# ANALYSIS OF FACTORS AFFECTING THE GRINDABILITY OF K-FELDSPAR IN STIRRED BALL MILL BY FULL FACTORIAL DESIGN

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**Keywords**
*K-feldspar*
*Stirred ball mill*
*Narrow grain size*
*Full factorial design*

**Abstract**
*This study's aim is to investigate the wet and dry grindability of K-feldspar in the stirred ball mill. In the evaluation of the grinding process, a two-level and four-factor factorial design was applied and the effects of variables such as ball size, stirrer speed, grinding time, and interstitial filling ratio on the particle size distribution were investigated. Experiment results showed that K-feldspar can be efficiently milled using a stirred ball mill in a narrow particle size range (1-10 microns).*

# KARIŞTIRMALI BİLYALI DEĞİRMENDE K-FELDSPATIN ÖĞÜTÜLEBİLİRLİĞİNİ ETKİLEYEN FAKTÖRLERİN TAM FAKTÖRİYEL TASARIMLA ANALİZİ

**Anahtar Kelimeler**
*K-feldspat*
*Karıştırmalı bilyalı değirmen*
*Dar tane boyutu*
*Tam faktöriyel tasarım*

**Öz**
*Bu çalışmanın amacı, karıştırmalı bilyalı değirmende K-feldspatın yaş ve kuru öğütülebilirliğini araştırmaktır. Öğütme işleminin değerlendirilmesinde iki seviyeli ve dört faktörlü faktöriyel tasarım uygulanmış ve bilye boyutu, karıştırıcı hızı, öğütme süresi ve boşluk doldurma oranı gibi değişkenlerin tane boyutu dağılımına etkileri araştırılmıştır. Deney sonuçları, K-feldspatın, karıştırmalı bilyalı değirmen ile dar bir tane boyutu aralığında (1-10 mikron) verimli bir şekilde öğütülebileceğini göstermiştir.*

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|---|---|---|---|
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## 1. Introduction

K-feldspar is a basic raw material used in the porcelain body to form the melter (-75 microns) and glassy phase (-10 microns). It reduces melting temperatures by reacting with raw materials such as kaolin and quartz (Burleson, 2003). As a result, the firing time and temperature of porcelain are reduced. In porcelain production, feldspar and quartz are generally used by wet grinding down below 75 microns with conventional ball mills. One of the most important reasons for grinding potassium feldspar to this size is to enhance its fluxing capability by increasing its surface area. As a result, the fluxing capacity of the material will also increase by grinding K-feldspar into micro-fine sizes (-10 microns) in stirred ball mill.

Stirred ball mills are more useful in grinding below 75 microns than conventional ball mills because of their easy operation, simple design, low energy consumption, and high grinding rate (Bernhardt, Reinsch, and Husemann, 1999; Choi, Lee, Lee, Chung, and Choi, 2007; Dowdle, 1994; Goodson, Larson, and Sheehan, 1985; Just and Yang, 1997; Padden and Reed, 1993; Szegvari and Yang, 1995). Due to the use of small grinding media with high-stress densities, the amount of energy produced per unit time and volume in agitated ball mills is quite high, resulting in an effective size reduction of

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the products. As a result, stirred ball mills have become increasingly popular in industries such as mining, ceramics and paint in recent years for micro-fine and ultra-fine (-1 micron) grinding (Becker, 1987; Ma, Hu, Zhang, and Pan, 1998; Reed, 1995; Szegvari, 1994; Szegvari and Yang, 1989).

These mills use a shaft with pins or discs to stir the grinding media. In addition to wet and dry grinding, the stirred ball mills can be used vertical or horizontal oriented. Depending on the dimensions in the feed size distribution, the grinding media diameters can also change from a few millimeters to several microns. Depending on the needs of the industry, grinding media that are made of ceramic, carbon steel, stainless steel, and alumina can be used.

Mill efficiency can be improved by adjusting the variable levels and providing proper grinding conditions. Therefore, it is crucial to understand and control the grinding process. However, testing all possible parameters at every level is time-consuming. Hence, a well-structured experimental design is needed to get results in a shorter time. Today, the full factorial designs are used successfully in many fields to evaluate the process and plan experiments correctly (Dehghan, Noaparast, Kolahdoozan, and Mousavi, 2008; Martínez-L and Ortiz, 2003). The full factorial designs contain combinations of all independent variables used in the evaluation. The independent variables and interactions that influence the dependent variable should be investigated thoroughly in this case.

In the context of the study, a two-level and four-factor factorial design was applied and the effects of variables such as ball size, stirrer speed, grinding time, and interstitial filling ratio on the particle size distribution were investigated. The variables influencing the wet and dry grindability of K-feldspar in the stirred ball mill were also analyzed.

## 2. Experimental

### 2.1. Equipment

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 use at stirrer 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 usually smaller than 100 microns. In this case, milled material size is most likely to be between 1 and 10 microns. The HD-01 series mills have water-cooled jackets for cooling (or heating) the grinding tank. The grinding system has 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.

The grinding system consists of a grinding tank and a rotating shaft in the center. A constant 6.35 mm gap is left between the lower tip of the shaft and the tank base. The high-speed rotating shaft stirs the material in the tank with the grinding media. 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.


Figure 1. The Model HD-01 Stirred Ball Mill

### 2.2. Material and Method

The -75 micron-sized K-feldspar sample used as the feeding material in the experiments was provided by Kütahya Porcelain Inc. A pycnometer was used to determine the density of the sample and its average density was calculated as 2.65 g/cm$^3$ following five measurements.

Two different sizes of alumina balls were used in the grinding experiments. Ball loading into the mill was determined as 40% of the mill tank's effective volume. The necessary calculations for the mill charge were calculated using Equations 1, 2, and 3 (Austin, Klimpel, and Luckie, 1984; Celik, 1988; Ipek, Ucbas, and Hosten, 2005). Table 1 lists the experimental conditions.

$$J=\left(\frac{\text{Ball mass/Ball density}}{\text{Mill volume}}\right)\cdot\left(\frac{1}{0.6}\right) \quad (1)$$

$$f_c = \left(\frac{\text{Powder mass}/\text{Powder density}}{\text{Mill volume}}\right)\cdot\left(\frac{1}{0.6}\right) \tag{2}$$

$$U = \frac{\text{Fractional powder filling}}{0.4\cdot\text{Fractional ball filling}} \tag{3}$$

where $J$ is *fractional ball filling*, $f_c$ is *fractional powder filling* and $U$ is interstitial filling ratio.

Table 1

Stirred Mill Characteristics and Experimental Settings

| | | |
|---|---|---|
| Tank diameter, cm | | 8 |
| Tank length, cm | | 12 |
| Ball total mass, g | | 552 |
| Ball density, g/cm$^3$ | | 3.9 |
| The fractional ball filling | | 0.22 |
| Sample mass, g | for U=0.75 | 112.5 |
| | for U=1.00 | 150 |
| Slurry concentration (weight), % | | 70 |

After each experiment, all the material taken from the mill was sieved, and the ground sample and grinding media were separated from each other. The particle size distributions of the sample were measured by The Malvern Mastersizer 2000 Particle Size Analyzer.

Experiments were performed by $2^4$ full factorial designs. The main effects and interactions were calculated using the Yates algorithm. Results are presented according to this methodology.

If the main effects and interactions of the variables to be selected on a dependent variable are to be examined, the full factorial design can be used. The main effect is defined as the effects of changes in the levels of an independent variable on a dependent variable. In such designs, factors are changed together and all possible combinations of the level of the factors are investigated (Dowdle, 1994).

As the factors number (k) in the full factorial designs increase, the number of effects that can be estimated also increases. For instance, $2^4$ full factorial designs have 16 factor-level combinations. There are four main effects in a 16-experiment, six two-factor interactions, four three-factor interactions, and one four-factor interaction. In most cases, the principle of the sparsity of effects applies. The main effects and low-order interactions typically dominate the system. Interactions with three or more factors are usually negligible. When the k value is greater than or equal to 4, experiments can be performed one-repeat, and it is common practice to use higher-order interactions in estimating experimental error (Mansouri, Khonsari, Holgerson, and Aung, 2002; Montgomery, 2009; Tamhane, 2009).

Particle size (-10 microns) was chosen as a dependent (response) variable in the factorial design. Experiments shown in Yates notation pattern were performed in random order. Table 2 shows the independent variables and their levels used in the experiments. In the design stages, the high-level was denoted by a '+' while the low-level was denoted by a '-.'The authors declare that research and publication ethics were followed in this study.

Table 2

The Independent Variables and Levels Used in The Design

| | Levels | |
|---|---|---|
| Variables | ( - ) | ( + ) |
| Ball size, mm | 3 | 5 |
| Stirrer speed, rpm | 300 | 600 |
| Grinding time, minute | 30 | 150 |
| Interstitial filling ratio,% | 0.75 | 1 |

### 3. Results and Discussion

This study investigated the wet and dry grindability of K-Feldspar as well as the main effects and interactions of the four variables presented in Table 2 on particle size (-10 micron).

Yates algorithm was used to determine the main effects and their interactions, and ANOVA was used to analyze how the independent variables interact with each other and the effects of these interactions on the dependent variable.

The design matrix representation for the four factors ($2^4$) factorial design is given in Table 3. Table 4, showing the combined data of the Yates algorithm and the ANOVA table, was used to help determine the importance of factors in dry grinding experiments. These two tables are presented together because all data can be seen easily in a single table and create ease of operation. Table 5 is used to highlight the results of wet milling experiments and the importance of factors.

ANOVA uses the F statistic. The F test compares the amount of systematic variance in the data with the unsystematic variance. The calculated F value ($F_C$) is decided by comparing it with the critical value (table value) determined according to certain confidence levels (α) and degree of freedom. Commonly used confidence levels are 99% ($\alpha = 0.01$), 95% ($\alpha = 0.05$) and 90% ($\alpha = 0.10$). Unless otherwise stated in engineering calculations, a 95% confidence level is mostly preferred.

In Tables (4 and 5) where the dry and wet grinding test results are given, the degrees of freedom ($d_f$=1) of the variables and their interactions have the same value. In the same tables, the degrees of freedom of the error ($d_f$=3) also have the same value. The confidence level of 95% and degrees of freedom were taken as 1 and 3, respectively, to find the Table value corresponding to each grinding condition. In this case, the value from the relevant F table is compared with the $F_C$ value for the decision. If the $F_C$ value is greater than the $F_T$ value, the decision is effective. Conversely, if the calculated F value ($F_C$) is less than the table value ($F_T$), then the decision is not important.

Table 3

Design Matrix Representation for Four-Factor ($2^4$) Factorial Design (Bilir and Ipek, 2011)

| Yates Order | Effects: Ball size | Effects: Stirrer speed | Effects: Grinding time | Effects: Interstitial filling ratio | Levels: Ball size | Levels: Stirrer speed | Levels: Grinding time | Levels: Interstitial filling ratio |
|---|---|---|---|---|---|---|---|---|
| (1) | - | - | - | - | 3 | 300 | 30 | 0.75 |
| a | + | - | - | - | 5 | 300 | 30 | 0.75 |
| b | - | + | - | - | 3 | 600 | 30 | 0.75 |
| ab | + | + | - | - | 5 | 600 | 30 | 0.75 |
| c | - | - | + | - | 3 | 300 | 150 | 0.75 |
| ac | + | - | + | - | 5 | 300 | 150 | 0.75 |
| bc | - | + | + | - | 3 | 600 | 150 | 0.75 |
| abc | + | + | + | - | 5 | 600 | 150 | 0.75 |
| d | - | - | - | + | 3 | 300 | 30 | 1 |
| ad | + | - | - | + | 5 | 300 | 30 | 1 |
| bd | - | + | - | + | 3 | 600 | 30 | 1 |
| abd | + | + | - | + | 5 | 600 | 30 | 1 |
| cd | - | - | + | + | 3 | 300 | 150 | 1 |
| acd | + | - | + | + | 5 | 300 | 150 | 1 |
| bcd | - | + | + | + | 3 | 600 | 150 | 1 |
| abcd | + | + | + | + | 5 | 600 | 150 | 1 |

Table 4

ANOVA Table for Dry Grinding (Bilir and Ipek, 2011)

| Yates Order | -10 µm (%) | | | | Total Effect | *Sum of Square* | *Degrees of Freedom* | *Mean Square* | $F_C$ | $F_T$ | Decision |
|---|---|---|---|---|---|---|---|---|---|---|---|
| (1) | 45.0 | 84.5 | 222.4 | 536.1 | 1038.1 | --- | --- | --- | --- | --- | --- |
| a | 39.5 | 137.9 | 313.7 | 502.0 | -25.7 | 41.3 | 1 | 41.3 | 0.71 | 10.13 | *NS* |
| **b** | **72.0** | **165.5** | **189.0** | **-7.1** | **106.9** | **714.2** | **1** | **714.2** | **12.34** | **10.13** | Effective |
| ab | 65.9 | 148.2 | 313.0 | -18.6 | -0.9 | 0.1 | 1 | 0.1 | 0.00 | 10.13 | *NS* |
| **c** | **89.1** | **75.9** | **-11.6** | **36.1** | **215.3** | **2897.1** | **1** | **2897.1** | **50.05** | **10.13** | Effective |
| ac | 76.4 | 113.1 | 4.5 | 70.8 | 1.9 | 0.2 | 1 | 0.2 | 0.00 | 10.13 | *NS* |
| bc | 65.5 | 139.7 | -2.2 | 29.3 | -74.3 | 345.0 | 1 | 345.0 | 5.96 | 10.13 | *NS* |
| abc* | 82.7 | 173.3 | -16.4 | -30.2 | 30.7 | 58.9 | 1 | 58.9 | 1.02 | 10.13 | |
| d | 34.7 | -5.5 | 53.4 | 91.3 | -34.1 | 72.7 | 1 | 72.7 | 1.26 | 10.13 | *NS* |
| ad | 41.2 | -6.1 | -17.3 | 124.0 | -11.5 | 8.3 | 1 | 8.3 | 0.14 | 10.13 | *NS* |
| bd | 60.9 | -12.7 | 37.2 | 16.1 | 34.7 | 75.3 | 1 | 75.3 | 1.30 | 10.13 | *NS* |
| abd | 52.2 | 17.2 | 33.6 | -14.2 | -59.5 | 221.3 | 1 | 221.3 | 3.82 | 10.13 | *NS* |
| cd | 70.2 | 6.5 | -0.6 | -70.7 | 32.7 | 66.8 | 1 | 66.8 | 1.15 | 10.13 | *NS* |
| acd* | 69.5 | -8.7 | 29.9 | -3.6 | -30.3 | 57.4 | 1 | 57.4 | 0.99 | 10.13 | |
| bcd | 94.5 | -0.7 | -15.2 | 30.5 | 67.1 | 281.4 | 1 | 281.4 | 4.86 | 10.13 | *NS* |
| abcd* | 78.8 | -15.7 | -15.0 | 0.2 | -30.3 | 57.4 | 1 | 57.4 | 0.99 | 10.13 | |
| | | | | | Error | 173.7 | 3 | 57.9 | | | |
| | | | | | Total | 4893.2 | 15 | | | | |

* High-level interactions used in error prediction

*NS : Not significant*

When Figure 2 and Table 4, which show the dry grinding experiments, are evaluated, it is seen that the effect of grinding time and stirrer speed on grinding efficiency (according to particle size) is significant. The total effect values of these two main variables on grain size (-10 microns) are 106.9 and 215.3, respectively. According to the total effect values, the most important variable in dry grinding conditions is grinding time. Grinding time has powerful influences on the grinding efficiency, depending on the material property and the value of the specific surface area. Increasing the grinding time results in the formation of very fine-sized products and the increase of the specific surface area value. It should be considered that the product, which becomes very fine during long grinding times, will create a cushioning effect that slows down the breakage rate (Cayirli and Gokcen, 2021; Cuhadaroglu and Kara, 2015; Samanli, Cuhadaroglu, Ucbas, and Ipek, 2010). Ball size and interstitial filling ratio are among the variables that affect grinding efficiency. In general, increasing the ball size and decreasing the interstitial filling ratio increase the ground product's surface area (Altun, Benzer, and Enderle, 2013). However, depending on the total effect values in the ANOVA table, it can be interpreted that the effects of these two variables on the grinding efficiency are less important than the others. These variables' total effect values are -25.7 and -34.1, respectively.

While interpreting, variance analysis table decisions should be taken into consideration. The numerical magnitude and sign of the total effect values of the effective variables are checked. According to Table 4, the sign of both variables is '+'. This means that when the level value of the relevant variable is changed from the low-level value to the high-level value, the effect on the dependent variable is increasing. If the sign of the relevant variable is '-', it means that when the level value of the variable is changed from a low-level value to a high-level value, its effect on the dependent variable is reduced.

In dry grinding experiments, for the particle size distribution to be within the targeted size range, the ball size and interstitial filling ratio should be at low levels, and the grinding time and stirrer speed should be at high levels.

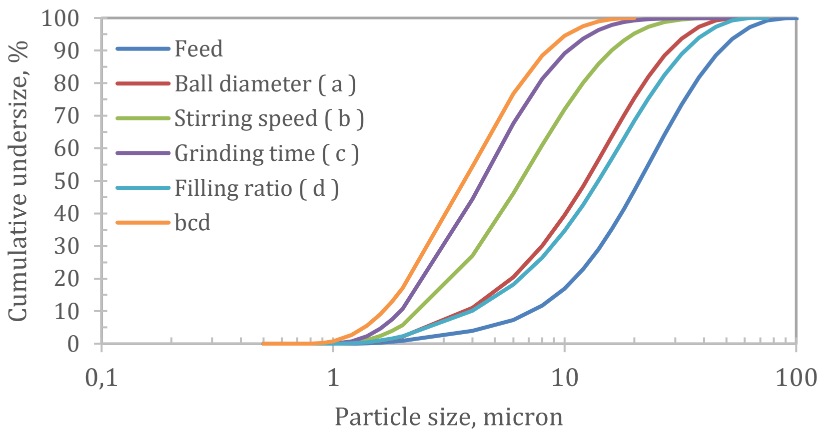


Figure 2. K-Feldspar's Dry Grinding Experiments Results

Table 5

ANOVA Table for Wet Grinding (Bilir and Ipek, 2011)

| Yates Order | -10 µm (%) | | | | Total Effect | *Sum of Square* | *Degrees of Freedom* | *Mean Square* | $F_C$ | $F_T$ | Decision |
|---|---|---|---|---|---|---|---|---|---|---|---|
| (1) | 39.2 | 88.2 | 239.0 | 615.6 | 1127.4 | --- | --- | --- | --- | --- | --- |
| **a** | **49.0** | **150.8** | **376.6** | **511.8** | **93.4** | **545.2** | **1** | **545.2** | **37.68** | **10.13** | Effective |
| **b** | **70.0** | **179.6** | **182.2** | **29.8** | **185.0** | **2139.1** | **1** | **2139.1** | **147.84** | **10.13** | Effective |
| ab | 80.8 | 197.0 | 329.6 | 63.6 | -27.4 | 46.9 | 1 | 46.9 | 3.24 | 10.13 | *NS* |
| **c** | **83.7** | **68.9** | **20.6** | **80.0** | **285.0** | **5076.6** | **1** | **5076.6** | **350.85** | **10.13** | Effective |
| ac | 95.9 | 113.3 | 9.2 | 105.0 | -13.4 | 11.2 | 1 | 11.2 | 0.78 | 10.13 | *NS* |
| bc | 100.0 | 134.5 | 32.8 | -14.2 | -29.0 | 52.6 | 1 | 52.6 | 3.63 | 10.13 | *NS* |
| **abc** | **97.0** | **195.1** | **30.8** | **-13.2** | **-57.0** | **203.1** | **1** | **203.1** | **14.03** | **10.13** | Effective |
| **d** | **29.7** | **9.8** | **62.6** | **137.6** | **-103.8** | **673.4** | **1** | **673.4** | **46.54** | **10.13** | Effective |
| ad | 39.2 | 10.8 | 17.4 | 147.4 | 33.8 | 71.4 | 1 | 71.4 | 4.93 | 10.13 | *NS* |
| bd | 45.0 | 12.2 | 44.4 | -11.4 | 25.0 | 39.1 | 1 | 39.1 | 2.70 | 10.13 | *NS* |
| abd* | 68.3 | -3.0 | 60.6 | -2.0 | 1.0 | 0.1 | 1 | 0.1 | 0.00 | 10.13 | |
| cd | 52.8 | 9.5 | 1.0 | -45.2 | 9.8 | 6.0 | 1 | 6.0 | 0.41 | 10.13 | *NS* |
| acd* | 81.7 | 23.3 | -15.2 | 16.2 | 9.4 | 5.5 | 1 | 5.5 | 0.38 | 10.13 | |
| **bcd** | **96.6** | **28.9** | **13.8** | **-16.2** | **61.4** | **235.6** | **1** | **235.6** | **16.28** | **10.13** | Effective |
| abcd* | 98.5 | 1.9 | -27.0 | -40.8 | -24.6 | 37.8 | 1 | 37.8 | 2.61 | 10.13 | |
| | | | | | Error | 43.4 | 3 | 14.5 | | | |
| | | | | | Total | 9110.2 | 15 | | | | |

* High-level interactions used in error prediction

*NS : Not significant*

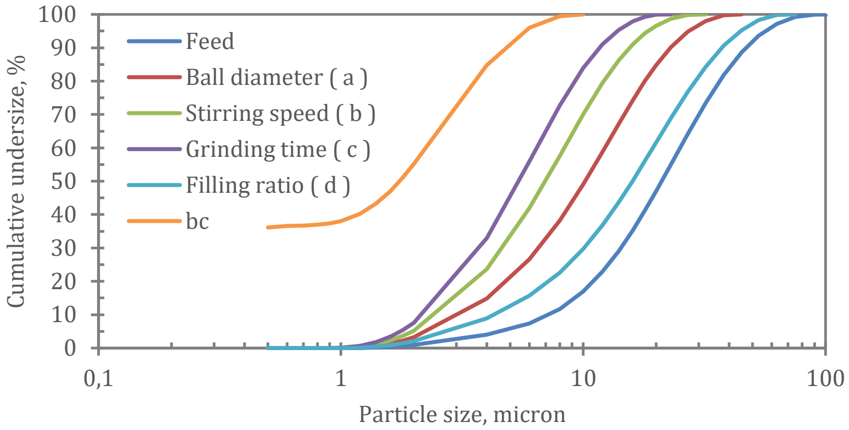


Figure 3. K-Feldspar's Wet Grinding Experiments Results

When Figure 3 and Table 5 showing wet grinding experiments are evaluated, it is seen that the effect of all independent variables subject to the design on grinding efficiency is significant. The total effect values of the four main variables on the grain size (-10 micron) are 93.4, 185.0, 285.0, and -103.8, respectively. According to the total effect values, the most important variable in wet grinding experiments is the grinding time. The remaining variables, in order of importance, are as follows: stirrer speed, interstitial filling ratio, and ball size. According to the ANOVA table, stirrer speed and ball size have '+' sign, while interstitial filling ratio has '-' sign.

In wet grinding tests, for the particle size distribution to be within the targeted size range, the ball size, stirrer speed, and grinding time should be at high levels, and the interstitial filling ratio should be at a low level.

## 4. Conclusions

The effects of variables such as grinding time and stirrer speed in dry grinding and ball size, stirrer speed, grinding time, and interstitial filling ratio in wet grinding were more pronounced when compared to variables such as interstitial filling ratio and ball size in dry grinding. In dry grinding, increasing the grinding time causes the formation of very fine-sized products and a decrease in grinding efficiency. The finer particles formed by the long grinding times slow down the breaking rate of the product with its cushioning effect. Furthermore, grinding may slow down at high interstitial filling ratios because increasing material amount reduces the ball-particle contact number. Consequently, the influence of these two variables on the particle size distribution is less in dry milling for this reason.

Considering all experiments in the $2^4$ full factorial experimental design, it has been shown that the stirred ball mill can efficiently grind potassium feldspar to the targeted particle size distribution (1-10 microns range).

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## Contribution of Researchers

Kemal BILIR contributed to scientific publication research, preparation of samples, performing experiments and analyzes, the development and writing the article, discussion of results, and project management. Halil IPEK contributed to the development, writing, and review of the article, evaluation of the results and, project consultancy.

## Conflict of Interest

The authors declared no conflict of interest.

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