Measuring the flow properties of powders

FT4 Powder Rheometer
Successful powder processing requires the ability to reliably and repeatably predict flow behaviour under a variety of processing conditions. Insight into a powder’s flow characteristics provides a firm basis for informed decision making at every stage - from material formulation, through specifying plant and processing conditions, to storage and transport of the finished product.

By introducing a dynamic method of measuring powder flow properties, Freeman Technology has made possible the direct comparison of flowability data for different powders. A program of continuing development makes today’s FT4 Powder Rheometer a sophisticated universal powder testing system that is helping address a very broad range of powder processing challenges.

Core principle
The FT4’s unique, patented ability to measure both axial force and rotational force or torque acting on the rheometer blade whilst moving through the powder sample, allows the generation of highly discriminating, sensitive and reproducible data. The energy needed to make powders flow is automatically calculated from this data to provide dynamic flowability parameters for all packing conditions and flow regimes. The versatility of the FT4 allows a range of system modules to be used, including shear cells that provide shear properties data to complement the dynamic measurements.

Industrial applications
FT4 users find that better understanding translates directly into more confident prediction of powder behaviour, and more appropriate processing. In industry sectors as diverse as pharmaceuticals, fine chemicals, cosmetics, toners, powder coatings and ceramics, the FT4 is helping understand the challenges of:

- Batch variability
- Establishing flowability criteria
- Avoiding blockages
- Dosing weight variability
- The effects of fines
- Storage effects
- Matching powders to plant
- Flow additives optimisation
- Segregation

“With the FT4 it is possible, for the first time, to construct databases of reliable and differentiating flowability and processability data.” Reg Freeman
FT4 capabilities

Environmental Variables
- Aeration level
- Humidity
- Vibration
- Temperature
- Storage
- Pressure

Physical Variables of Powders
- Particle size
- Shape
- Hardness
- Size distribution
- Surface texture

Plant & Process Design
- Plant design data
- Wall friction data
- Predicting processability

Formulation
- Data to inform formulation decisions and design in flowability

Sectors
- Pharmaceutical
- Toners
- Powder coatings
- Fine chemicals
- Bulk materials
- Ceramics
- Metals and oxides
- Cosmetics

Types
- Storage
- Blending
- Dosing
- Fluidised bed

Challenges
- Attrition
- Agglomeration
- Segregation
- Bridging
- Caking

Methods of Analysis
- FT4 analysis

Measurement Capabilities
- Flowability
- Bulk density
- Aeration
- De-aeration
- Permeability
- Consolidation
- Shear strength
- Cohesion
- Moisture content
- Flow additives
- Electrostatic charge
- And more...

Applications
- Powder coatings
- Tableting
- Granulation end point
- Powder coating
- Flowability specifications
- Mulling

Specific Applications
- Vial filling
- Capsule filling
- Mould filling
- Bag filling
- Quality control

Processes

Challenges
- Attrition
- Agglomeration
- Segregation
- Bridging
- Caking

Graph showing the effects of attrition on the flow energy of powder. Initial measurements were made on unmilled lactose (time = 0) and thereafter at increasing milling times.

The data shows that the flow energy changes by a factor of more than 2 overall.

Flow additive study

Using a standard stability measurement program, the influence of flow additives on powder flow properties can be measured. The data shows how resistance to flow reduces with increasing levels of flow additive, and how little improvement is seen when additive levels are increased from 5 to 10%. However, with additive levels as low as 2%, flowability can be improved by a factor of more than 2.

Powder coating

By measuring the fluidisation characteristics of a powder, discriminating between ‘good’, ‘medium’ and ‘poor’ batches of material becomes routine.

Quality control standards can be set and out of specification material eliminated to avoid processing problems.
Dynamic measurements simulate complex process conditions – automated operation keeps analysis simple

Dynamic measurement of powder flow allows evaluation of multiple physical and environmental factors on a powder’s behaviour. The design of the FT4, in particular its high level of automation, makes routine measurement simple and data interpretation straightforward.

Typically, these fully automated dynamic measurements involve a unique conditioning process. Together with the elimination of any operator influence, this ensures truly repeatable testing and reliable comparison of results – between powders, between instruments and between sites.

Determination of the basic flowability energy (BFE) is followed by a number of standard tests designed to evaluate the effects of other variables including consolidation and aeration.

Preparing and loading a sample
Dynamic measurements begin with the evaluation of a normalised powder. Gentle displacement of the whole sample loosens and slightly aerates the powder, conditioning it to produce a homogeneously packed powder bed – effectively neutralising the effects of previous storage and handling.

The 2-minute Basic Flowability Energy (BFE) measurement
The BFE is the energy required to displace a precise volume of conditioned powder at a given flow pattern and flow rate. This test typically takes 2 minutes to complete and is independent of the operator. BFE assessment provides both a baseline for further testing and a quick, reproducible measurement for checking that a known material exhibits the expected rheology.

Gaining further insight
Further flowability parameters may be determined and defined in terms of the factor by which the BFE value is changed. For example, testing at variable flow rate, testing of consolidated powders and tests made whilst the sample is being aerated produce FRI, CI and AR values respectively (see table). An even more comprehensive characterisation might include the following:

- Bulk properties such as bulk density, compressibility and permeability
- How powder physical properties such as particle size distribution affect flowability
- Shear properties using the FT4’s automated shear cells
- Processability issues such as attrition, agglomeration, segregation, caking, adhesion and moisture

<table>
<thead>
<tr>
<th>Defining Parameter</th>
<th>Definition</th>
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<tbody>
<tr>
<td>Basic Flowability Energy</td>
<td>BFE</td>
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<tr>
<td>Stability Index</td>
<td>SI</td>
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<tr>
<td>Flow Rate Index</td>
<td>FRI</td>
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<tr>
<td>Aeration Ratio</td>
<td>AR</td>
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<tr>
<td>Consolidation Index</td>
<td>CI</td>
</tr>
<tr>
<td>Specific Energy</td>
<td>SE</td>
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<tr>
<td>Conditioned Bulk Density</td>
<td>CBD</td>
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Plug-and-play measurement options
+ full automation = universal powder tester

Full automation of powder testing using the FT4 Powder Rheometer enables accurate and reproducible determination of a range of critical parameters. Eliminating user variability allows confident discrimination of even small differences between powders. Unattended operation frees analysts for other tasks.

Built around the core dynamic measurement system are the following fully automated measurement options:

Aeration and de-aeration – ensuring predictable, consistent processing
The FT4’s Aeration Control Unit ensures fine control of air flow through a powder under test. This allows fully automated measurement of flow properties and permeability as a function of increasing levels of aeration or de-aeration. Even small amounts of entrained air can affect a powder’s bulk properties, so the unit enables evaluation at very low air velocities. A wide working range allows full examination of fluidisation behaviour, with its important practical processing implications for many industries.

Bulk properties – characterised using automated tests
- Bulk density measurements of conditioned, tapped and consolidated powders
- Permeability (pressure drop at fixed air throughput) as a function of varying bulk pressure
- Compressibility of a powder bulk as a function of bulk pressure

Shear strength measurement – complementing dynamic flow data
The precise control and extensive analysis capabilities of the FT4 have been exploited in the development of systems and methodologies for fully automated shear strength measurements. Shear strength can be measured in both consolidated and unconsolidated samples, using sample volumes as low as 3 ml.

The FT4 shear cells are delivered complete with a library of testing programs. Simply select the test and load the sample - the rest is fully automated.

- Standard shear cell for samples in the range 20 to 50 ml
- Small volume shear cell for samples as small as 3 ml and below (depending on the physical properties of the powder)
- Automated determination of yield loci at one or more pre-consolidated levels
- Automated Mohr circle derivation and data analysis
- Force and position control modes that allow testing at zero pre-consolidation and near zero normal stress
Instrument specifications

The FT4 Powder Rheometer uses the established and patented Freeman Technology principle, where a precision blade displaces powder as it moves along a helical path through the sample.

A wide range of flow patterns and flow rates can be achieved depending on the direction and speed of movement. All forces acting on the blade, including axial force and rotational force or torque, are measured. These data are used to calculate the work done in displacing the powder – the basis of the flowability assessment. In contrast with traditional methods which generate a single number result, this approach recognises the complex nature of powders and the need to evaluate flow properties in relation to the many key variables, including flow rate, aeration, consolidation and moisture content.

The FT4 uses an embedded high-specification processor. Built in networking capabilities mean that users can access powder flowability data remotely as well as carrying out analyses in the laboratory. A universal bus provides serial daisy chaining of all automated accessories. The system features a digital control system and digital data acquisition for force, torque and position measurement. This provides precision control, a broad speed range and high sensitivity.

Automated testing is fast and straightforward, with a typical test program being completed in around 4 minutes.

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