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Of course our dispersion systems are equipped with integrated safety devices according to EC machinery directive 2006/42/EG.
Innovative dispersion and fine grinding systems for laboratory, pilot plant and production made in Germany

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Please visit us in our excellently equipped laboratory and pilot plant for a personal product demonstration with your own products. Our skilled engineers will be pleased to show you the impressive range of services of our patented dispersing and grinding systems DISPERMAT\textsuperscript{®} and TORUSMILL\textsuperscript{®}.

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Dispersing in Laboratory, Pilot Plant and Production with DISPERMAT® Dissolvers

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The most frequent application of high speed dispersion is to incorporate extremely fine solid particles into fluids, to produce colloidal suspensions.

Colloidal suspensions are characterised by their behaviour that the finely divided small particles do not settle under the force of gravity. A sequence of related steps take place during the dispersing process.

These are:

- the wetting of the surface of the solid particles by the fluid components of the millbase
- the mechanical breakdown of associated particles leading to smaller particles (agglomerates and aggregates)
- the smaller particles generated during the dispersion are stabilised, preventing renewed association (flocculation).

Special interaction between the solid particles and the fluid components of the millbase determine their wetting and resistance to flocculation.
THE DOUGHNUT EFFECT

The best dispersion results with a DISPERMAT® are obtained when the geometry of the dispersion container, the diameter, the peripheral velocity and the height of the dissolver disc above the bottom of the vessel as well as rheological millbase properties are matched to one another. After adding pigments and fillers to the resin solution, the millbase is brought into a laminar rolling flow pattern by increasing the speed of the shaft until no standing material can be seen at the wall of the container. At the correct speed, a channel begins to form around the shaft and a part of the dissolver disc becomes visible. At this point, the millbase will form a doughnut-like flow pattern.

The doughnut-like flow pattern is a signal that the maximum mechanical power possible is being transferred into the millbase and furthermore that the millbase is being agitated so that all the agglomerates will eventually reach the dissolver disc.

The doughnut effect develops because the millbase is accelerated outwards from the tip of the dissolver disc. When it hits the wall of the vessel, the stream is divided into two parts. The one going downwards flows back to the middle of the dissolver disc along the bottom of the dispersion vessel and rises up to hit the disc once again. The second part flowing upwards has the same circular path, which is limited in by the force of gravity and the rheological properties of the millbase.

The flow pattern of the doughnut effect is greatly influenced by the amount of pigment and filler in the millbase. When the solids content is not high enough, the viscosity tends to be too low. This leads to splashing and generation of bubbles during dispersion.

In addition, the mechanical power input is limited and the deagglomerating capability of the dissolver disc is affected. Conversely, if the solids content is too high, then the viscosity will be too high for the doughnut flow pattern to develop.

The flow of the millbase may also be hindered by a yield value of viscosity. This will result in a tearing action of the dissolver disc, which may at times even turn without having contact with the millbase.
THE DISPERSING EFFECT OF THE DISSOLVER DISC ON AGGLOMERATES

When the vanes of the disc are moved through the millbase at a high velocity, areas of higher and lower pressure are generated in front of and behind the vane. The alternating stress acting on the agglomerates in these areas facilitate their dispersion. In addition to this, a smashing impact should be considered for larger agglomerates being hit by the edges and the surfaces of the vanes. However, a considerable share of the total dispersion work takes place at the surface of the dissolver disc. Due to the fast movement of the blade, a gradient of shear builds up on these surfaces in which the dispersion takes place.

The shear stress which acts particularly between the lower disc surface and the bottom of the container largely depends upon the distance between the two. The efficiency of the shear gradient may be enhanced by decreasing this separation since the shear rate within the gap is increased and since a higher rotational speed may be chosen due to the fact, that the change from laminar to turbulent flow takes place at higher rotational speeds.

When higher speeds are used, more mechanical power is introduced into the millbase. The best dispersion results are obtained with the highest possible mechanical power input, as long as the doughnut flow pattern (laminar flow) is maintained.

The mechanical power is a product of rotational speed and momentum (torque) of the shaft ($\pi = 3,141...$).

$$P = 2 \pi n M$$

Circumference velocity related to the rotational speed of the shaft

Geometry within the dispersion container

- $D =$ Container diameter
- $d =$ Dissolver disc diameter
- $a =$ Distance between dissolver disc and bottom of container
- $f =$ Amount of millbase

The coloured area indicates the optimum range of circumference velocity between 18 – 25 m/s
In practice, a simple procedure has proven to yield satisfactory results:
First the liquid component is put into the dispersion container.
Then, under moderate agitation by the dissolver disc, pigments and fillers are added slowly. The dissolver disc speed can then be increased until the doughnut effect is detected at a higher rpm (circumference velocities of approximately 18-25 m/s).

After premixing, the walls of the dispersion container and the shaft should be cleaned removed adhering millbase. Then the dispersion is carried out at high peripheral velocities that guarantee the formation of the doughnut effect.

At this stage, the capability of the DISPERMAT® to transfer high mechanical power into the millbase should be exploited. One must not be afraid to use high rotational speeds. If e.g. an dissolver disc of 25 mm diameter is used, the DISPERMAT® must be run at a rotational speed of 15,000 rpm in order to obtain peripheral velocities of 20 m/s. The final dispersion result is normally reached after 10 to 15 minutes.

Use of the dissolver for a longer period of time is not likely to improve the result. Sample analysis shows that further deagglomeration does not take place. The particle size of demanding or difficult products can be reduced further using a DISPERMAT® bead mill or basket mill.
An important fact is that the dispersion results obtained with a DISPERMAT® can be scaled up to a production size dissolver. It was mentioned earlier that the dispersion depends upon the rate with which the agglomerates are transported into the zones of shear and on the mechanical power which is transferred into the millbase. The mechanical power is the parameter that limits the maximum degree of dispersion which can be achieved. The rate at which the transportation of the agglomerates into the vicinity of the dissolver disc takes place, determines the time necessary to reach the optimum dispersion result.

The deagglomeration process mainly takes place within the area of shear which surrounds the dissolver disc. The most effective shearing conditions are found at the tip of the dissolver disc, as this part is moved through the millbase at the highest speed.

It is for this reason, that the tip speed (peripheral velocity) is to be considered as the key parameter for scaling up laboratory results to production. This statement refers to the maximum achievable degree of dispersion and not to the time necessary to obtain it. The DISPERMAT® will normally be faster in dispersing than a production scale machine, as the distance of the agglomerates must cover to reach the disc are shorter than in larger equipment.

Exact correlation between the dispersion result with a DISPERMAT® and a larger dissolver will naturally also depend upon comparable temperature conditions. For temperature control, the use of a double wall temperature control container is recommended.

For a laboratory dissolver to reach the peripheral velocities necessary for dispersion, it must be able to run at high speeds with utmost accuracy and reproducibility. When using the dissolver discs of different diameters, the circumference velocities may easily be calculated by the following formula.

\[
v = \frac{\pi \cdot d \cdot n}{60}
\]

where:
- \(v\) = circumference velocity m/s
- \(\pi\) = 3.141...
- \(d\) = diameter of the dissolver disc in m
- \(n\) = revolutions of shaft in rpm
CIRCUMFERENCE VELOCITY RELATED TO THE ROTATIONAL SPEED OF THE SHAFT FOR VARIOUS DISSOLVER DISC DIAMETERS

Please find here three useful examples of the relationship of the circumference velocity and the shaft speed and the disc diameter. The coloured area indicates the optimum range of circumference velocity between 18 - 25 m/s.

**Example for a millbase volume of 100 ml**
- Dissolver disc- Ø in mm
- Container capacity: 250 ml
- Inner-Ø of container: 65 mm
- Container height: 85 mm
- Dissolver disc diameter: 30 mm
- Shaft speed: 11500 - 16000 rpm
- Peripheral velocity of disc: 18 - 25 m/sec

**Example for a millbase volume of 2500 ml**
- Dissolver disc- Ø in mm
- Container capacity: 5000 ml
- Inner-Ø of container: 180 mm
- Container height: 200 mm
- Dissolver disc diameter: 80 mm
- Shaft speed: 4300 - 6000 rpm
- Peripheral velocity of disc: 18 - 25 m/sec

**Example for a millbase volume of 30 litres**
- Dissolver disc- Ø in mm
- Container capacity: 30 litres
- Inner-Ø of container: 440 mm
- Container height: 440 mm
- Dissolver disc diameter: 200 mm
- Shaft speed: 1700 - 2400 rpm
- Peripheral velocity of disc: 18 - 25 m/sec

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container capacity 250 ml
inner-Ø of container 65 mm
container height 85 mm
dissolver disc diameter 30 mm
shaft speed 11500 - 16000 rpm
peripheral velocity of disc 18 – 25 m/sec

container capacity 5000 ml
inner-Ø of container 180 mm
container height 200 mm
dissolver disc diameter 80 mm
shaft speed 4300 - 6000 rpm
peripheral velocity of disc 18 – 25 m/sec

container capacity 30 litres
inner-Ø of container 440 mm
container height 440 mm
dissolver disc diameter 200 mm
shaft speed 1700 – 2400 rpm
peripheral velocity of disc 18 – 25 m/sec
Duration of the Dispersion Operation
The quality of formulations dispersed using a DISPERMAT® with a sufficient high speed generally reaches its final value after a short period of time (approximately 10 -15 minutes). Increasing the dispersion time to more than 20 minutes does not normally lead to improved results.

Doughnut Effect
The doughnut flow pattern should be maintained during the course of the whole dispersion.

Shaft Speed
The mechanical power input should be optimised by using the highest possible rotational speed and thereby the greatest peripheral velocity, without destroying the doughnut flow pattern.

Geometrical Considerations
The distance between the dissolver disc and the bottom of the vessel can be changed to obtain better results and to make higher rotational speeds possible.

Amount of Millbase
Better flow characteristics may be achieved by using more or less millbase in the container.

Dissolver Disc
The use of smaller or larger dissolver disc may lead to better results.

Pigment and Filler Concentration
A high viscosity millbase of tacky consistency with dilatant flow is recommended. This may be obtained by increasing the percentage of solids, but without destroying the doughnut flow pattern.

Flocculation
Does flocculation take place after dispersion? If so, check additives.

Vacuum
At a dispersion under vacuum high-viscous products can be produced to the greatest possible extend without air bubble inclusions.

Temperature
When dispersing, high energy transfer into the millbase will lead to an increase in temperature. In many cases this destroys the flow characteristics of the formulation. In addition, thermally sensitive paint ingredients may be harmed. Using a water cooled vessel will solve the problem.

Raw materials
Partial re-formulation of the paint using more suitable resins, pigments, fillers or additives: It should be kept in mind, that the DISPERMAT® is a dispersion device and not a piece of milling machinery. Therefore it is incapable of grinding primary particles down to a smaller size.

Additives
By adding suitable additives the result of the dispersion process can be strongly improved.

STEPS TO IMPROVE DISPERSION RESULTS
In cases where the quality of dispersion does not meet the required standard, the following parameters should be checked:
To obtain excellent dispersion results, the millbase must exhibit certain rheological properties. Unfortunately, the flow behaviour of a millbase may not be expressed by one single parameter, such as the apparent viscosity. Viscosity is the measure of the internal friction of a fluid, and is a product specific Constant Value which is defined as the quotient of shear stress ($\tau$) and shear rate ($D$).

Only Newtonian fluids retain a constant viscosity and are independent of variations in shear rate (that is i.e. water, mineral oil, etc.). All other substances which have a viscosity which is dependent on shear rate are classified as Non-Newtonian and are more commonly found than Newtonian liquids.

**Newtonian fluid:**
Is a fluid whose viscosity is independent of the shear rate at which it is measured.

**Plastic substance:**
Viscosity decreases when the shear rate velocity gradient in increased.

**Pseudoplastic substance:**
Viscosity decreases when the shear rate is increased.

**Dilatant substance:**
Viscosity increases when the shear rate is increased.

To characterise the behaviour of millbases, information is needed concerning apparent viscosity, plastic behaviour, yield stress, thixotropy, rheopexy, dilantancy.

- **Low viscous** $\mu < 500$ mPs
- **Medium viscous** $\mu = 500 - 5000$ mPs
- **High viscous** $\mu > 5000$ mPs

Viscosity $\mu = \frac{\text{shear stress } \tau}{\text{shear rate } D}$ [Pa · s]
Fine grinding in Laboratory, Pilot Plant and Production with DISPERMAT® and TORUSMILL®

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In many technical processes it is necessary to divide solid material into fine particles and distribute them evenly within a liquid carrier. This process is generally known as “dispersion”.

During dispersion, the adhesive forces that act between the extremely fine solid matter powder particles must be overcome. When the requirements on fineness are high or the solid matter is difficult to disperse, a dispersion with the dissolver is often insufficient.

Due to their ability to process a wide variety of solid matters that are difficult to disperse, high speed bead mills have gained particular acceptance.

In the dispersion process, three partial steps run in parallel:

1. The wetting of the surface of the solid matter to be processed, by liquid components of the millbase.
2. The mechanical division of agglomerates into smaller agglomerates and primary particles.
3. The stabilisation of primary particles, agglomerates and aggregates against renewed attraction (= flocculation).

While the stabilisation against flocculation is primarily a property of a colloid-chemical system, which depends on the interaction of the liquid components (in varnishes for instance: binders, solvents and additives) with the solid matter parts (e.g. pigments and filters) or on that of the solid particles with each other, the dispersion machinery used plays a vital part in the mechanical division and more important aids the wetting process.

The actual dispersion system in a bead mill consists of a milling chamber and an agitator; the milling chamber is filled with the grinding beads (material e.g. glass, zircon oxide, steel) and the product to be dispersed. In the milling vessel, the grinding medium is kept moving by the agitator, which itself is driven by a motor.

The dispersion process takes place between the grinding beads sliding on each other and between the rotor and/or the vessel sides and the grinding beads.
Just like the dispersion, the mechanical process can be divided into three steps:

- wetting
- mechanical division
- flocculation stabilisation

The step of mechanical division itself can also be separated. To enable the agglomerates to be dispersed, they must:

- get into a dispersing situation, e.g. into the shearing zone between two grinding beads (spatial condition) and
- be stressed enough so that they break (energetic condition).

The mechanical division may be illustrated by comparing it with the attempt to crack a nut with a hammer.

In order to break the shell, the nut must be hit in the first place (spatial condition), but it must also be hit hard enough (energetic condition). For a proper understanding it is important to realise that both conditions - spatial and energetic - must be fulfilled at the same time. Although this model may seem rather trivial, it clearly demonstrates the function of a dispersing machine.

For reasons of simplicity, one should imagine a batch bead mill, filled with a millbase which with progressing state of dispersion illustrates a measurable change of a technical property.

In paints, this may for instance be the colour strength, the gloss, the viscosity or the fineness (to be measured with a Hegman Gauge according to DIN 53203).

Our example uses colour strength. When all operating parameters, grinding bead filling quantity, bead type, speed, cooling etc. are kept constant, the measured colour strength reaches a finite value related to the time of dispersion.

Longer dispersion will not improve the colour strength. Only by increasing the speed it is possible to further increase the colour strength.

The reason for this behaviour is that in a very long dispersion all agglomerates have the opportunity to get into the zones of the maximum shearing effect.

Those that are dispersed under these conditions cause a visible increase in the colour strength. Those that have such a high stability that they are not divided under the conditions of the maximum available shearing effect, are still undispersed. By increasing the speed, zones with stronger shearing effect develop where more stable agglomerates can also be dispersed. Therefore, the colour strength may continue to rise with increased speed.

Only after a sufficiently long dispersion time combined with sufficiently high speeds, can it be expected that all agglomerates are dispersed. Only then the spatial as well as the energetic conditions required for a full dispersion are met. Too low a speed can generally not be compensated by longer dispersion and vice versa.
In principle, two methods can be distinguished in the operation of the DISPERMAT® SL bead mills. Either, the complete millbase is collected after each pass through the bead mill (single or multiple pass), or else the millbase is fed directly back into the supply vessel from the outlet of the milling chamber (re-circulation method).

In the single pass method, the product is filled into a feed vessel and pressed through the milling chamber via continuously adjustable pneumatic transport system or with the feed press.

In the re-circulation method, the product is filled into the feed vessel and repeatedly pumped through the milling chamber with an integrated, continuously adjustable pumping and stirring system.

The method of operation to be chosen depends on the type of task.

Easily dispersible pigments can often be processed with the single pass method, whereas with pigments that are more or difficult to disperse the re-circulation method is more efficient.

Over a period of time the re-circulation method ensures, that every agglomerate will get into a dispersion situation. Here, it has spatial and energetic condition and is dispersed. This means that the re-circulation system is more efficient and economic.
Bead mill DISPERMAT® SL-nano
Basic scientific research has shown that the mechanical power that is transferred into the millbase is closely related to the dispersion result. The mechanical power determines the energy that is transmitted by the agitator via the grinding beads to the product.

The power $P$ is calculated from the speed $n$ of the agitator and the torque $M$ generated on the agitator according to the following equation:

$$P = 2 \pi n M$$

- $P = \text{power} \ [\text{Nm/s} = \text{J/s} = \text{W}]$
- $\pi = 3.141...$
- $n = \text{speed} \ [1/s]$
- $M = \text{torque} \ [\text{Nm}]$

The higher the realised mechanical power input the higher is the energy, brought into the container. It does not matter whether the power input which leads to the existing energy density, is applied with a high speed and low torque or vice versa.

To get all agglomerates at least one time in a zone of highest energy density, which means that if the dispersion condition with longer dispersion time does not change at a given bead filling charge and sufficient long dispersion time, the dispersion result depends only on the amount of the mechanical power.

The torque therefore depends directly on the flow characteristics of the millbase. If the viscosity changes during dispersion at constant speed, the power input changes automatically.
If the viscosity decreases during dispersion, the mechanical power drops, and if it increases, the mechanical power rises. If the formulation is operated under a stronger cooling, the mechanical power input is higher due to the higher viscosity of the mill base. With a lower cooling the power input is correspondingly lower.

This for example, is the reason why dispersion results may literally depend on the season, because in winter, the cooling water may be much colder than in summer!

The DISPERMAT® SL solves this problem by enabling the mechanical power input for dispersion to be pre-set. During dispersion the torque of the rotor is continuously measured and the speed controlled, so that the product of n and M leads to precisely the pre-set mechanical power.

Apart from the agitator geometry and the viscosity of the mill base, the torque transmitted by the shaft onto the mill base also depends on the type, quantity and size of the grinding beads. High bead filling volumes increase the torque on the agitator shaft and also increase the probability that agglomerates come into a spatial dispersion situation.
The relationship between the effects of energy and time enables the dispersion process to be optimised. If the required dispersion result is not achieved, it must first be determined whether this can be changed by increasing the dispersion time.

The power input can be increased with higher speeds. This will normally improve the dispersion. Smaller beads and/or beads with a higher density (e.g., zircon oxide or steel) can also improve the dispersion result.

Further, the bead charge can be increased to about 80%. In order to operate the bead mill economically, dispersion should be made with as much solid matter as possible. If after the dispersion there is some flocculation, a suitable dispersing aid may help. A partial modification of the millbase formulation by using more suitable raw materials can also be made.

**STEPS TO IMPROVE DISPERSION RESULTS**

- increased dispersion time
- increased the power input by increasing the speed
- increased the power input by increasing the torque
- improved cooling
- smaller beads or beads with a higher density
- increased bead charge
- modification of the millbase (e.g., by using additives)
Considering the many influences on the spatial and energetic conditions of dispersion and their difference in various bead mills, it is not surprising to learn that the transfer of the results from one machine to another is not automatically possible. Even if the same bead mill is used, but with different discs, the dwell time distribution of the millbase will be changed and, despite the same number of passes (single pass) or same dispersion time (re-circulation mode) the dispersion result will also change.

Nevertheless, if different bead mills are to be compared with each other, it is generally the case that production machines have less adjustment possibilities. First, the typical result has to be determined on a known millbase.

With this typical result (e.g. fineness), a test series should be made with the DISPERMAT® SL laboratory bead mill with M-control. The impeller speed should be adjusted and the dispersion continued until the result matches that which can be achieved in production. If a DISPERMAT® SL with C-control is available, the tests should be made using mechanical power input.

When comparable results have been achieved, the settings on the DISPERMAT® SL can be used to determine results that are possible in production.

When milling with constant power input, not only can complicated dispersion processes be performed in a reproducible manner, but different dispersions can be compared exactly.

The dispersion results from production machinery are easily repeated with the DISPERMAT® SL and formulations worked out in the laboratory can be transferred into production. With the DISPERMAT® SL, problematic parameters like product temperature, cooling water temperature or rheological behaviour of the mill base, may be ignored as long as they do not reach limits critical for the product.