Application note No. 54 | 2024

Sample preparation of raw materials for refractories- Improved efficiency using online tool condition monitoring

Abstract

The manufacture of refractories requires raw materials that comply with strict specifications in terms of chemical and mineralogical properties. Due to the wide variety of different materials, grinding is one of the most challenging steps in the sample preparation process. Many materials require a high energy input to achieve a sufficiently small particle size but at the same time have a tendency to agglomerate. In this application note we describe the effect of ethanol on the grinding efficiency of a vibratory disc mill. We also show how the TCM module of PrepMaster Analytics can be used to detect inefficient grinding or agglomeration online. This makes the TCM module a valuable tool for application development and laboratory monitoring.

Key words

• **Refractories** • **Bauxite** • **Clay** • **Chamotte** • **Grinding** • **Tool Condition Monitoring**

Introduction

Refractories are materials that are resistant to decomposition by heat or chemical attack. Their main characteristics are that they maintain their stability and rigidity at high temperatures, withstand heat shocks and are chemically inert. Refractory engineering is a key technology in various production processes that occur in the cement, steel, non-ferrous, and other industries.

Being mostly ceramics, refractories are inorganic, non-metallic compounds that can be porous or non-porous and have a wide range of crystallinity (crystalline, polycrystalline, amorphous, composite). Refractories are usually formed from oxides, carbides, or nitrides of the following elements: silicon, aluminium, magnesium, calcium, boron, chromium, and zirconium.

For the manufacture of high-quality refractory materials, it is particularly important to strictly control the quality of raw materials. This is because there is a huge variety of raw materials, a wide range of sources and a many different requirements for refractory materials. Sample preparation and analysis are therefore very demanding and often follow standards like, e.g. ISO12677 or ISO 21079.

A critical step in sample preparation is the comminution of the raw material to particle sizes suitable for analytical techniques such as X-ray spectroscopy or further sample processing such as acid digestion. One of the main challenges is the variety of different materials, which can lead to different grinding results due to different properties. In particular, the formation of agglomerates must be considered as these may interfere with subsequent processes.

In this application note, we investigated the grinding process of four different raw materials used in production of refractories: clay, bauxite and two different types of chamotte. During the grinding process we simultaneously monitored the grinding power of the vibratory disc mill. This allowed us to assess the grinding efficiency online.

Methods

All grinding trials were carried out on the HP-MP combined mill and press (Herzog, Germany). For this application note we used identical grinding parameters for clay, bauxite, chamotte type 1 and 2 (Figure 1) at a rotation speed of 1400 rpm for 120 s. For each material, two samples of 30 g were ground- one without any grinding aid, the other with 100 µl of ethanol.

Figure 1: *Photographs of four raw materials used for production of refractories. For this application note, we assessed the grindability of clay, bauxite and two types of chamotte.*

During each grinding trial, the grinding power was automatically assessed by using the Disc Mill TCM module of the PrepMaster Analytics software Suite.

After each trial, we measured the loss of material within the grinding vessel. Additionally, a laser diffraction particle size analyzer (Mastersizer 3000, Malvern Panalytical, UK) was used to determine the particle size distribution. Depending on the amount of sample available, up to five individual grain size analyses were performed per sample.

Results

<u>Grinding of clay</u>

After grinding without ethanol almost all the sample material remained in the grinding vessel. Only about 1 % of the sample volume was discharged. The grain size analysis showed a multi-peaked pattern with a wide distribution with the peak at approx. 1000 µm being particularly prominent (Figure 2, B). The grinding power graph displayed a continuous increase from about 0.61 kW at 5 s to about 0.81 kW at 120 s (Figure 2, A).

After grinding with ethanol 79 % of the sample was released from the grinding vessel. The grain size analysis showed a single peak at 4 µm with no evidence of formation of agglomerates (Figure 2, D). The grinding power showed a decrease from 0.62 to values around 0.41 (Figure 2, C).

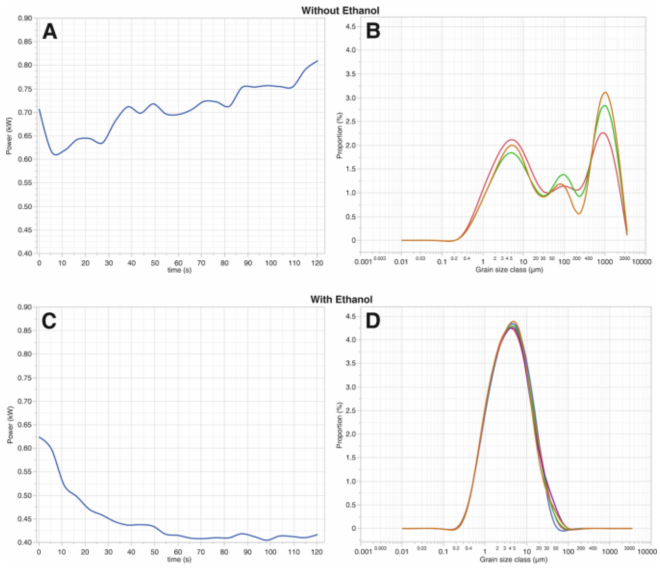

Figure 2: *Grinding power curve and grain size analysis for grinding of clay*

Grinding of bauxite

After grinding of bauxite without ethanol 59 % of the material was released from the grinding vessel. Grain size analysis revealed a bimodal distribution with peaks around 4 µm and 200 µm indicative of agglomerates. The grinding power curve showed an increase to a maximum of 0.72 kW at the end of the grinding process (Figure 3, A-B).

Grinding with ethanol resulted in a more effective material release of 86 %. In the grain size analysis, only one peak appeared around 4 µm without any indications of agglomeration. The grinding power showed a decrease from about 0.61 kW to values around 0.41 kW (Figure 3, C-D).

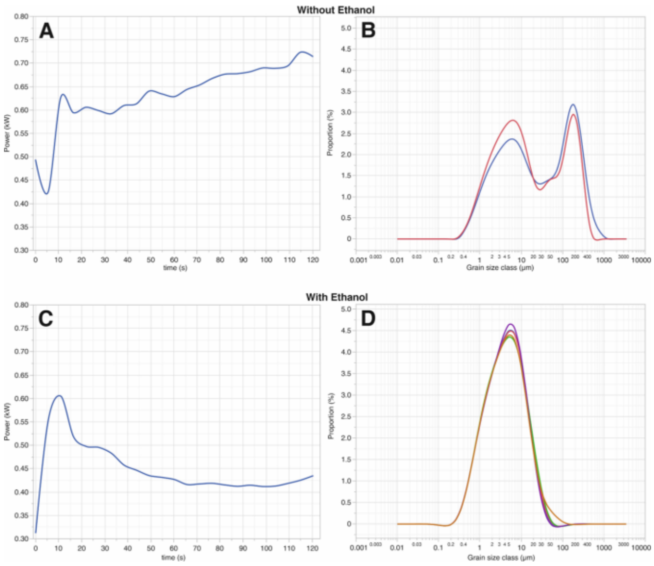

Figure 3: *Grinding curve and grain size analysis for grinding of bauxite*

Grinding of chamotte type 1

Grinding of chamotte type 1 without ethanol led to a poor material release of only 3 %. Grain size analysis showed at least three peaks between 1 and 400 µm. In the grinding power graph we found an increase from 0.60 kW to 0.80 kW within the first 70 s. The power then remained at a value around 0.80 kW until cthe end of the grinding process (Figure 4, A-B).

Grinding with ethanol increased the sample proprtion to be released from the grinding vessel to 77 %. The grain size analysis showed only one peak around 4 µm. In the grinding power diagram the values contiously decreased from 0.66 kW to values at 0.4 kW (Figure 4, C-D)

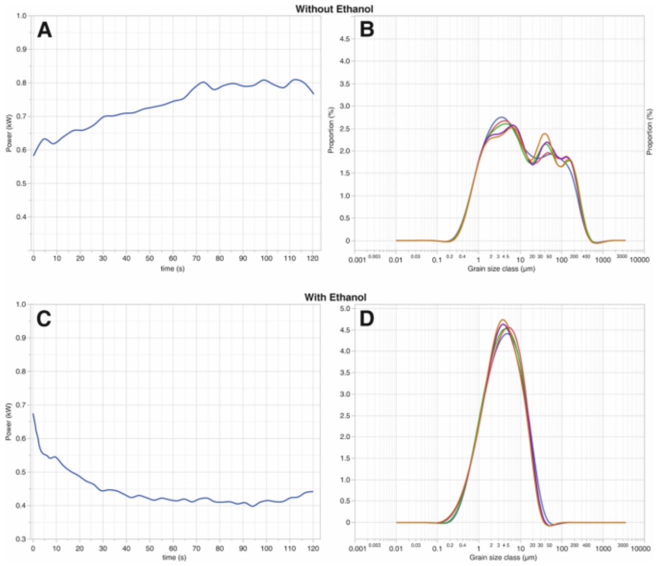

Figure 4: *Grinding curve and grain size analysis for grinding of chamotte type 1*

Grinding of chamotte type 2

Grinding of chamotte type 2 without ethanol led to a material release of only 8 %. The grain size analysis showed a multimodal distribution within the range of 0.2 µm to 100 µm and a significant peak at 1000 µm indicative of agglomeration. The power graph revealed a continous increase from 0.60 kW to 0.80 kW (Figure 5, A-B).

Grinding with ethanol improved the release of chamotte type 2 to 76 %. In the grain size analysis, we found a bimodal pattern with peaks around 3 µm and 400 µm. The graph showed an initial drop from 0.55 kW to 0.43 kW up to 50 s. The curve then rose rapidly to 0.70 kW at the end of the grinding procedure (Figure 5, C-D).

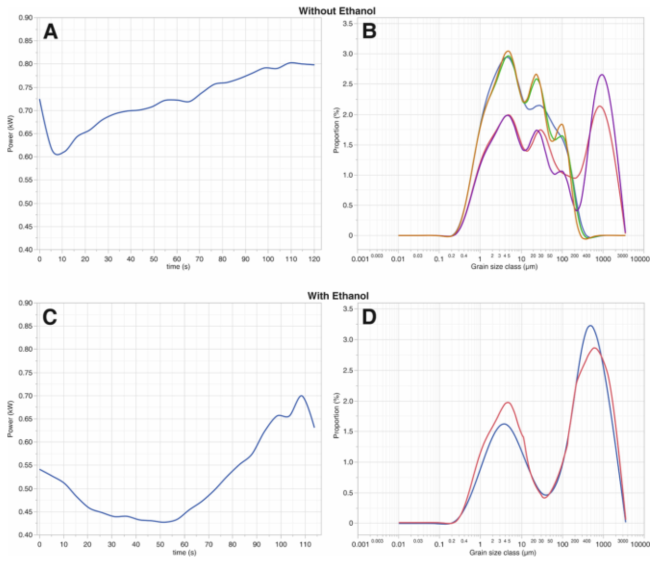

Figure 5: *Grinding curve and grain size analysis for grinding of chamotte type 2*

Discussion

This study shows that the grinding of raw materials used for the production of refractories can be significantly improved by the addition of ethanol. Ethanol resulted in an improvement in grinding efficiency as shown by a shift from a multimodal to a monomodal distribution pattern in particle size analysis, with only one maximum in the range of about 4 µm. Furthermore, the addition of 100 µl ethanol prevented the formation of agglomerates in all materials except for chamotte type 2. This was particularly evident in clay and bauxite where the peaks at 1000 and 200 µm were eliminated by ethanol.

Interestingly, the grinding result correlated with the grinding power values recorded in parallel. Efficient grinding without agglomeration was correlated with a continuous decrease in power throughout the grinding process. In contrast, inefficient grinding with agglomeration was associated with an increase in grinding power. The shape of the power curve allowed specific predictions about the grinding process and outcome. A continuous increase in power up to the end of grinding, as with clay and bauxite, was correlated with a pronounced agglomerate formation with a significant fraction in the 1000 µm range. However, an initial increase with subsequent constant power, as in the case of chamotte type 1, was accompanied by only moderate to low agglomerate formation.

In the case of chamotte type 2, the addition of ethanol resulted in an initial decrease in power followed by a sharp increase. This indicates that ethanol was initially sufficient to prevent agglomeration. As the material surface increases during the grinding process, ethanol may no longer be sufficient due to an under-dosing. This then leads to agglomeration formation, which is accompanied by a significant increase in grinding power.

This study is one of a series of other application notes (1-4) showing that online monitoring of grinding efficiency is possible using the TCM module of PrepMaster Analytics. This is a helpful support in developing applications for new sample materials. It also allows early detection of inefficient grinding processes during routine operation. In this case, further preparation and analysis steps could either be aborted or appropriate countermeasures be initiated, such as the addition of a further dose of ethanol.

References

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