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Effect of the Grinding Media Fill Level, the Quality and the Fraction Width of Grinding Media during Comminution with Agitator Bead Mills

Business Unit
GRINDING & DISPERSING

EFFECT OF THE GRINDING MEDIA FILL LEVEL, THE QUALITY & THE FRACTION WID

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1. GRINDING MEDIA FILL LEVEL

1.1. Definition of the grinding media fill level

The grinding media filling level is defined very differently by the manufacturers of agitator bead mills. Therefore, in many cases, direct comparison of grinding media fill levels is difficult. Depending on the geometry of the grinding system and the philosophy of the manufacturer, a 100% grinding media volume, $V_{GM,100\%}$, is usually defined. For the mill used, this value indicates the maximum volume of a grinding system that may be filled with grinding media in order to enable reliable operation of the mill. The grinding media filling level φ_{GM} indicates which percentage of this 100% grinding media volume was filled with grinding media.

The grinding media volume in the grinding chamber is to be specified as bulk volume, $V_{GM,bulk}$, as a function of the bulk density, $\varphi_{GM,bulk}$, of the grinding media. The grinding media manufacturer usually states the bulk density in the grinding media specifications. The grinding chamber should **always** be filled with a grinding media mass calculated from the required bulk volume of grinding media and the bulk density of the grinding media specified by the manufacturer (see Eq. (1)).

$$\varphi_{GM} = \frac{V_{GM,bulk}}{V_{GM,100\%}}$$

$$m_{GM} = \varphi_{GM} \cdot V_{GM,100\%} \cdot \rho_{GM,bulk} \qquad (1)$$

Filling the grinding chamber beyond a defined volume of grinding media carries the risk that the grinding chamber will be overfilled or underfilled. Simple series tests have shown that the deviation in the grinding media mass was sometimes greater than 20% when different test subjects measured a volume of 1 liter of identical grinding media. The reason for this is that some of the participants in the test series wanted to determine the volume very precisely and, instead, determined a tamped volume of the grinding media, while others only determined the volume of the grinding media approximately and thus often filled a smaller mass of grinding media.

1.2. Change in the Grinding Media Fill Level due to Grinding Media Wear

The grinding media fill level, φ_{GM}, has a direct effect on the power input into a grinding chamber (see Fig. 1). The power input is also a function of the density and viscosity of the product suspension, the agitator speed, the grinding media density and, of course, the grinding chamber volume of the mill.

During operation of an agitator bead mill, countless collisions of grinding media are generated, which inevitably lead to grinding media wear and thus to contamination of product suspension and to a reduction of the grinding media filling level over time.

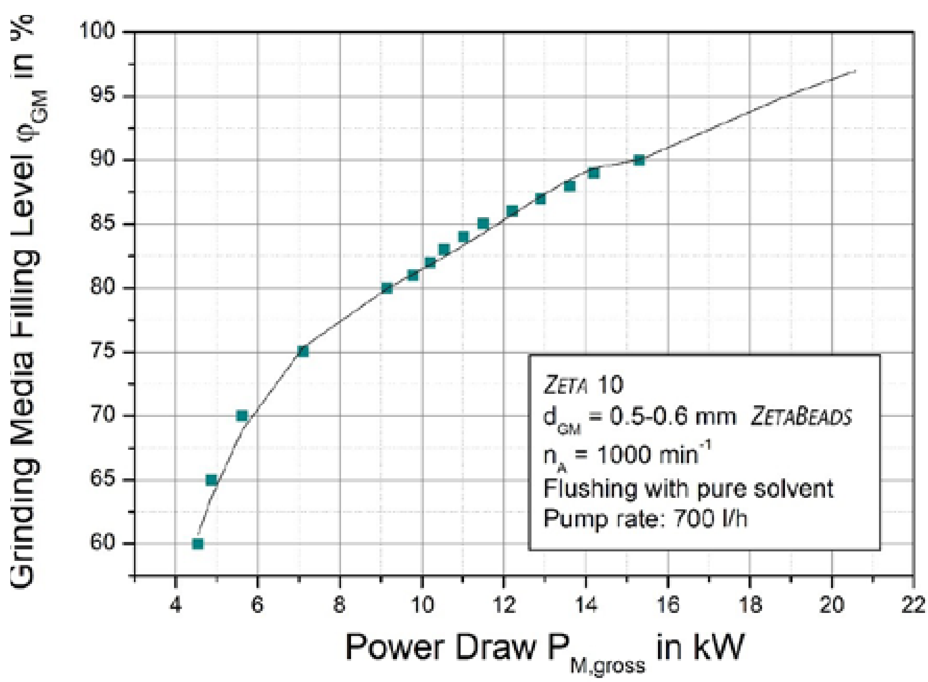
A simple method of measuring the grinding media fill level of a mill is to use the power input of the mill as a function of the grinding media fill quantity as a measured value. For this purpose, when a new mill is commissioned, a characteristic curve of the mill must be created, with the help of which the grinding media fill level can be determined very precisely when the mill is being purged, without the need to open the grinding chamber or invest additional time for this purpose.

Fig. 1 shows such a characteristic curve for a *Zeta*® 10 mill. The mill is purged with a special cleaning fluid after each batch. The cleaning fluid depends on the product and can be water, a special solvent or even an oil. If different products are processed in the mill that require purging with different cleaning fluids and operation with different grinding media, an independent characteristic curve must be recorded for each cleaning fluid and each type of grinding media.

The grinding media filling level used in NETZSCH agitator bead mills is usually in a range between 70 and 90%. In rare cases, less than 70% of the 100% grinding media volume, $V_{GM,100\%}$, is filled with grinding media. In other exceptional cases, grinding media fill levels, φ_{GM}, of up to 95% are used.

Fig. 1

Power input as a function of grinding media fill level



In this case, a grinding media filling level of 90% was defined. During start-up of the mill, the initial filling level was only 60%. The mill was then operated with an agitator speed of 1 000 min^{-1}, while it was purged with cleaning fluid at a defined flow rate of 700 l/h. The resulting gross power input, $P_{M,Brutto}$, was approx. 4.5 kW. Finally, this procedure was repeated, increasing the grinding media fill level until the entire grinding media charge was present in the grinding chamber.

The resulting machine characteristic curve can now be used during normal purge operation between two batches in order to determine very precisely which grinding media mass has been passed into the product due to wear, or how much grinding media must be refilled in order to adjust the grinding media fill level back to 90% and keep the power input at a constant level.

Example:

The 100% grinding media volume, $V_{GM,100\%}$, of the *Zeta*® 10 mill used here is specified as 9.5 liters in the operation manual. This means that 1% of the entire grinding media volume corresponds to 95 ml. The grinding media used here were *ZetaBeads*® with a diameter of 0.5 - 0.6 mm. The bulk density, $\rho_{GM,bulk}$, specified by the manufacturer is 3.65 kg/dm^3.

At a grinding media fill level, φ_{GM}, of 90%, the gross power input into the mill in purge mode was 15.3 kW. After grinding the first batch, a power input of 14.2 kW was measured in purge mode. This corresponds to a grinding media filling level, φ_{GM}, of 89%. This means that approx. 347 g of grinding media material has been passed into the product batch due to wear. To raise the power input back to the previous level, 347 g of grinding media of the original fraction should be refilled.

1.3. Effect of the Grinding Media Fill Level

The grinding media filling level doesn't just have a decisive effect on the power input into the grinding chamber of the machine. Investigations by Joost [1], Kwade [2] and Schwedes [3] have shown that the grinding media filling level also has a significant effect on both the specific energy requirement and the grinding media wear. These results have been confirmed in practice through numerous experiences with a wide variety of products (s. Fig. 2 and Fig. 3).

In particular, the effect of the grinding media fill level on grinding media wear is of great importance in the design of processes where minimal contamination of the product is required, e.g., for active pharmaceutical ingredients. In such comminution processes, a grinding media fill level of 60 - 70% is frequently used. Although this reduces the production output and requires a higher specific energy input overall in order to achieve the required product quality, the absolute grinding media wear and thus the contamination of the product can be reduced.

Fig. 2

Comminution results as a function of the specific energy input, for the comminution of fused corundum [1]

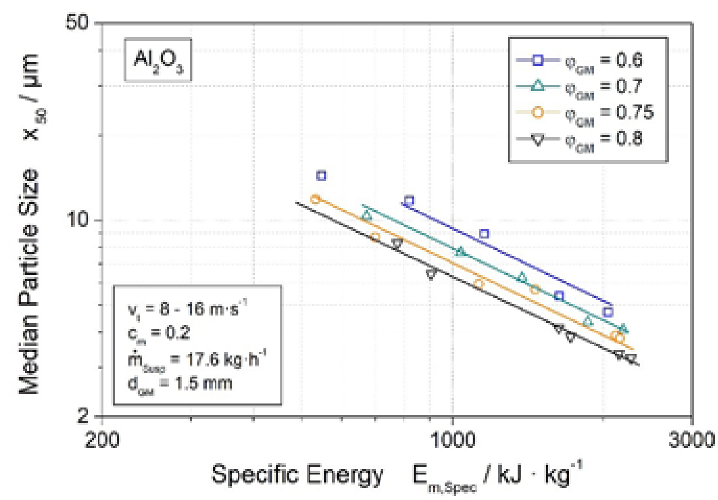
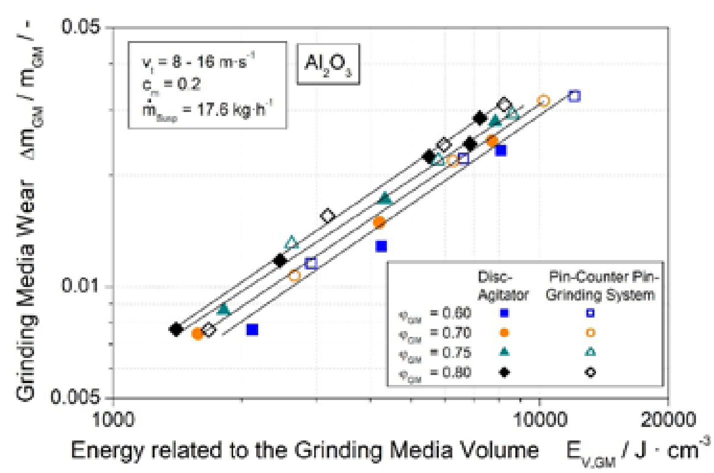


Fig. 3

Grinding media wear during comminution of fused corundum [1], as a function of the energy input related to the grinding chamber volume



2. EFFECT OF THE FRACTION WIDTH OF THE GRINDING MEDIA

Another parameter is the fraction width of the grinding media. Kwade [3, 4] carried out interesting investigations in this regard, in which he used mixtures of glass grinding beads of different sizes for the comminution of calcium carbonate. Grinding media with a diameter of 838 µm and 1 500 µm were used. The pure fractions and mixtures of the grinding media were used in each case (see Fig. 4).

It was shown that comminution with a mixture of different grinding media sizes was actually more energy efficient overall than the pure grinding media fraction with a diameter of 1 500 µm. However, this result only came about because with an initial particle size of the product of less than 10 µm, the smaller grinding media with a diameter of 838 µm were able to transfer sufficient stress energies to the product particles, which led to comminution in almost every stress process. The best result was therefore achieved when the pure fraction of the smaller grinding media was used.

One additional observation when mixing different grinding media fractions is the increase in grinding media wear. This is attributed to the fact that large grinding media collide, stressing the smaller grinding media to some extent. On the other hand, the energy demand is considerably higher to achieve the required quality.

Further, it was shown repeatedly that the use of broader grinding media fractions results in a broader particle size distribution in the product [5], (see Fig. 5).

This finding can be explained by the fact that, due to the higher energy requirement overall, the fines fraction also continues to increase while ineffective collisions between large and small grinding beads also repeatedly occur, particularly when the largest product particles are stressed.

Fig. 4

Comminution result as a function of specific energy input for the comminution of calcium carbonate with mixtures of grinding media of different sizes in different ratios [3]

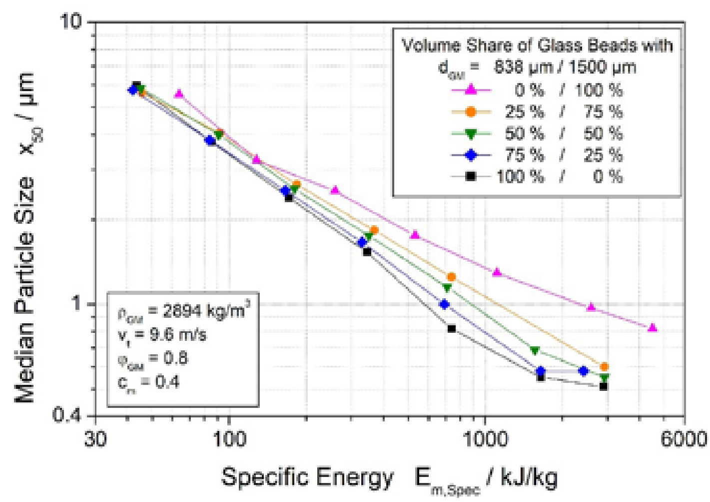
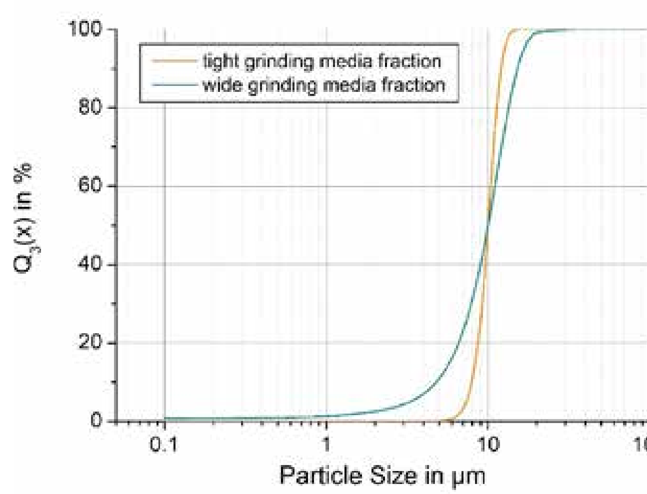


Fig. 5

Effect of the grinding media fraction width on the particle size distribution (qualitative)



The result is that, at the same time, with the same average particle size, the proportion of coarser particles in the entire system is also determined, such that in many cases, the particle size distribution, i.e., the fraction width of the grinding media charge, has a direct effect on the product quality.

2.1. Exchange or Screening of the Grinding Media Charge

In practice, because of grinding media wear and refilling with grinding media of the narrowest possible original grinding media fraction, it is usually impossible to avoid the occurrence of a „natural fraction width" of the grinding media. Only under very special conditions, for extremely expensive products or processes for the manufacture of pharmaceutical products, are the grinding media changed after each product batch.

The grinding media are normally used as long as possible, since they represent a significant cost factor. If one wants to estimate as accurately as possible the point in time at which a maintenance interval with grinding media exchange makes sense, one should first develop an idea of the evolution of the grinding media size in the grinding chamber.

To this end, the following consideration was made:

The grinding media volume is calculated:

$$V_{GM} = \frac{d_{GM}^3 \cdot \pi}{6}$$

The volume of grinding media that has become smaller through surface wear is described by:

$$V_{GM,reduced} = \frac{d_{GM}^3 \cdot \pi}{6} \cdot (1 - Wear)$$

$$d_{GM,reduced} = \sqrt[3]{\frac{d_{GM}^3 \cdot \pi}{6} \cdot (1 - Wear) \cdot \frac{6}{\pi}}$$

If the diameter of the grinding media that has been reduced in size due to wear is considered with respect to the diameter of the original grinding media, the following correlation is obtained:

$$\frac{d_{GM,reduced}}{d_{GM}} = \frac{\sqrt[3]{\frac{d_{GM}^3 \cdot \pi}{6} \cdot (1 - Wear) \cdot \frac{6}{\pi}}}{d_{GM}}$$

$$\frac{d_{GM,reduced}}{d_{GM}} = \sqrt[3]{(1 - Wear)}$$

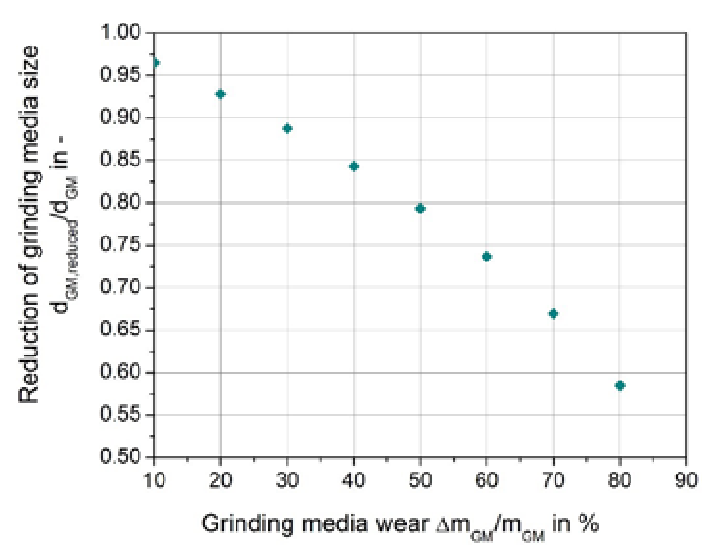
Fig. 6 shows this correlation plotted as a function of the percentage of grinding media wear. This consideration illustrates that even after a total of 50% grinding media wear over the entire process period, the grinding media still have a diameter corresponding to 79.4% of the original size, assuming uniform wear occurring exclusively on the surface of the grinding media.

This requirement is only fulfilled if grinding media breakage can be ruled out. Replacement of the grinding media fill or screening of the grinding media fill is necessary if there is a risk that grinding media that are too small may clog and block the separation screen or even pass through it. Another aspect is the steepness of the particle size distribution.

Through regular measurement of the grinding media wear and conscientious documentation of the refill quantity with grinding media of the original fraction, a very good prognosis can be made as to when a maintenance visit for grinding media replacement, during which the machine must be opened, should take place.

Fig. 6

Evolution of the grinding media size as a function of wear



3. EFFECT OF THE GRINDING MEDIA QUALITY

Regardless of the application, grinding media that are used for grinding in agitator bead mills must meet special requirements. These requirements are essentially:

- constant microstructural properties
- the highest density possible
- high hardness, low elasticity
- high abrasion and wear resistance
- corrosion resistance
- high fracture toughness

Ceramic materials such as zirconia or alumina are particularly suited to fulfilling these requirements.

The quality of the grinding media essentially impacts the grinding media wear and the associated product contamination. However, grinding media of inferior quality can also be found on the market, with which there is a risk of grinding media breakage during operation of the agitator bead mill. If grinding media break during operation, this leads to clogging of the screen in the grinding media separation system. In the worst case, fragments can penetrate into the mechanical seal and cause serious damage there. Any grinding media breakage can be associated with downtimes, loss of production and frequently with the need for additional spare parts.

Another quality criterion for grinding media is their surface finish. High-quality grinding media have a smooth surface. Grinding media that are supplied with a rough surface show a significantly higher level of wear and tear in the first few hours of operation, which can then decrease with increasing operating time.

NETZSCH-*Beads*® are the right grinding media for every application. NETZSCH-*Beads*® are purchased from manufacturers of high-quality grinding media and branded as NETZSCH-*Beads*® after internal quality control. The grinding media are subject to strict quality criteria.

Since the grinding media are the real tools in the comminution process, guarantees and performance commitments for the mills can only be given if it is ensured that the grinding media employed have the appropriate quality and wear resistance.

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