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Documentation and Processing of Test Data - Error Analysis

Business Unit
GRINDING & DISPERSING

DOCUMENTATION AND PROCESSING OF TEST DATA - ERROR ANALYSIS

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1. INTRODUCTION

Test and laboratory data as well as the results of analyses are the key for successful development of new processes. First of all, it is desirable that the results of tests, regardless of who worked on them, are always documented according to the same criteria so that a representative and reproducible report is created. Furthermore, the report should be laid out in such a way that the results, comments and notes, when read later, provide a clear and complete description of the experimental procedure and the results without additional explanations from the previous processor.

Numerous errors can be avoided in the preparation and performance of experiments, in sampling, sample processing, analysis and documentation. This brief article provides suggestions and ideas on how, for example, the results of tests with agitator bead mills can be documented and stored, and which errors can creep in during the experiment, sampling, characterization and evaluation.

2. TEST PREPARATION

Meticulous preparation is the basic prerequisite for successful performance of experiments. The essential preparatory steps include precise definition of the test objective, analysis of the initial situation (raw material analysis) and preparation of the protocol.

Here, the test objective can be quite different. On the one hand, the investigations to be carried out may be part of a feasibility study, the target of which may be a defined particle size, particle size distribution, color intensity, transparency, gloss or a specific solubility or even a flavor.

On the other hand, the objective of the investigations can also be process optimization or scaling up to a production scale. In this case, additional information is logged or additional measurements are performed.

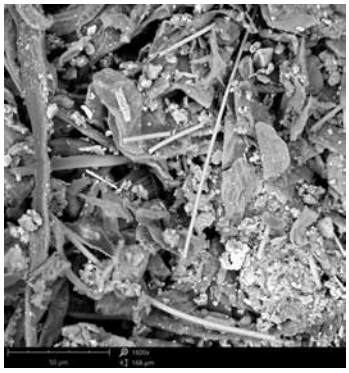
In this protocol, the objective, test setup, materials used and recipe components, definition of the test procedure, people involved and the material pretreatment are documented before the actual test is carried out.

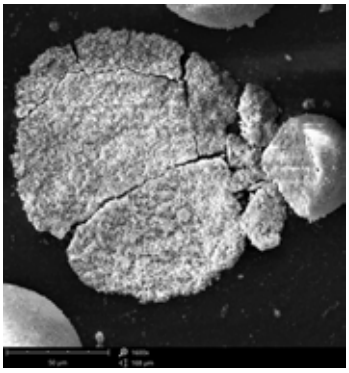
The exact documentation of the test setup with machine components, material design, pump and grinding media used is just as important as the subsequent documentation of test data and results. Before a determination is made as to the grinding system and grinding media to be used, it is helpful to characterize the test material by means of particle size analysis and, if available, by scanning electron microscope (SEM) observation.

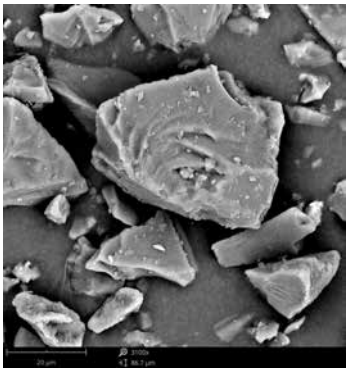
In addition, screening the starting material with a test sieve is often very informative. With particle size analysis or the scanning electron microscope, only very small amounts of sample are characterized. When screening with a test sieve, larger sample quantities can be classified. This is particularly helpful when information about any coarse material that may be present is required.

Fig. 1

REM images of samples with different compositions and structures







Particle size analysis, e.g., by means of laser diffraction techniques, static light scattering or dynamic light scattering techniques, provides no information on composition, morphology, surface structure or particle shape.

Shown in Fig. 1 are three samples of test materials with completely different compositions and structures. While multiple components with very different shapes and different particle sizes are mixed in the material in the image on the left, the image in the center shows a large, broken agglomerate made up of very small primary particles. In contrast, the image on the right shows large primary particles.

Information regarding the composition, particle shape, structure or about the presence of fibrous components are very helpful when selecting the grinding system and operating parameters.

Likewise, the Material Safety Data Sheets (MSDS), must be studied in order to clarify whether special measures concerning safety and environmental aspects are required during testing to ensure that the product suspension is processed with due care.

2.1. Setup of the Test Facility

Before setting up the test facility, various aspects must be clarified:

- selection of a suitable method for predispersion
- selection of the mill type, material design, and operation mode of the mill
- selection of the grinding media type and size
- measurement of the no-load characteristic of the test machine during the test
- selection of the most suitable pump (gear pump, peristaltic pump or diaphragm pump), considering the possible necessity of pulsation damper
- for circulation mode - selection of a suitable agitator in the batch tank.

All of these aspects are paramount for a successful test run. Comprehensive explanations and pointers on process design and optimization are provided in the NETZSCH – Compendium – Comminution and Particle Characterization [1].

When setting up the test facility, special attention should be paid to several points.

First of all, it should be verified that all machines and system components used are clean. If the test preparation is incomplete, the test assembly can be flushed with the dispersion medium used before the test begins. For this purpose, a specific quantity (e.g., 2 kg of liquid) is weighed in and added to the batch container during a test in circulation mode. With the mill switched on, the liquid is then pumped through the entire system in a closed loop. Any residue remaining on machine components or grinding media that may be present can then be removed with the rinsing liquid. At the same time, this method can be used to check whether all pipe transitions and the grinding container are tight. After draining off the flushing liquid, the mass is weighed again. The difference has been left behind in the grinding system. If severe contamination is found, this step can be repeated several times. The quantity remaining in the grinding system must be taken into account when preparing the test batch.
If the system is to be flushed in passage mode prior to a test, it is not necessary to determine the amount of flushing agent that remains in the grinding chamber. In any case, in the subsequent test, sampling is only advisable when a static state has been established. Experience has shown that this occurs after the contents of the grinding chamber have been completely exchanged approx. four times (see chapter Sampling).

For experiments in passage mode, it must be ensured that sufficient test material is available to achieve the static state, since it is only after this occurs that sampling and quality analysis lead to reproducible and useful results.

It is useful to carry out a functional check of the pump and machine as well as any necessary change of sealing fluid before the setup has been completed.

2.2. Laboratory Agitator Bead Mill *LabStar*

The *LabStar* is a true multi-talent. This laboratory machine can be operated as a classic disk agitator bead mill with *Discus*® grinding system as well with a peg grinding system *Zeta*® or *Neos*®. Furthermore, the *LabStar* can be used in different modes of operation like circulation-, passage or even multi pass operations. The mill is available in different material executions such as steel, Nelast and various ceramics. Additional features are the swiveling grinding tank and very user-friendly handling. Thanks to an adapter system it is possible to combine the grinding systems of the *Micro-* & *Mini-Series* with grinding chamber volumes of approx. 100 ml or 200 ml with the *LabStar*. This makes this mill extremely flexible for all tasks in the area of research and development.

The *LabStar* is the smallest agitator bead mill with which the specific energy required for every application can be determined safe and reproducible for reliable scale-up calculations to the production scale based on laboratory tests.

2.3. Filling of Grinding Media

Grinding media should always be filled according to the weight of the grinding media mass, not as a volume. The reason for this is the fact that the exact determination of a bulk volume is very difficult and different people often get very different masses of grinding media when they measure the same volume.

In a simple test in the laboratory, 10 different people were asked to measure a volume of one liter of the same grinding media 10 times in succession. For one person, the deviations in the masses amounted to approx. 6% between the maximum and the minimum weight. When comparing all 100 samples from all test subjects, the maximum difference was approx. 20%, although all samples were measured with the same beaker. It is therefore much more representative to weigh the grinding media mass using the bulk density specified by the manufacturer and pour it into the grinding chamber. Here, it is only necessary to ensure that the balance is correctly tared when the grinding media is weighed.

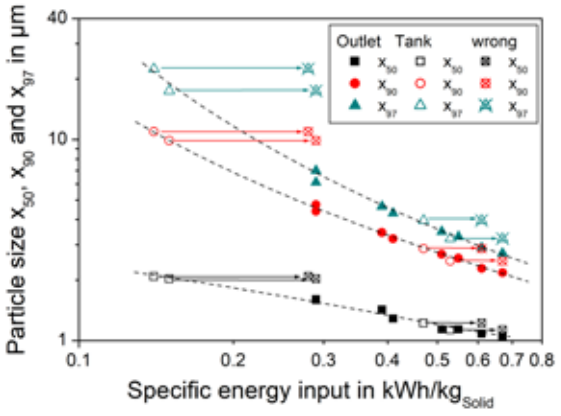
3. SAMPLING

3.1. Sampling in Circulation Mode

For a test in circulation mode, it must be determined where the test sample should be taken, since different specific energy inputs result when sampling at the machine outlet or from the batch tank (see Fig. 2).

Fig. 2

Results from two identical comminution tests



This is illustrated by the following example. If a circulation process is started, suspension is pumped from the batch tank through the mill. When the first product exits the mill, sampling from the tank and at the mill outlet would show that the product quality of the sample taken from the tank has not yet changed, because the product material has not yet passed through the mill, while the quality of the sample from the outlet has already changed. Therefore, there is always a quality difference between the product quality in the tank and at the outlet of the mill, which corresponds to one passage through the mill. Therefore, after reaching the desired product quality at the discharge of the mill, the product can also be filled via a discharge passage without changing the product quality.

The specific energy input must be calculated differently for sampling in the tank (Eqs. (1) and (2)) or at the outlet (Eqs. (3) and (4)). When calculating the specific energy input, it is useful to express the net energy input in terms of the mass of the suspension if the final product is the product suspension. If only the solid is significant as the product, the net energy input is expressed in terms of the mass of the solid when calculating the specific energy.

$$E_{m,Spec} = \frac{E_{M,net}}{m_{Susp}} \quad (1)$$

$$E_{m,Spec} = \frac{E_{M,net}}{m_{Solid}} \quad (2)$$

$$E_{m,Spec} = \frac{E_{M,net}}{m_{Susp}} + \frac{P_{M,net}}{\dot{m}_{Susp}} \quad (3)$$

$$E_{m,Spec} = \frac{E_{M,net}}{m} + \frac{P_{M,net} \cdot \frac{m_{Susp}}{\dot{m}_{Susp}}}{m} \quad (4)$$

where	$E_{m,Spec}$	[Wh/kg]	specific energy input
	$P_{M,net}$	[W]	net power input
	$E_{M,net}$	[Wh]	net grinding energy
	m_{Susp}	[kg]	suspension mass
	m_{Solid}	[kg]	solid mass
	$\dot{m}_{Susp}$	[kg/h]	suspension mass flow

Fig. 2 illustrates what happens during the evaluation if the sample is taken from the batch tank by mistake rather than at the outlet (as intended for this test), but the specific energy is calculated as if the sample had been taken at the mill outlet. The corresponding results have been crossed out in the diagram and marked as „incorrect“.

However, if the specific energy input is calculated according to the sampling location, the results fit into the expected curve (open symbols).

3.2. Sampling during Processes in Passage Mode

In agitator bead mills, the continuously flowing product suspension is subject to a mixing process which causes portions of the product to remain in the grinding chamber for different lengths of time. This results in a so-called residence time distribution of the product suspension and thus differing degrees and frequencies at which all particles are stressed as well.

If the transport behavior of the solid and liquid phases is similar, i.e., if the particles are sufficiently small, the residence time distribution of the product suspension can be measured by pulsed injection of a salt solution at the grinding chamber inlet and measurement of the impulse response at the grinding chamber outlet using a conductivity meter.

Shortly before entering the grinding chamber, pulses of saline solution were injected into the feed suspension. The impulse response was recorded in the discharge pipe with a conductivity meter. Taking into account the residence time in the inlet and outlet pipes, the residence time density distribution E(Θ) in the grinding chamber can be determined directly from the measured conductivity by normalization.

Fig. 3 shows a residence time distribution measured in this way for a typical operating setting. The density distribution E (Θ) is shown as a function of the dimensionless time Θ. To calculate the dimensionless time Θ, the real process duration is related to the ideal filling time or the theoretical dwell time. Here, the theoretical dwell time indicates how long a suspension component would stay in the grinding chamber if the suspension were to pass the grinding chamber in an ideal plug flow. The ideal filling time tF results as follows:

$$t_F = \frac{V_{GC} - V_{GM}}{\dot{V}_{Susp}}$$

$$V_{GM} \approx V_{GC} \cdot \varphi_{GM} \cdot (1 - \varepsilon_{GM}) \quad (5)$$

$$t_{stat} = 4 \cdot t_F$$

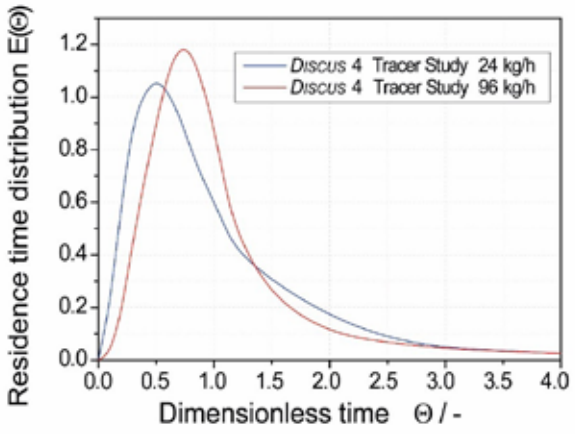
where	V_{GC}	[m³]	grinding chamber volume
	V_{GM}	[m³]	volume of solids in the grinding media
	ε_{GM}	[-]	porosity of the grinding media fill
	φ_{GM}	[%]	grinding media fill level
	$\dot{V}_{Susp}$	[m³/h]	volumetric flow of the product suspension

If the suspension were to move through the grinding chamber like a plug without mixing effects and without a velocity profile due to wall friction effects, a peak would result exactly at a dedimensioned time of 1.

However, it turns out that due to mixing effects in the mill, a large part of the suspension components moves through the grinding chamber much faster, while other components need at least 4 times as long as the ideal fill time.

Fig. 3

Tracer study of the dwell time distribution in passage mode



It is therefore generally defined that the static state is established during grinding in agitator bead mills after a waiting time tstat of at least 4 times the ideal fill time. Only after this waiting time has the total volume of the grinding chamber been completely replaced at least once and only then does the characterization of samples lead to a representative result.

Example:
A predispersed batch of a product is ground in fully continuous operation using a *Discus*® 60 disk agitator bead mill. The grinding media fill level of the mill is 80%. The product throughput is 500 $l_{suspension}$ / h. After a new agitator shaft speed has been set, it should be determined how long it is necessary to wait until the static state has been reestablished:

$$t_r = \frac{V_{GC} - V_{GM}}{\dot{V}_{Susp}} = \frac{(55\,l - 55\,l \cdot 0{,}80 \cdot 0{,}6)\,h \cdot 60\,min}{500\,l \cdot h} = 3.43\,min$$

$$t_{stat} = 4 \cdot t_r = \underline{13.73\,min}$$

The static state is therefore restored only after approx. 14 minutes.

This aspect must be taken into account, in particular when planning the trials, so that sufficient material is available to carry out the planned parameter settings. In the example described here, approx. 125 liters of the product would be pumped through the mill until a static condition of the grinding has been established again.

4. SAMPLE PREPARATION AND CHARACTERIZATION

4.1. Development of a Sample Preparation and Analysis Specification

Before starting the first tests, one should have a precise idea of the particle size distribution, the solids concentration, the formulation and the threshold values for contamination and temperature or the admissibility of the residual coarse material and the emerging fines. It is often worthwhile as well to ask the customer if he can provide a standard. It is not uncommon for the customer to have a product sample that exactly matches his ideas with regard to particle size distribution, color, gloss or rheological properties.

Since the results of different particle size analyzers can differ significantly, even with comparable technologies and using identical parameters such as refractive index, viscosity or temperature, comparative measurements are useful.

The customer should also provide existing SOPs (Standard Operating Procedure), if available. The term SOP is understood to mean a standard procedure or a standardized approach in which operational sequences are described with respect to the verification of results and their documentation, particularly in critical segments with potential impacts on the environment, health and safety.

Often, samples taken from the process cannot be analyzed directly in the original concentration. If samples must be diluted, it is important to validate the effect of the change in concentration of an existing additive, for example, on possible reagglomeration of particles. For this purpose, various analyses should be carried out with different concentrations of the additive and post-treatment of the sample with ultrasound.

However, it should be noted here that post-treatment of a sample with an intensive ultrasonic disintegrator can falsify the results of the tests if the objective of the comminution process in the mill is deagglomeration.

In any case, the exact procedure for sample preparation and characterization should be defined before the start of the test. This SOP should then be followed in order to exclude subjective influences to the greatest extent possible.

4.2. Occurrence of Deviations in Results

In sample analysis, it is not uncommon that findings do not meet expectations. It may happen that during an analysis coarse fractions are detected which, according to previous experience, should not be present. It should be noted here that an electronic measuring device always outputs the measurement result as a function of the sample being analyzed, independent of feelings, experiences and expectations.

In the event of unexpected results, it should always first be assumed that these have a real cause. If, for example, a coarse fraction is analyzed, this can have various causes. In any case, further investigations should clarify whether these are actual product particles, agglomerates, impurities or air bubbles.

If there are deviations from the expected test results, these should always be documented in full, if it cannot be soundly clarified that there was a mismeasurement caused, for example, by air bubbles or operating errors.

5. TEST REPORT

On the following page is a proposal for a standard report similar to that used in NETZSCH technical centers worldwide. In such a report, all essential parameters, such as speeds, throughputs, pressures, temperatures, power inputs, etc. are recorded in a clear format. At the same time, information about the machine, the machine configuration, the mode of operation, the sealing medium, the grinding media used, as well as test results are documented in a clear format.

If the test preparation, execution and results are always documented in the same format, this makes evaluation, comparison with other tests and discussion much easier.

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REFERENCES

[1] Mende, S., et all.
NETZSCH Kompendium, Zerkleinerung & Charakterisierung von Partikeln, September 2016

Responsible person / Operator: Machine: Test number:

	Installed power		kW		Date					
Machine equipment										
Pump										
Separeting system	mm	ø	mm	Sealing liquid						
Grinding bead type			mm	Mass	kg	%				
Product				max. temperature		°C				
Solid		kg		Solid content		%				
Dispersant		kg		Total mass		kg				
Solvent		kg		Density		kg/l				
Material preparation				Duration	min					
Quality requirements										
Target fineness	d_{50}	µm	d_{90}	µm	d_{95}	µm				

Operation Mode											
		0	1	2	3	4	5	6	7	8	9
Grinding time	t in min										
Agitator speed	n in 1/min										
Troughput	m in kg_{Susp}/h										
Pump speed	n in 1/min										
Pressure	p_M in bar										
Material temperature	T_{in} in °C										
Material temperature	T_{out} in °C										
Gross power	P_{total} in kW										
Net power	P_{net} in kW										
Net grinding energy	E_{net} in kWh										
Specific energy input	E in kWh/kg_{Susp}										
Sample from tank or outlet — Particle size	d_{50} in µm										
	d_{90} in µm										
	d_{95} in µm										
Other measurements											
Theoretical cycles	n_{circ}										
Production capacity	m_c in kg_{Susp}/h										

Cooling		Grinding chamber	Mechanical seal	Circulation tank	Chiller	total
Temperature inlet	T_{in} in °C					
Temperature outlet	T_{out} in °C					
Cooling water flow rate	m_{kW} in l/h					

Remarks

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