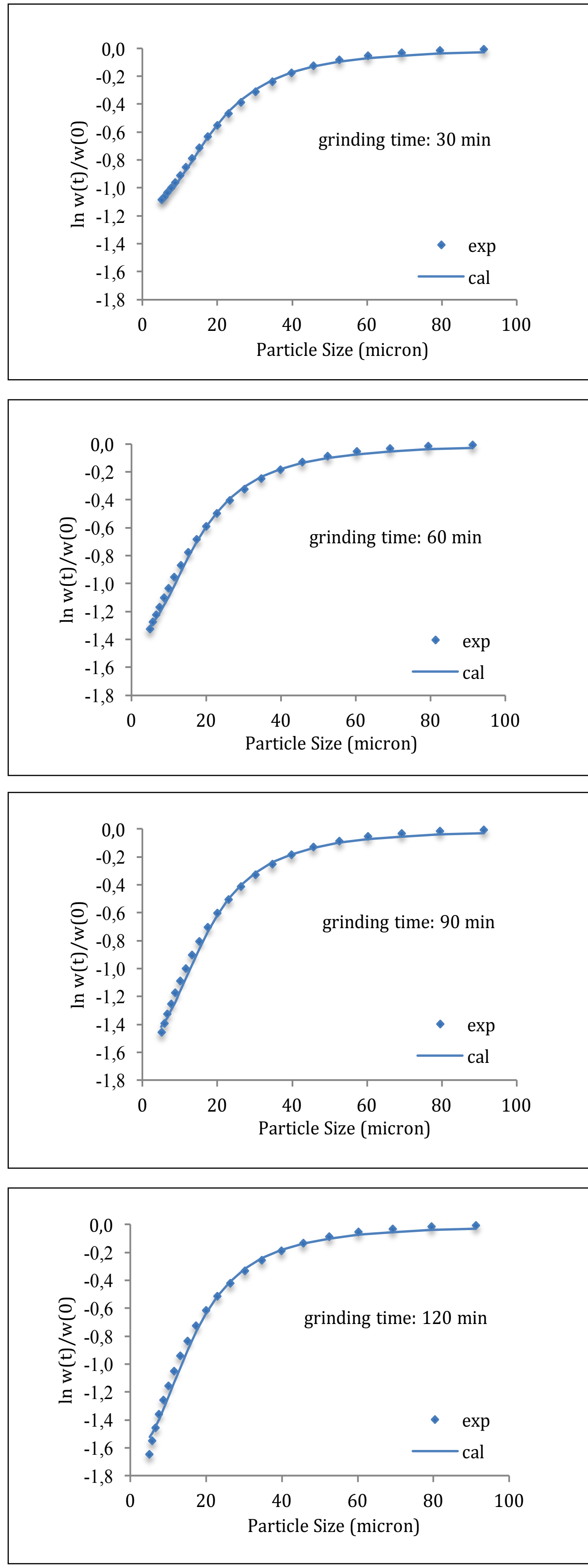


Figure 8. *Disappearance rate of material with EG at varying dosages*

The same model structure has been used in modelling studies for each grinding aid dosage. Model fitting studies addressed high accuracy in predictions as given in Figure 9. In order to simplify the illustrations only two sets of data from TEA and EG are presented as an example. In other test condition similar graphs have been obtained.



a: Results with TEA at 1000 ppm



b: Results with EG at 1000 ppm

Figure 9. *Deviation of fitted data from experimental data set (a: TEA, b: EG*

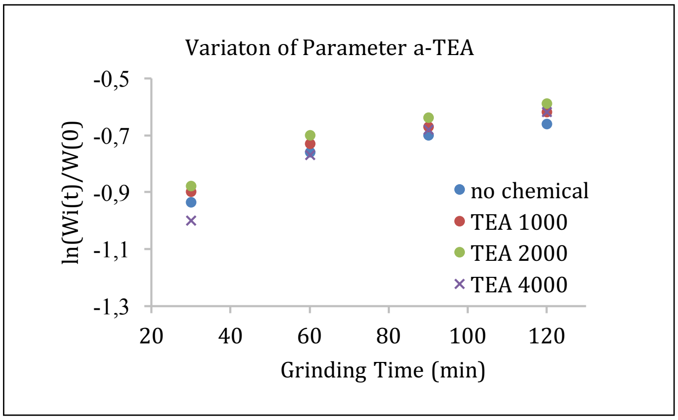


Figure 10. *Variation of Parameter a with TEA dosage*

During the calculations, as explained above the effect of different grinding aids and dosages could be reflected on the Parameter a. The effects of operational conditions on the model Parameter a were examined at different grinding times for TEA and EG at varying dosages, and the results are illustrated in Figures 10 and 11, respectively.

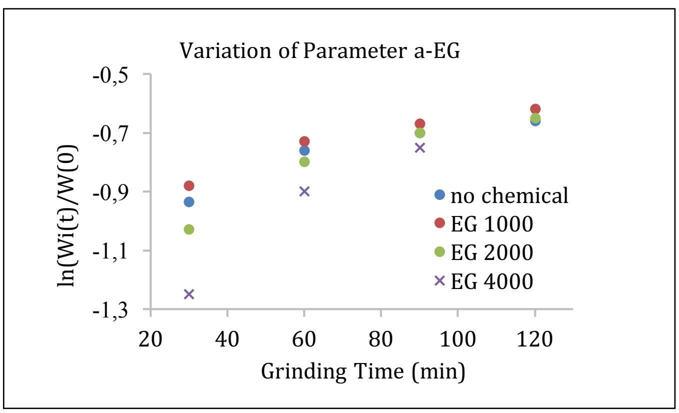


Figure 11. *Variation of Parameter a with EG dosage*

As can be followed from the graphs in Figures 10 and 11, the grinding rate seems to be slowing down as expected at increased grinding times. This can be explained as a cushioning effect or inefficient grinding in the existing conditions. In order to address the efficiency of grinding aid types, the comparison is given for different grinding aids at the very shortest and longest grind time of the tests. The data address that both TEA and EG are effective on grinding performance of potassium feldspar while the EG has created an adverse effect especially at higher dosages. The performance improvement of the grinding operation is quantified by calculating the A parameter. In this specific case, at higher dosages measured for TEA, the Parameter a varies 15 % compared to without grinding aid condition. A similar trend could be obtained for EG use as well. But as the grinding aid dosage reduces the improvement on grinding efficiency as reflected on Parameter a is limited to 8 % change (as a comparison to no grinding aid condition). However, high dosage use of EG indicates performance loss compared to the conditions without using grinding aids. This behaviour points out the critical concentration of grinding aids in the system and overdosing may end up in performance losses. This is related to the re-agglomeration behaviour of dispersed particles. Therefore, for each material type, grinding aid should be appropriately designed, and the dosage use has to be optimised by quantified test data. According to the test results, the modelling approach is useful to achieve this quantification process.

Conclusions

Batch vertical stirred mill can be modelled by relying on the rate disappearance factor by size. The Farazdaghi-Harris equation is well suited to express this situation mathematically.

The model parameters of the equation are thought to be representing the grinding conditions, the design of the system, and material properties. As the tests are conducted at the same system with the same material, no change has been observed on the related parameters. This allows reflecting the grind time and the effect of grinding aids on a single parameter (Parameter a).

Additionally, the grinding aid type is important for each material used. This phenomenon has been reported in many studies suggesting that GAs is solid-specific although there is no correlation of global surface chemical properties (such as functional groups, molar masses) with their effectiveness. From rheology studies, the observations can be attributed to the varying degree of flow properties such as flow index, bulk density, internal friction factor and shearing cohesion (Chipakwe et al., 2020). The specific formulation has to be implemented, and for each aid, an optimization study should be run for determining the best dosage conditions. Specifically, for comparative studies, the effect of the grinding aids and dosage can be quantified by calculating Parameter a by using the recommended model.

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