SEPR
Ceramic beads and powders department

# Dispersion in a pressurized mill with electrofused ceramic beads

Christophe BERT, Bruno ROPERS, Emmanuel SERTAIN – SEPR

This paper presents the results of a study conducted at the SEPR Research Center.

The milling or dispersion of pigments is an important step in the manufacturing of paints, inks and dyes.

This study shows the influence of operating variables on milling efficiency and material wear.

SAINT-GOBAIN
INDUSTRIAL CERAMICS

# 1. MATERIALS AND METHODS

## 1.1. Mill

The results presented were obtained using a horizontal laboratory bead mill consisting of a 1.4-litre stainless steel chamber fitted with a shaft and hardened steel discs (80 mm diameter). The separation system at the mill outlet is set at 0.2 mm.

Materials are fed into the mill by a peristaltic pump with variable flow.

## 1.2. Suspension

The test suspension was a paint base (density: 1.12) containing a siliceous mineral compound (cellite powder) at 37wt% concentration in an organic phase. The mean diameter of the mineral particles was approximately 7 µm. These particles tend to form agglomerates. The suspension was prepared in an agitated vessel using a pulser with peripheral teeth. Before dispersion, the viscosity of the suspension was around 2000 cP.

## 1.3. Experimental conditions

The study focused on five variables: residence time in the mill, disc tip speed, fill ratio of the milling chamber, bead size and type of beads (ER 120 electrofused ceramic or glass). Each of these variables can be controlled on an industrial scale.

The influence of each variable was systematically studied while the other parameters were kept constant as control values. In Table 1 the control values are shown in boldface type.

| | | |
|---|---|---|
| Residence time | Flow rate (l/h): | 3 **6** 9 |
| | Number of passes (s): | 1 **2** 3 |
| Disc tip speed (m/s) | | 8 **10** 14 |
| Fill ratio (%) | | 71.4 78.6 **85.7** 92.9 |
| Bead size (mm) | | 0.6/0.8 0.8/1.0 **0.8/1.25**<br>1.0/1.25 1.25/1.6 1.6/2.0 |
| Type of beads | | **ER 120 electrofused ceramic**<br>soda-lime glass |

**Table 1:** Operating parameters and control values.

The influence of each variable is assessed using three meaningful parameters for this type of operation: degree of dispersion, mill wear and bead wear.

The degree of dispersion was evaluated using a NORTH gauge, well known to operators as a device permitting rapid and reproducible measurements. Although simplistic and affording only moderate precision, this method allows the evaluation of the size of the coarsest agglomerates in the suspension, which could be visible in the paint film. Furthermore, it does not require any preparatory steps (e.g., dilution) which could falsify the measurements.

The degree of dispersion (fineness) is evaluated by taking successive samples at the mill outlet, at regular intervals. Several measurements are taken systematically; the mean value of the measurements is the one reported. In the following paragraphs, they are expressed in micrometers (µm) based on the conversion scale with the North fineness given in Table 2.

| NORTH gauge reading | 10 | 9 | 8 | 7 | 6 | 5 | 4 | 3 | 2 | 1 | 0 |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Size of the agglomerates (µm) | 0 | 10.2 | 20.3 | 30.5 | 40.6 | 50.8 | 61.0 | 71.1 | 81.3 | 91.4 | 101.6 |

**Table 2:** Conversion scale: NORTH fineness/micrometers

Mill wear is characterized by the weight loss of the steel discs. The utilization time is taken into consideration, so mill wear is expressed as a percentage of original weight per unit of time.

Similarly, bead wear is expressed as weight loss per unit of time.

# 2. EXPERIMENTAL RESULTS

## 2.1. Influence of flow rate and number of passes

The residence time of the agglomerates of elementary particles in the mill depends on the flow rate, the number of passes and the mill chamber geometry. This geometry not only depends on the shaft and disc volume but also on the grinding media.

The influence of residence time on dispersion efficiency can be studied by varying the flow rate and/or the number of passes in the mill while keeping the bead load constant.

Under these conditions, a residence time scale can be defined on the basis of standard utilization times. The unit residence time has been set at a flow rate of 6 litres/hour and 2 passes. For example, this is the residence time obtained with a flow rate of 3 litres/hour and 1 pass or with 9 litres/hour and 3 passes.

### Comments:

The choice of an arbitrary residence time scale is rendered necessary by the complexity of the flow pattern inside the mill. A simple calculation of the ratio of free volume (net volume of the chamber minus the volume of the bead) to flow rate would very roughly describe this complex phenomenon. This ratio, or ideal filling time, like a piston effect, is generally different from the mean residence time which involves a statistical distribution of residence times.

The results obtained are shown in Table 3, where the corresponding residence time "t" and the size "T" of the coarsest agglomerates at the end of the operation are indicated for each combination of the flow rate and number of passes. Figure 1 shows the variation of "T" as a function of "t".

As expected, when the residence time increases, the size of the agglomerates decreases asymptotically at high "t" values, illustrating the growing difficulty of breaking up agglomerates, the size of which is progressively reduced as the number of the elementary particles that constitute them decreases (see Figure 1). The apparent size limit of the coarsest agglomerates at the end of the treatment was around 15 µm compared to the mean diameter of the siliceous particles at 7 µm. Considering the spread of the size of the elementary particles, it can be assumed that optimum dispersion has been reached at this stage.

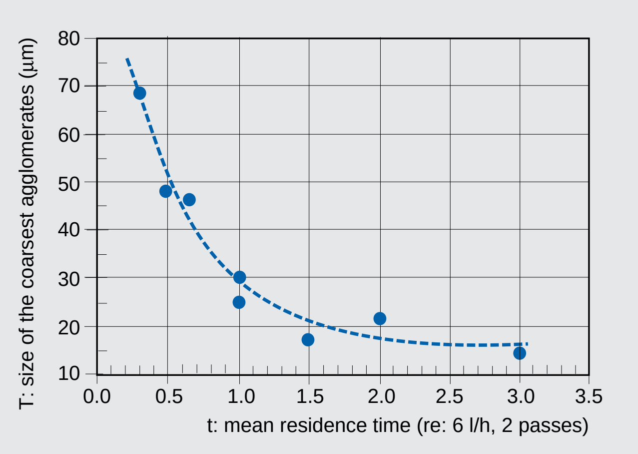


**Figure 1: Influence of residence time on dispersion efficiency.**

Moreover, Table 3 shows that it can be more effective to increase the number of passes than to resort to lower flow rates. It can be seen that for the same residence time (t = 1), better fineness is obtained with a flow rate of 6 litres/hour and 2 passes or 9 litres/hour and 3 passes than with a flow rate of 3 litres/hour and 1 pass. Similarly, the combination of 6 litres/hour and 3 passes (t = 1.5) leads to fineness superior to that observed with the combination of 3 litres/hour and 2 passes (t = 2). This behaviour cannot be attributed solely to the imprecision of the North gauge measurements. It may be due to the homogenizing obtained that results from increasing the number of passes. The probability for agglomerates to follow a preferential "calm" path and arrive intact at the mill outlet decreases when two passes are used. This phenomenon can also be attributed to the wetting effect of the particles during the first pass.

| Flow rate (l/h) | | Number of passes | | |
|---|---|---|---|---|
| | | 1 | 2 | 3 |
| 9 | **T =** | **69** | **47** | **25** |
| | t = | 0.33 | 0.66 | 1 |
| 6 | **T =** | **48** | **25** | **17** |
| | t = | 0.5 | 1 | 1.5 |
| 3 | **T =** | **30** | **21** | **15** |
| | t = | 1 | 2 | 3 |

**Table 3: Size T of the agglomerates (µm) for each combination of flow rate and number of passes.**

For a given product quantity being treated, bead and disc wear depend only slightly on residence time.

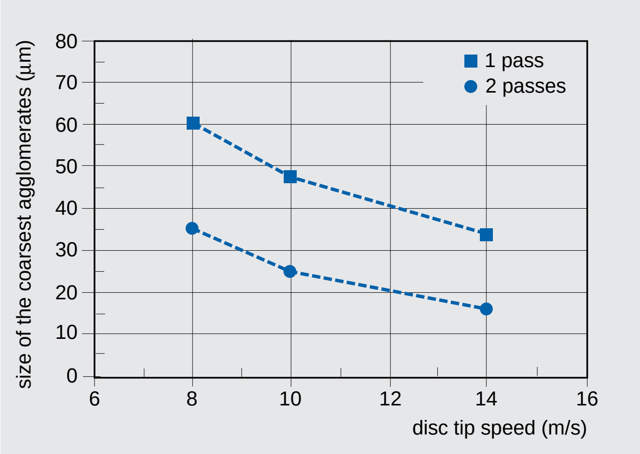


**Figure 2:** Influence of the disc rotation speed on agglomerate fineness.

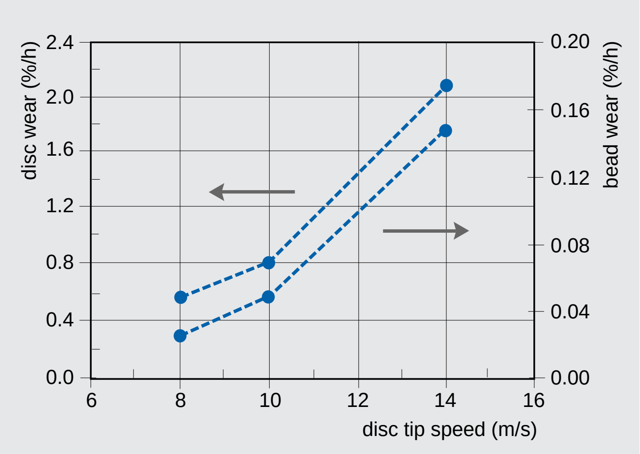


**Figure 3:** Influence of disc rotation speed on bead and mill wear.

## 2.2. Influence of disc rotation speed

Dispersion efficiency is improved by increasing the rotation speed of the shaft and discs as more energy (electrical and then mechanical) is applied to the system (see Figure 2).

Nevertheless, bead and disc wear is sensitive to the rotation speed (see Figure 3). Bead wear is multiplied by 6 when speed varies from 6 m/s to 14 m/s. Disc wear also increases significantly. Although it is difficult to develop the analysis further due to the limited number of experimental measurements, the results seem to be in agreement with studies describing the wear of moving particles (e.g.: abrasives) by the exponential speed law $V^n$. The exponent value "n" is close to 3 for disc wear and 2 for bead wear, close to those currently given in the literature (usually between 1.7 and 2.7).

It is noteworthy that the ratio of wear observed during these tests is representative in relative terms, but not in absolute value, of that encountered in industrial production, as wear is intensified in small laboratory mills.

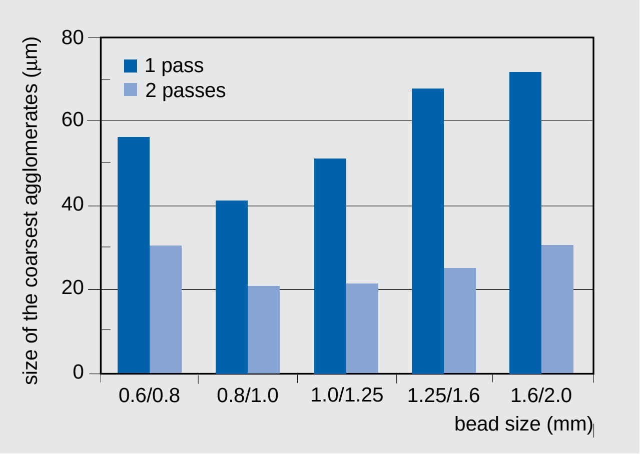


**Figure 4:** Influence of bead size on dispersion efficiency.

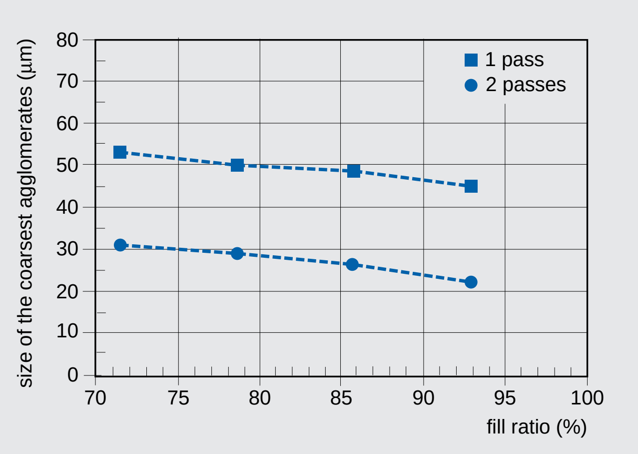


**Figure 5**: Influence of fill ratio on agglomerate fineness.

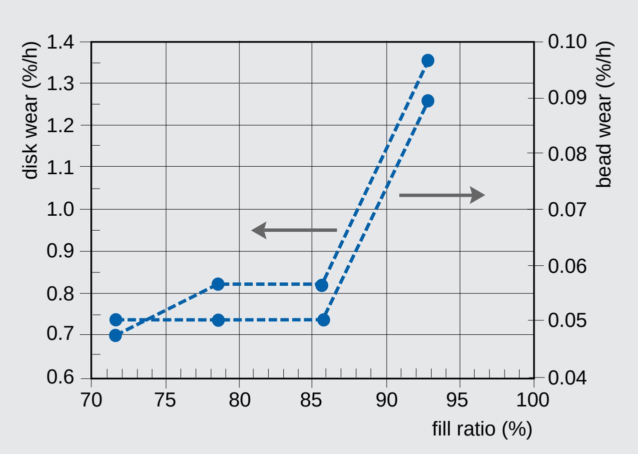


**Figure 6**: Influence of fill ratio on bead and mill wear.

## 2.3. Influence of bead size

The histograms in Figure 4 show the results obtained by varying the size of the ceramic beads, all other parameters being equal. These results can be easily interpreted considering the following phenomena:

- When bead size decreases, the number of beads per unit of volume increases. This greater number of contact points enhances the dispersion.

- However, when the beads are too small, the impact energy is no longer sufficient to break up agglomerates, and the dispersion efficiency is limited.

The bead size limit depends on formulation characteristics including viscosity and cohesion of the agglomerates.

Under our test conditions, the optimal fineness was obtained with a bead size of 0.8/1.0 mm (see Figure 4), as a result of these two opposing factors, i.e. contact points versus particle size.

The influence of bead size is more marked for a single pass than for several passes. Both bead and mill wear tend to decrease when the bead size decreases.

## 2.4. Influence of fill ratio

The fill ratio is an essential parameter which must be well controlled.

While an increase in fill ratio improves dispersion efficiency (see Figure 5), it significantly influences the mill chamber, disc and bead wear.

This point is illustrated in Figure 6. The material wear (beads and mill parts) increases with the fill ratio, with an abrupt slope change for a fill ratio greater than 85%. Under our experimental conditions, this bead fill ratio of approximately 85% seemed to offer a good compromise between dispersion efficiency and material wear.

If a conventional horizontal mill is used, fill ratio should not exceed 85%. Mill manufacturers frequently recommend fill ratios between 80% and 85%.

## 2.5. Influence of bead type

ER 120 (68.5% $ZrO_2$, 31% $SiO_2$) ceramic beads were compared with soda-lime glass beads (65% $SiO_2$, 10% $Na_2O$, 6% $K_2O$, 6% CaO, 6% BaO) of the same size (0.8/1.25 mm). The comparison criteria were dispersion efficiency and mill (discs) and bead wear.

| | Size of the coarsest agglomerates (µm) | |
|---|---|---|
| | glass beads | ER 120 ceramic beads |
| 1 pass | 59 | 48 |
| 2 passes | 35 | 25 |

**Table 4: Comparison of fineness obtained with ER 120 and glass beads.**

A 20 to 30% increase in the fineness of the agglomerates was observed when ceramic beads were used (see Table 4). This significant improvement is associated with the difference in density between the two types of beads (see Table 5). Ceramic beads, which have a higher density, develop more kinetic energy than glass beads, thus providing improved dispersion efficiency.

| | glass beads | ER 120 ceramic beads |
|---|---|---|
| density | 2.6 | 3.8 |
| bulk weight (kg/l) | 1.5 | 2.3 |

**Table 5: Comparison of ER 120 and glass bead densities.**

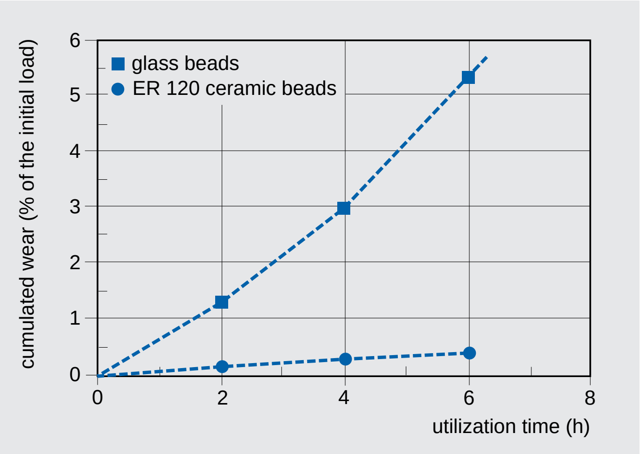
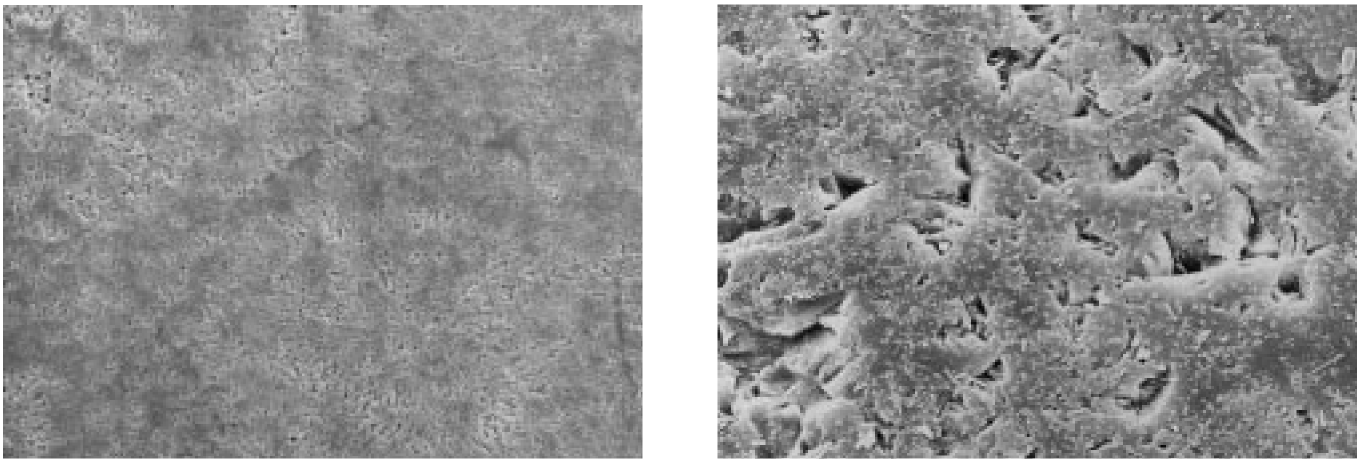


**Figure 7**: comparative wear of ceramic and glass beads.

Observations of material wear are also highly favourable to ceramic beads: the discs showed less wear when ER 120 beads were used (0.82%/h compared to 0.92%/h, under these experimental conditions). This reduced wear, observed in spite of the higher density, must be attributed to the surface condition of the electrofused ceramic beads. ER 120's smooth surface is progressively polished during use. Electrofused ceramic beads are less abrasive than glass beads, the surface of which deteriorates during use. Photographs 1 and 2 compare the surface of glass beads and ER 120 ceramic beads after use under the same conditions.

Moreover, shattering of glass beads leads to faster wear than that observed with electrofused ceramic beads. After two hours of milling, the wear rate for glass beads is 10 times higher (wt%/hour). This is due to the cohesion of the ceramic structure, which endows ER 120 with high toughness and mechanical strength. Figure 7 presents the cumulative wear of ceramic beads and glass beads as a function of time. The wear of ceramic beads increases almost linearly while the wear of glass beads continues to accelerate.

Photo 1 — Photo 2



ER 120 — 10 µm

Glass — 10 µm

**Photos 1 and 2:** Surface condition of ceramic beads (1) and soda lime glass beads (2), after the same use (Scanning Electronic Microscope).

## CONCLUSION

Tests were performed using a 1.4-litre horizontal laboratory mill and a paint base containing siliceous mineral at 37wt% concentration in an organic solvent with 2000 cP viscosity. The following tendencies were observed:

◆ At identical occupation times of the mill, a multiple pass / high flow rate system can, in some cases, provide more effective dispersion than a single pass/low flow rate system.

◆ The efficiency of dispersion increases with the shaft rotation speed and fill ratio, but so do bead and mill wear. An optimum fill ratio was determined under our test conditions.

◆ Decreasing the bead size causes the effectiveness of dispersion to increase up to the point where the energy of the impacts between beads and agglomerates is not sufficient to separate the elementary particles.

◆ ER 120 ceramic beads allow improved efficiency and reduced wear and therefore offer an obvious advantage over glass beads.

Although there is always some uncertainty involved in extrapolating laboratory results to a larger scale, the broad phenomena highlighted by this study reflect and are consistent with industrial experience.

SAINT-GOBAIN
ZIRPRO

B.P. 25 -Villa Park - 84131 LE PONTET Cedex - FRANCE
Tel. : +33 (0)4 90 39 99 14 - Fax : +33 (0)4 90 39 99 98