Influence of grinding aids using pine resins or microcelluose on the grindability of clinker

Abstract

We previously used the tool condition monitoring (TCM) module of the combined milling and pelletizing machine HP-MP to determine the influence of various grinding aids (GAs) on the grinding process of clinker. We found significant differences between microcelluose (MCC)- and polyethylene wax-based GAs. Here, we investigated the differences between MCC-based GAs and pine resins-based GAs. Compared to the MCC-based GAs, the pine resins-based GA showed a significant higher initial energy demand probably reflecting the difficult dispersion of the viscous GA among the sample material. In the further course of grinding, we found a significantly lower energy demand and higher acceleration compared to the MCC-based GA. Correspondingly, the achievable specific surface was significantly higher for pine resins- based GA compared to MCC-based GAs.

Key words

• **Clinker** • **Grinding aid** • **Pine resins** • **Microcellulose** • **Agglomeration**

Introduction

In a previous application note [1] we showed that tool condition monitoring (TCM) of a vibrating disc mill allows to objectively determine the impact of solid grinding aids (GAs) on the grindability of clinker. Accordingly, the temporal pattern of acceleration and power input as well as the specific energy demand revealed significant differences between the various GAs.

In the previous study, we compared the effects of microcellulose (MCC)-based GAs with those of a polyethylene wax-based GA. We were able to demonstrate that the MCC-based GAs exhibited a specific TCM pattern reflecting particle size reduction and, above a certain specific surface area, agglomerate formation. By contrast, the less effective clinker grinding by using polyethylene wax was accompanied by a significantly higher power input and energy demand while the acceleration of the grinding vessel was reduced.

In this application note, we aim to compare the effects and TCM pattern of MCC-based GAs with those of a GA composed of pine resins and a small amount of castor oil (Figure 1). The GA made of pine resins and castor oil is frequently used in cement laboratories as it allows the easy production of a stable pressed pellet.

Furthermore, the pine resins- based GA enables the preparation of a sample which is equally suitable for the analysis by r-ray fluorescence and x-ray diffractometry.

Figure 1: *Photographs of the grinding aids of (A) the type HMPA 40 (MCC) and (B) the type HMPA 100 (pine resins)*

Methods

We ground ordinary Portland cement (OPC) clinker from a German cement producer using the combined laboratory mill and press of the type HP-MP (Herzog, Germany). The HP-MP was equipped with the Herzog tool condition monitoring (TCM) module for the real-time evaluation of grinding performance.

We conducted a total of three test series, with 15 samples of 30 g clinker material being ground in each test series. Initially, the clinker was ground without any GA (test series A), subsequently with the solid grinding aid of the type HMPA 40 (test series B) and HMPA 100 (test series C). All investigated solid GAs are Herzog standard additives frequently used in the sample preparation of various materials. The HMPA 40 mainly consists of MCC and a small amount of Licowax C in a mixing ration of 1:9. The HMPA 100 is mainly composed of pine resins with a small amount of castor oil.

All tests were performed at the same rotation speed of 1400 rpm. Each sample within the test series A-C was ground with a different grinding time: The first sample was ground for 20 s, then the grinding time was stepwise increased by 20 s up to 300 s for the last sample. For each sample, the ground material was discharged into a cup and the specific surface was determined by using laser granulometry (Mastersizer 3000, Malvern, UK).

The trials were carried out within a small-footprint automation system consisting of the HP-MP and the granulometer connected by a belt system. All samples of a test series were loaded into the input magazine of the HP-MP. After completion of grinding, the ground sample was transported to the granulometer for determination of the grain size distribution and specific surface.

During each grinding trial, the grinding power of the HP-MP and the acceleration of the grinding vessel were automatically recorded at a sampling frequency of 100 Hz. The course of grinding power and acceleration was plotted over time for each test series (Figure 2). Based on the integral of the grinding power we determined the specific energy demand and calculated the grindability for each test series.

We plotted the specific surface and the grindability over time (Figure 3 A, B) as well as the specific energy demand over the specific surface (Figure 3 C)

All data of acceleration, specific grinding power, specific energy demand, grindability and granulometry were automatically recorded, processed, and displayed by using the PrepMaster Analytics software (Herzog, Germany).

Results

Temporal pattern of grinding power and acceleration

During grinding without any GA, we found a continuous increase of the grinding power until approx. 200 s (Figure 2 A). From this point, the power dropped from 0.55 kW to values between 0.45 and 0.50 kW. Parallel to the increase in power, the acceleration showed a continuous decline. The drop in power at 200 s was accompanied by a simultaneous drop in acceleration and subsequent plateau formation.

During grinding with HMPA 40, we found an initial increase of grinding power to 0.25 kW at

about 15 s (Figure 2 B). From here, the power decreased continuously to 0.2 kW forming a plateau from about 60 s to 160 s. Acceleration also showed a peak at about 15 s followed by a sudden drop of acceleration until 30 s and a subsequent increase of acceleration until 160 s. At 160 s, the grinding power rapidly increased to 0.25 kW with a subsequent further continuous increase to 0.3 kW. The increase in grinding power was paralleled by a rapid decrease of acceleration followed by plateauing.

During grinding with HMPA 100, we observed a significant initial power peak of 0.45 kW at 15 s (Figure 2 C). Subsequently, the grinding power decreased to about 0.17 kW at 80 s. After 80 s we found further, albeit slower decline of the grinding power to 0.1 kV at 300 s. The transient major increase in power was accompanied by a transient decrease in acceleration. At 80 s, acceleration showed a significant gain followed by a slight further increase.

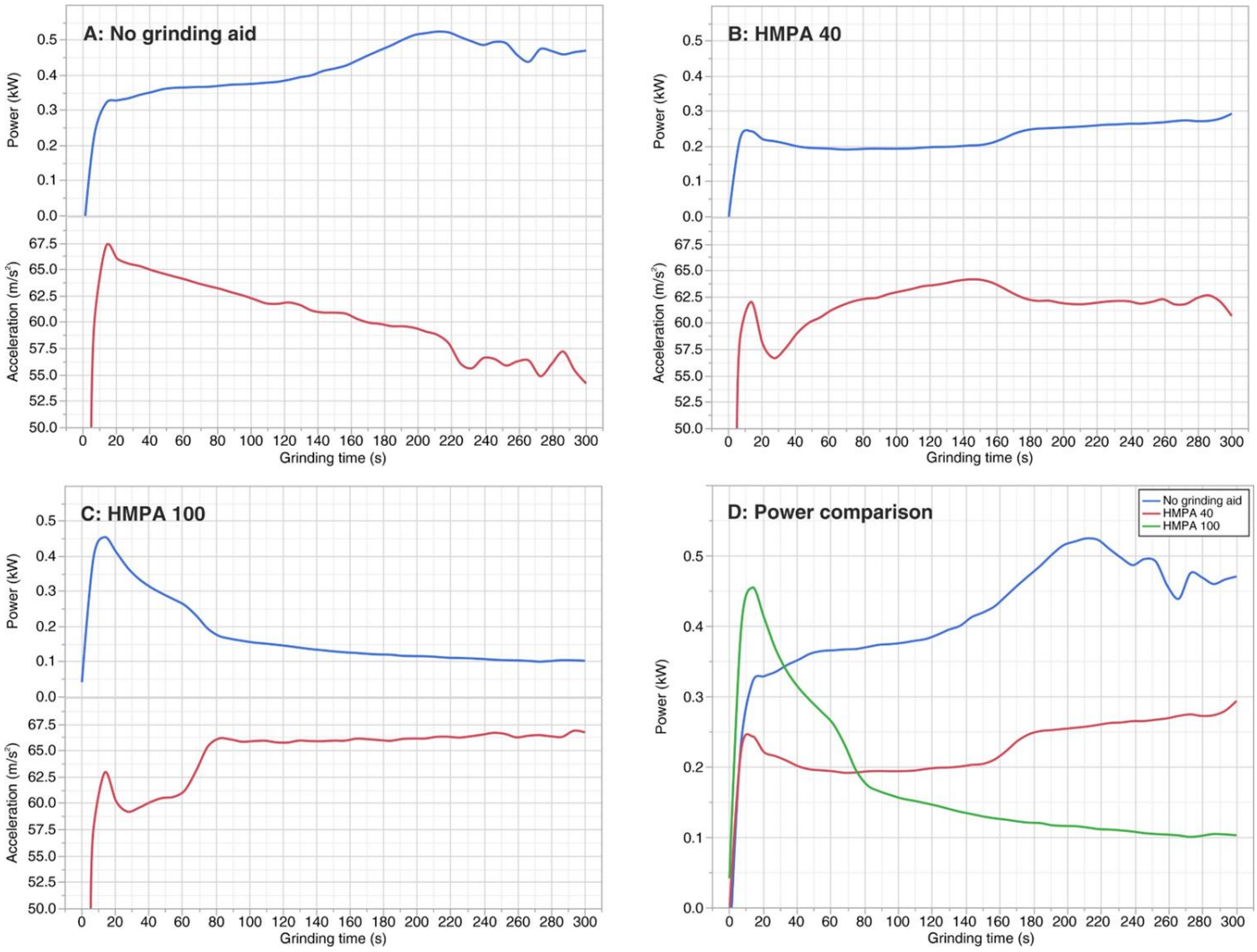


Figure 2: *Graphs showing the temporal pattern of the power input (blue in A-C) and acceleration of the grinding vessel (red in A-C) for the different GAs and use of no GA. (D) displays the power course separately shown in A-C.*

In Figure 2 D, we plotted the grinding power curves of the test series A-C on one graph. The graph clearly shows that the use of HMPA 100 was initially associated with a higher grinding power compared to the use of HMPA 40. From 80 s, the power of grinding with HMPA 100 decreased below the values for grinding with HMPA 40. Grinding without any GA showed a generally higher level compared to grinding with HMPA 40 or HMPA 100.

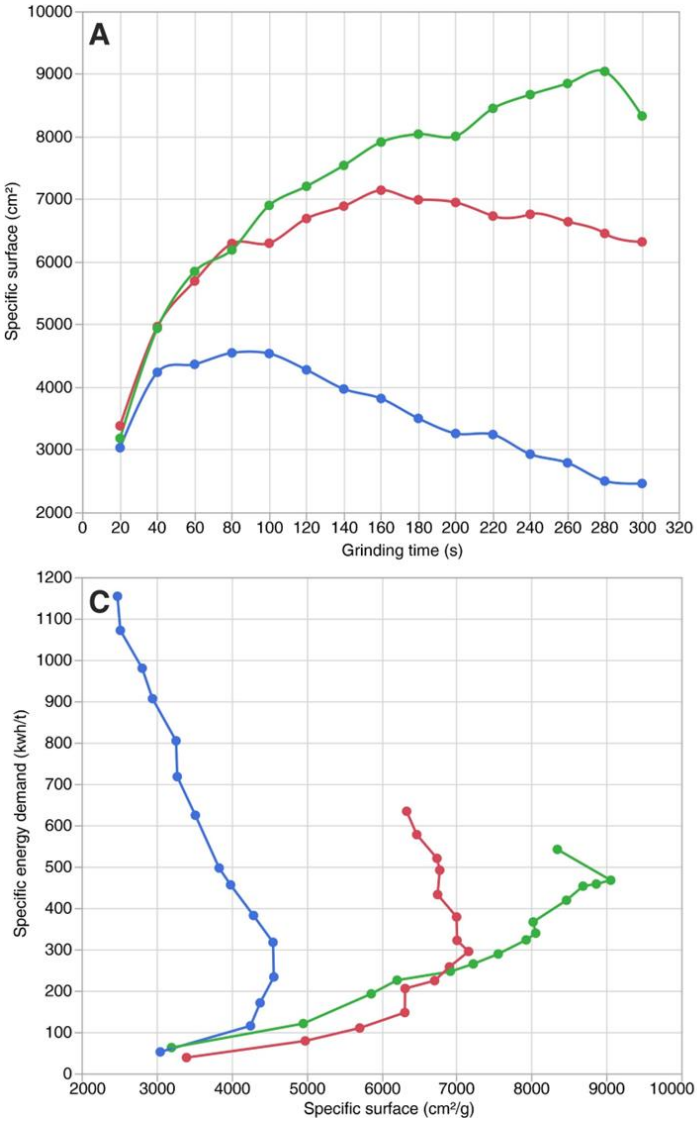
Influence of GA on specific surface, grindability and specific energy demand

The time series graph for the specific surface (Figure 3 A) revealed a significant different pattern for grinding without GA compared to grinding with HMPA 40 and HMPA 100. For grinding without GA, the maximum reachable specific surface was about 4500 cm^2/g. After 100 s of grinding, the specific surface

continuously decreased to values of about 2500 cm^2/g. For HMPA 40, the maximum specific surface was about 7000 cm^2/g. After 160 s, the specific surface was reduced to a value of about 7200 cm^2/g. For HMPA 100, the specific surface continuously rose to a value of 9000 cm^2/g.

The grindability was the lowest if no GA was used (Figure 3 B). From the beginning the grindability decreased strongly and approximated a value of zero. For HMPA 40, the grindability value sharply decreased until 100 s. Subsequently, it slowly declined to a value of 2.5 cm^2/J. For HMPA 100, the grindability decreased to 7.5 cm^2/J at 80 s and subsequently slowly declined to a value of about 5 cm^2/J at 300 s. From 160 s, the grindability using HMPA 100 was higher compared to HMPA 40.

Plotting the specific energy demand over the specific surface (Figure 4 C) revealed the influence of the grinding aid on the formation of agglomerates. Without GA, the maximum specific surface of 4500 cm^2/g was reached at a specific energy demand of about 300 kWh/t. The subsequent reduction of the specific surface was due to agglomeration of the sample material. For HMPA 40, the maximum specific surface was 7000 cm^2/g at a specific energy of 300 kWh/t and agglomeration beyond this point. For HMPA 100, a maximum specific surface of 9000 cm^2/g could be reached at a specific energy demand of about 480 kWh/t.



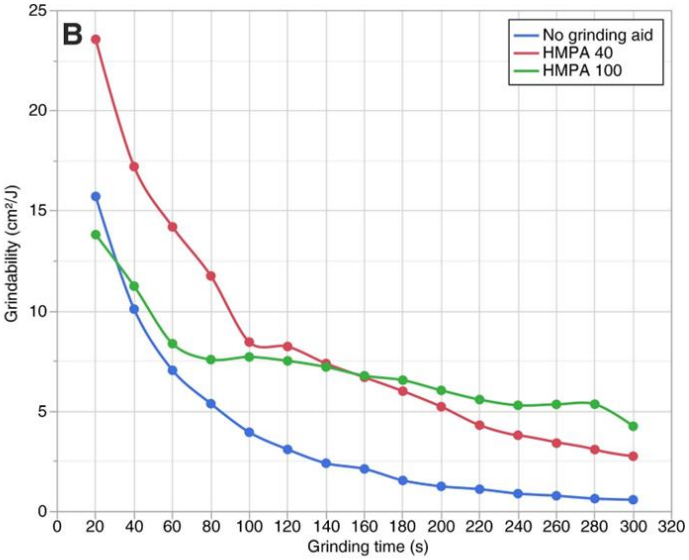


Figure 3: *Graphs showing (A) the course of the specific surface over time, (B) the course of grindability over time and (C) the correlation between the specific energy demand and the specific surface. Each graph shows the pattern for grinding without GA, with HMPA 40 and HMPA 100 .*

Discussion

The results of this application note confirm that TCM monitoring of the vibrating disc mill is a suitable method to investigate the effect of GAs. The assessment of grinding power, specific energy demand and grindability allow an unbiased comparison between different GAs and provides useful assistance in the development of applications for various sample materials. Moreover, monitoring of the grinding process as part of the laboratory routine can easily detect deviations in sample preparation that may have impact on the analytical accuracy and precision.

The study demonstrates that HMPA 100 results in the highest grinding efficiency of the clinker

investigated in this study. The use of HMPA 100 made it possible to achieve a specific surface area of up to 9000 cm^2/g without the formation of agglomeration. HMPA 40 also has a significant beneficial effect on grindability while the maximum attainable specific surface is lower with only 7000 cm^2/g. The total specific energy demand during grinding with HMPA 40 is comparable to HMPA 100. However, the temporal pattern of acceleration and power shows that a significant portion of the applied energy is not used to increase the surface area, but to destroy agglomerates. Without the use of any GAs, a specific surface area of only 4500 cm^2/g can be achieved. The total energy demand without GAs is about twice as high as with HMPA 40 or HMPA 100. When milling without a GA, most of the applied energy is not used for decreasing particle size but for agglomerate disintegration.

The power and acceleration graphs (Figure 2) provide insights into the mode of action of HMPA 40 and HMPA 100. The use of HMPA 40 is associated with a low initial power peak followed by a rapid drop to a power minimum at 40 s. This pattern indicates an instantaneous dispersion of the grinding aid among the host sample material. The physical properties of MCC as the main component of HMPA 40 are consistent with the observed temporal pattern. MCC is a material with a high flowability facilitating the fast spreading among the host material [2]. The distribution may be further aided by the lubricating properties of Licowax C added to the MCC. MCC has a filamentary structure which enhances interparticle contacts, neutralizes clinker particle charges and reduces the formation of agglomerates. However, if the specific clinker surface exceeds a threshold, the capacity of MCC to shield electrostatic forces of clinker particles is exhausted, and the formation of agglomerates is initiated. In the present study, the threshold is surpassed at a value of 7000 cm^2/g as indicated by the significant increase in power and decrease in acceleration.

Using HMPA 100, the initial power peak is significantly higher than compared to HMPA 40. This is likely due to the properties of the pine resin, which is the major ingredient of the HMPA 100. Pine resin is a naturally occurring substance featuring thermoplastic characteristics and a high viscosity [3]. A significant amount of energy must therefore be applied to convert the resin into a flowable state so that it can be dispersed among the host material. After 80 s, the energy demand is continuously decreasing and significantly lower compared to HMPA 40. Moreover, there is no evidence of agglomerate formation during the entire grinding cycle. The high efficacy in prevention of agglomerates may be due to the aliphatic hydrocarbon structure of the resin causing a highly effective reduction of the electrostatic forces of the host clinker particles.

References

[1] Herzog Application Note 51/2023

[2] L. Miethke, et al.: Opposing Effects of Additives in Dry Milling and Tableting of Organic Particles. Pharmaceutics, 2021, 13, 1434

[3] I. Belyamani, et al.: Rheological Properties of Southern Pine Oleoresins. Applied Reheology, 2015, 25, 1-12

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