The Field Guide to
**Food Powder
Characterization**

Next-Generation
Food Powder Analysis

With Anton Paar's portfolio of analytical instrumentation, deepen your understanding of food powders with next-generation analysis techniques. Develop the right formulation, overcome transportation and storage issues, and offer better, more enjoyable foods for consumers.

This e-book contains detailed measurement data including determination of density, particle size, powder flow, cohesion strength, compressibility, and permeability.

1 Formulation and R&D

Finding the right formulation is of utmost importance not only to ensure the desired product quality, but also to save on manufacturing costs. Optimizing food powder parameters such as particle size and particle size distribution enables the manufacturer to lower production costs and for the consumer to enjoy higher quality products. Improving formulations based on particle size reduces the amount of viscosity modifiers or carrying material needed.

Additional parameters of interest when formulating food powders are the crystalline phases, crystallite size, and percent amorphous content ... An in-depth understanding of these parameters enables precise control over macroscopic properties such as the texture or mouthfeel of food products. X-ray diffraction (XRD) can be used to identify and characterize these parameters – leading to optimized processes and improved final products.

1.1 PARTICLE SIZE ANALYSIS OF VARIOUS FOOD POWDERS

Particle size is an essential parameter when it comes to developing and optimizing food powder formulations. It also has further influence down the value chain in process steps such as transport and storage. The main method used to determine particle size and particle size distributions of food powders is laser diffraction. The size of the particle and the angle of the diffracted light are inversely proportional. In other words, the angle of diffracted light decreases when the particle size increases. The resulting pattern of this analysis is used to gather information on how to adjust formulations based on particle size.

Particle analysis of chocolate powder

Cocoa mass needs to be ground down into smaller particle sizes to enable as much cocoa butter to be released as possible. On the other hand, very small particles lead to higher surface area meaning that more of the expensive cocoa butter is needed to coat the particles. The particle size thus has to be optimized in order to reduce the need for viscosity modifiers [1][2][3]. Furthermore, because the human tongue is capable of detecting differences in particle size as small as 3 µm, larger particles (larger than 30 µm) feel gritty in the mouth, making homogeneous particle sizes within a formulation desirable [3].

The samples described herein are couverture chocolate powder, milk chocolate powder and agglomerated chocolate powder as well as the most common additives milk powder and sugar.
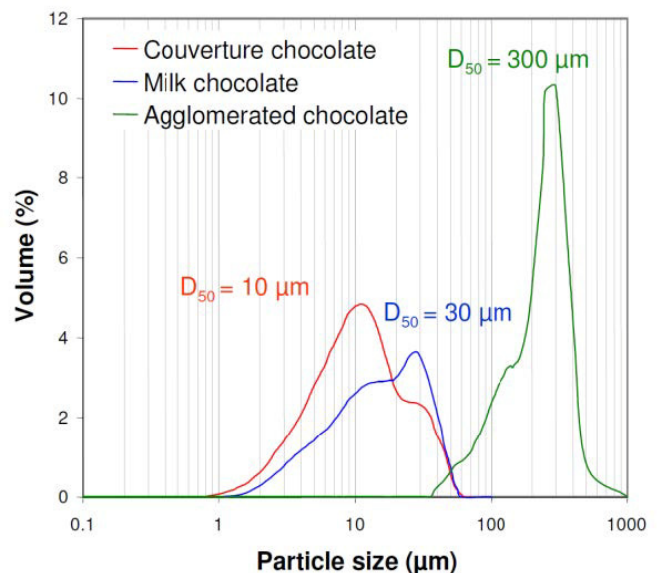

Figure 1: Particle size analysis of chocolate powders

As shown in Figure 1, all analyzed chocolate powders have a different particle size distribution (PSD). Couverture and milk chocolate have bimodal size distributions with diameters ranging from 10 µm to 30 µm. The particle diameter of agglomerated chocolate is 300 µm on average.
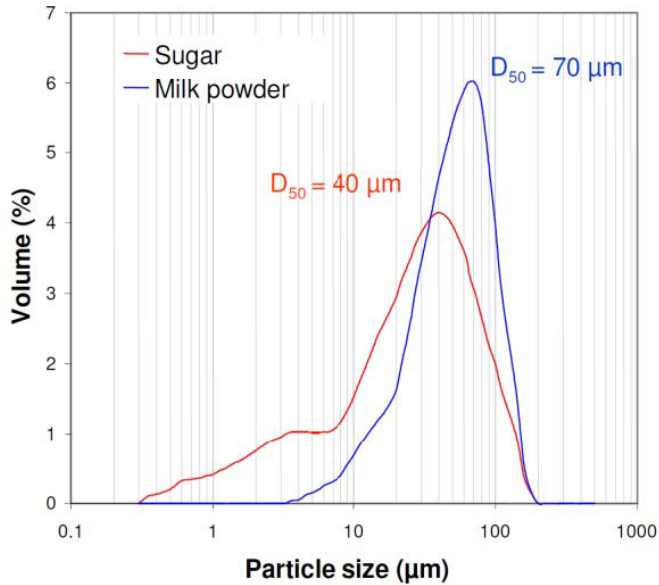

Figure 2: Particle size analysis of chocolate additives

PSD was also measured for the most common chocolate additives, milk powder and sugar. In figure 2 the PSD for these two samples is shown, with measurement returning average diameters of 40 µm for sugar and 70 µm for milk powder. Even though milk powder is ground during the manufacturing process, it is still apparent that adding it shifts the PSD of chocolate powder towards larger diameters compared with the (milk-free) couverture powder.

Particle analysis of sugar

Sugars in various forms are needed to fulfill different purposes. Examples are caster sugar and powdered sugar, both produced by pulverizing and milling down to a desired particle size. The close monitoring of particle size during sugar manufacturing is therefore essential.

The table below illustrates the differences in particle size of granulated and caster sugar.

SUGAR TYPE	D10 (µm)	D50 (µm)	D90 (µm)	SPAN
GRANULATED	878 ± 29	1113 ± 33	1525 ± 69	0.58 ± 0.03
CASTER	400.4 ± 20	680.6 ± 19	1083 ± 31	1.00 ± 0.06

Table 1: D10, D50 and D90 values of granulated sugar and caster sugar

The span of granulated sugar measured at a very low value of 0.58, indicative of a very narrow, monomodal dispersion. This suggests that the crystallization process used for production was well controlled and produced sugar crystals that were very homogeneous in size. The span for caster sugar was significantly higher (1.00) and points to a broader size distribution, suggesting that the method used to break down sugar crystals into smaller particles during production also introduced some level of heterogeneity.

Particle analysis of coffee

The particle size of ground coffee significantly influences the taste and has to be adapted to the brewing method. Espresso, for instance, uses a fine coffee powder, while filter brewing or French press preparations use higher particle sizes. Also, very fine, outlier particles release unwanted bitter flavors and should be avoided. Hence, a homogeneous size distribution is desirable. For industrially ground coffee this means that the particle size has to be continuously monitored.
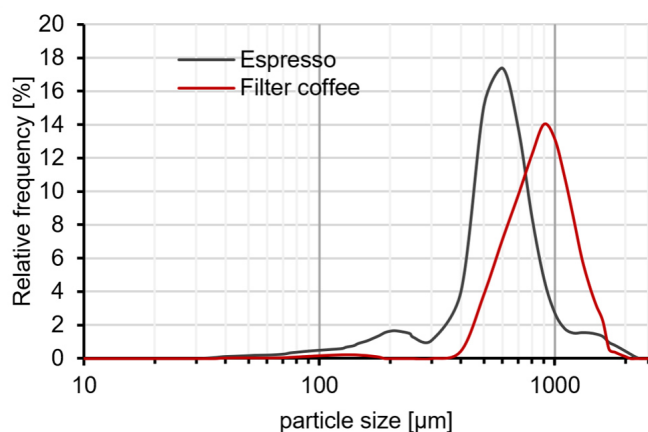

Figure 3: Representative volume-weighted particle size distribution of espresso coffee powder (grey curve) and of filter coffee powder (red curve)

Figure 3 compares the particle size distribution of an espresso powder with that of a filter coffee sample. The espresso powder sample has a particle size below 1 mm, with a main peak at 600 µm. The particle size of coffee powder used for filter coffee is much higher, with a main peak at 900 µm and a significant fraction above 1 mm.

The particle size distribution for both samples appears homogeneous, and the apparent lack of very fine particles suggests that the samples are of good quality.

Particle analysis of milk powder

Different requirements apply to milk powder for infants and toddlers. Infant formula should ensure better flowability and faster reconstitution in water. Particle size is again the parameter of interest when optimizing these properties.

The upcoming measurements were conducted with two different dispersion modes, Venturi and free-fall.

INFANT (I) AND TODDLER (T) FORMULA (VALUES IN µm)				
		D10	D50	D90
VENTURI	I	72	189	402
	T	48	130	315
FREE-FALL	I	501	765	1193
	T	478	746	1222

Table 2: Summary of D-volume weighted data of laser diffraction for the infant (I) and toddler (T) formula

The different size of primary particles measured in Venturi can be directly correlated with the reconstitution and dissolution velocity of the milk powder. The infant formula has bigger primary particles which mean a lower surface area than the toddler formula sample. The bigger particles further suggest higher porosity for the infant formula. For this reason, the infant formula reconstitutes faster in water. Moreover, the flowability is also better than that of the toddler milk because of the bigger agglomerate size measured in free-fall.

INSTRUMENTS SUITABLE FOR THESE MEASUREMENTS
PSA Series

1.2 X-RAY DIFFRACTION TO IDENTIFY CRYSTALLINE PHASES IN FOOD POWDERS

Sugars are commonly used powder ingredients in food manufacturing that come in various forms. Since each type of sugar has a different crystal structure, sugars can easily be differentiated from each other using X-ray diffraction (XRD). XRD can be used to investigate the crystallization of amorphous sugars – a process that is often undesirable due to accompanying detrimental property changes, or for quantification of the crystalline and amorphous components present in a powder mixture.

In the following analysis, XRD patterns of pure sugars and of some sugar-containing finished food products were recorded using a powder X-ray diffractometer. The crystalline sugars in the food products were then identified from the measured diffractograms. Four pure sugars, three different types of household sugar and six randomly selected sugar-containing products were ground to serve as sample materials for this study.

For further information on experimental details please read through the comprehensive application report: **Identifying Sugars in Food Products Using X-ray Diffraction**
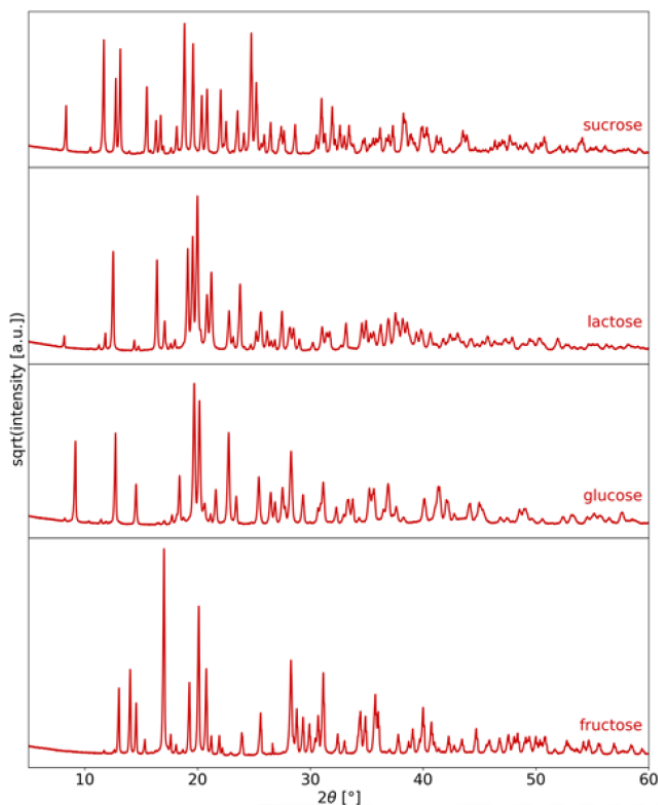
Figure 4: XRD measurements of four different pure sugars.

Figure 5: Diffractograms of different household sugars compared with the diffraction pattern of pure sucrose.

Figure 4 compares the measured diffraction patterns of pure sucrose, lactose, glucose and fructose samples. The different crystal structures of the four sugars result in completely different diffractograms. Like for many organic samples, the first Bragg peaks occur at small scattering angles – for sucrose, lactose and glucose even at 2θ values < 10°. This emphasizes the need for a low background at small 2θ angles. For larger 2θ angles (in particular for 2θ > 45°), strong peak overlap occurs even for these pure, single-phase samples due to the complex crystal structures of the samples. Thus, the low 2θ range is of most interest when investigating sugar-containing samples using XRD.

For a comparison of the three types of household sugars, the diffractograms magnified to the most relevant 2θ range are shown in Figure 5. At first view, all three diffractograms represent the diffractogram of pure sucrose.
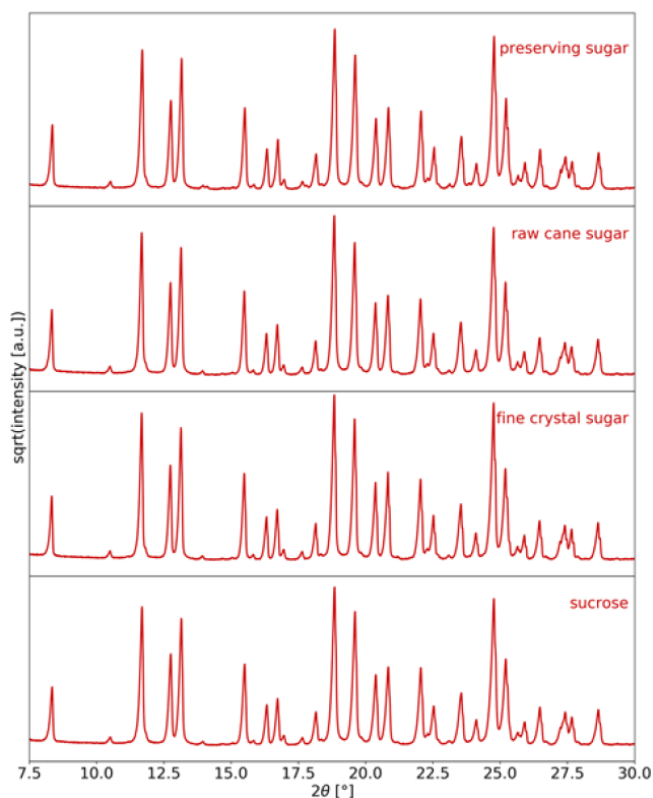
However, some small additional peaks can be found in the diffractogram of preserving sugar, in particular when plotting the intensities with a log scale (Figure 6). These can be ascribed to pectin, which usually accounts for 1 % to 2 % of preserving sugar, while the remaining content is sucrose. This highlights the potential of XRD to identify trace phases in a material; this is made easier due to the low measurement background observed in the measured data presented here.
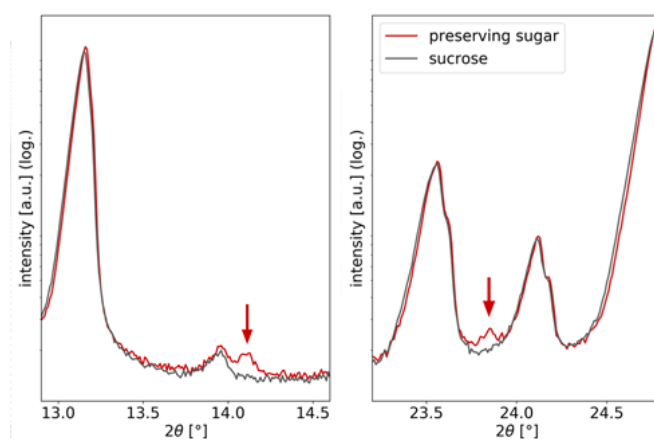

Figure 6: Two magnified parts of the diffractograms showing additional peaks from pectin in preserving sugar (red arrows)

Figure 7 compares the diffractograms of sucrose and dextrose to those of some sugar-containing food products.
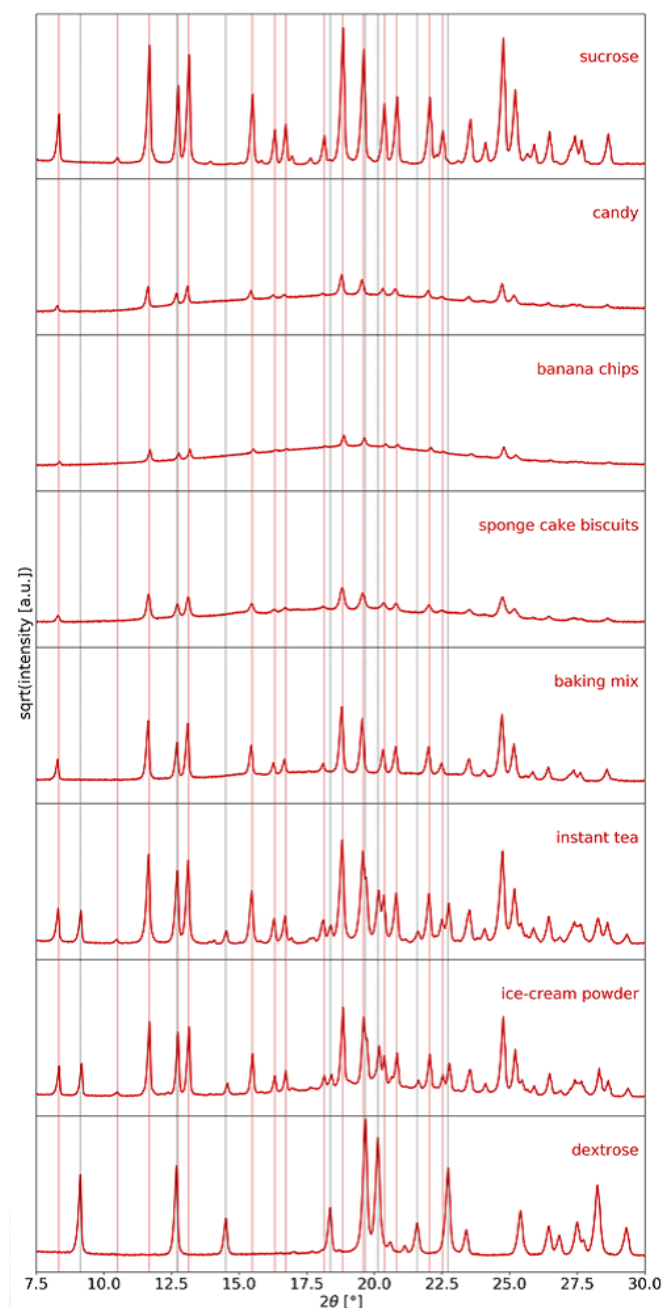

Figure 7: Diffractograms of sugar-containing food products compared to the diffraction patterns of pure sucrose and dextrose. Vertical lines indicate the main Bragg peak positions of sucrose (red) and dextrose (black)

The diffractograms of ice-cream powder and instant tea show sucrose as their main component. In both products, significant amounts of dextrose are also observed. For sponge cake biscuits, all observed Bragg peaks can be ascribed to sucrose. However, the absolute sucrose peak intensities are much lower than for pure sucrose, indicating that major phases other than crystalline sucrose are also present. Since the main components have an amorphous structure, they do not produce Bragg peaks, but instead a very wide hump of intensity is visible in the diffractogram. This is also true for the banana chips and the candy. A comparison to the list of ingredients shows that the hump

is mainly caused by amorphous or weakly crystalline starch and oil for the banana chips, while it is mainly caused by glucose syrup for the candy. Also, for both the banana chips and the candy, all Bragg peaks in the diffractograms can be ascribed to sucrose, indicating sucrose as the main crystalline component. Small peak shifts between the sucrose peaks of the different food product samples may be caused by different moisture contents of the sugars.

INSTRUMENTS SUITABLE FOR THESE MEASUREMENTS
XRDynamic 500

2 Transportation and Powder Flow Rheology

Powder rheology deals with characterizing powder flow behavior under realistic conditions. The combination of two powder cells – powder flow cell (PFC) and powder shear cell (PSC) – opens up the possibility of applying static and dynamic measurement methods to simulate various manufacturing and storage processes. The flow behavior of a powder is determined by a multitude of adhesive and repulsive forces, which depend on internal and external influences. Internal influences include, for example, particle size, size distribution and density, and also factors such as surface texture, surface area and porosity. External factors comprise moisture, temperature, gas throughput and degree of consolidation, among others. The flowability of the powder is therefore a non-inherent material property. Due to the multitude and complexity of the influencing factors, a theoretical prediction of the powder behavior is difficult and requires experimental characterization.
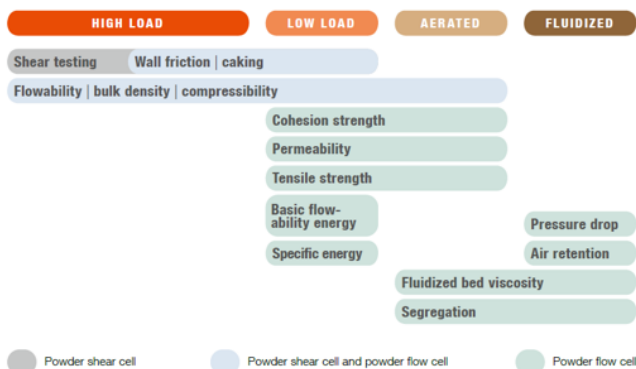

Figure 8: True powder rheology methods

2.1 RECOGNITION OF CHANGES IN POWDER FLOW PROPERTIES

Fluidization is a process were a granular material is converted from a solid-like state into a dynamic fluid-like state by introducing pressurized gas through the powder bed. Considering different production steps, fluidization can be found, for example, in fluidized bed reactors, pneumatic transport, and also during silo discharge when the particles are in free fall.

The powder flow cell helps to characterize the behavior of powders in the fluidized state, and during deaeration. This so-called deaeration process occurs after the powder has been fluidized and begins to release the trapped air without further compaction. In this way, it is possible to simulate real life applications like pneumatic conveying, and to identify de-mixing tendencies of particle blends.

The following sections provide an overview of different methods that can be applied to study and characterize different flow behaviors of food powders.

Cohesion strength
Cohesion strength describes the internal resistance of a powder to flow based on the adhesive forces between particles. The test involves an initial fluidization followed by a deaeration process. This aeration step is used as sample preparation and enables operator-independent measurements and erasing of the powder's memory. Due to the high repeatability and sensitivity of the test method, even small changes in the powder bed can be monitored. The ideal gas flow for fluidizing the powder can be determined with the pressure drop test. The measuring principle is based on a constant rotation of a two-blade stirrer and measurement of the resulting torque. The cohesion strength is a relative value calculated from the measured torque and a calibration factor.

Some food powders, such as coffee creamer, are transported and dosed by pneumatic conveying. Here, the pressure drop method and cohesion strength measurement can provide important information to optimize the individual process steps and avoid incorrect dosing and problems during transport. Figure 9 displays the results of cohesion strength measurements of two different coffee creamer samples in double determination. The results show that the visually indistinguishable samples exhibit significant differences in their flowability. The lower torque and the resulting lower cohesion strength of coffee cream A indicates lower internal friction (or lower bonding strength) between the particles. Higher cohesive forces were found for coffee creamer B, which can cause poorer flowability and potential problems during transport and dosing. Table 3 shows the cohesion strength value of the two coffee creamers with the deviation obtained in the double determination.
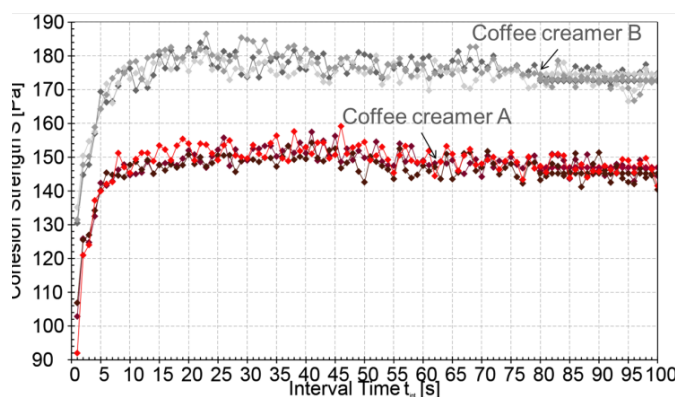

Figure 9: Cohesion strength results of two coffee creamer samples containing the same ingredients.

SAMPLE	MEAN COHESION STRENGTH ± STANDARD DEVIATION [Pa]
COFFEE CREAMER A	146 ± 1.2
COFFEE CREAMER B	174 ± 1.2

Table 3: Cohesion strength results ± standard deviation of the two coffee creamer samples obtained by averaging the last 20 data points of the cohesion strength curve.

Wall friction

Wall friction describes the friction between a bulk material and a solid surface. The wall friction angle is not only important for the design of silos, but also for the design of chutes and other equipment where the bulk solid flows over a solid surface. Influencing parameters are not only the roughness and cleanliness of the wall material, but also the material itself. Therefore, it is of great importance to adapt the test conditions to the real-life conditions to which the powder is subjected. In this context, the influence of the wall material on two different milk samples is presented.

Determination of the wall friction angle is performed by moving a measuring system equipped with a disc over the compressed bulk material. Infant formula and skim milk powder were tested with two different wall materials, namely stainless steel and polytetrafluoroethylene (PTFE, Teflon®). The samples are compressed with a normal stress of 3 kPa, 6 kPa and 9 kPa and the disc is rotated at a constant speed on the surface of the compressed powder bed. As a result, the shear stress between the wall material and the powder bed surface is measured. The recorded torque is displayed for both milk powder formulations at 9 kPa and the PTFE wall material in Figure 10. From the torque signal it can be seen that the interaction of skim milk powder with PTFE is significantly higher.
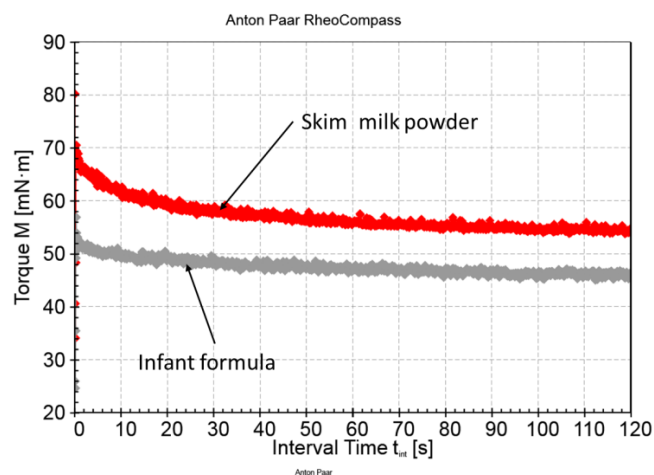
Figure 10: Results of wall friction for infant formula and skim milk powder at 9 kPa and PTFE wall material.

The characteristic wall shear stress for each applied normal stress is plotted on a shear stress - normal stress diagram. The line passing through the pairs of normal stress and wall shear stress marks the wall yield locus. The wall yield locus is defined as the yield limit and specifies the shear stress required to move the bulk material continuously across the wall surface. The mean wall friction angle results from the trend line of the data points running through the origin. An example of the wall yield point and wall friction angle plot is shown in Figure 11. As a general rule, the greater the wall friction angle, the greater the wall friction.

Figure 11: Exemplary plot of the wall yield locus and the mean wall friction angle.

Table 4 shows the calculated mean wall friction angles for the two milk powders, using stainless steel and PTFE as wall material. While the wall material has only a minor influence on infant formula, skimmed milk powder shows a significant increase in wall friction with stainless steel walls compared to PTFE. This indicates that PTFE-coated containers are much better suited for the storage and processing of skim milk powder than stainless steel ones.

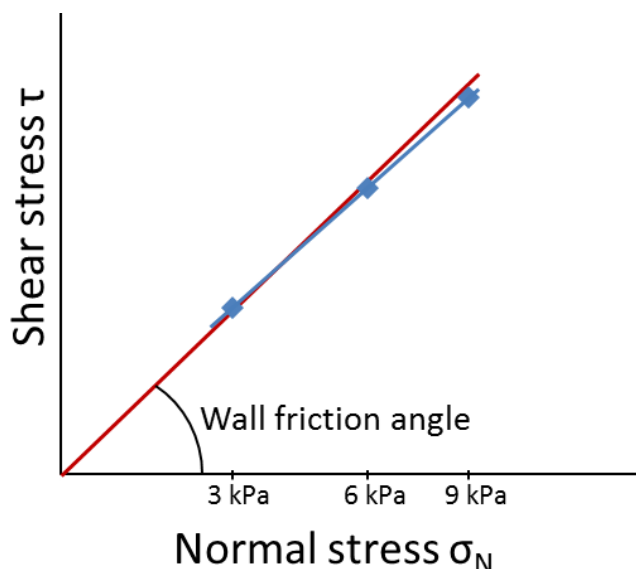|	STAINLESS STEEL [°]	PTFE [°]
INFANT FORMULA	25 ± 0.2	22 ± 1.8
SKIM MILK POWDER	57 ± 0.0	25 ± 0.8

Table 4: Results of mean wall friction angle for infant formula and skim milk powder with stainless steel and PTFE wall material.

INSTRUMENTS SUITABLE FOR THESE MEASUREMENTS
MCR Evolution Series
Powder flow cell (Fluidization only with PFC)

2.2. INVESTIGATION OF POWDER FLOW BEHAVIOR AS A FUNCTION OF EXTERNAL INFLUENCING PARAMETERS SUCH AS RELATIVE HUMIDITY

Compressibility
Compressibility is a measure of the relative change in volume that a sample undergoes when pressure is applied. The term compressibility thus refers to the increase in bulk density with increasing consolidation stress. Factors influencing compressibility are particle size and shape, and also water content and temperature. In this example, the influence of moisture on the compressibility of flour is shown in Figure 12.
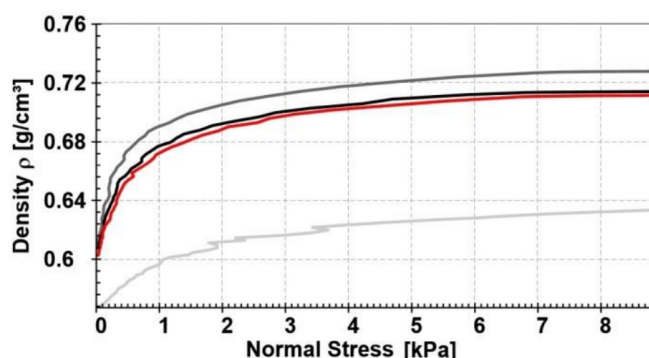

Figure 12: Compressibility of flour at ambient conditions (dark grey) and after conditioning the samples in the climate chamber at 35 °C and 95 %RH for 1 h (black), 2 h (red) and 24 h (light grey).

An increase in the water content in the flour sample leads to a general decrease in the achievable bulk density during compaction. Thus, the longer the flour was exposed to moisture, the higher the porosity was due to granulation and the increase in particle size. From the compression behavior, the flowability of the powder can be calculated using the Carr index and the Hausner ratio. The relationship between compression and flowability is as follows: The more a powder bed can be compressed, the worse its flowability.

This simple compression test can be used, for example, to avoid ratholing and arching in silos or hoppers and also load studies on walls and feeders can be performed.

Warren Spring cohesion
Warren-Spring cohesion is a method for directly measuring the cohesion strength of non-fluidizable powders, such as cohesive or very cohesive powders with the powder flow cell.

The test procedure involves an initial compression of the powder bed with a defined normal stress (3 kPa, 6 kPa or 9 kPa). After removing the vertical load, a vane paddle (Warren-Spring geometry) is lowered into the bulk material until the upper edges of the vanes are leveled with the surface of the bulk material. In a final step, the Warren-

Spring geometry begins to shear the compressed bulk material by rotating at a constant speed of 0.1 rpm and the resulting torque (M) is recorded. The Warren-Spring cohesion (S_{WS}) is calculated according to Equation 1.

$$S_{WS} = \frac{3 * M}{2\pi \, (R_0^{\square} - R_i^{\square})^3}$$

Equation 1: Calculation of Warren-Spring cohesion. (R0 and RI are defined as the outer and inner diameter of the Warren-Spring geometry)

The resulting curve in Figure 13 shows the Warren-Spring cohesion at the curve maximum where the consolidated powder fails and begins to flow due to the applied shear stress.
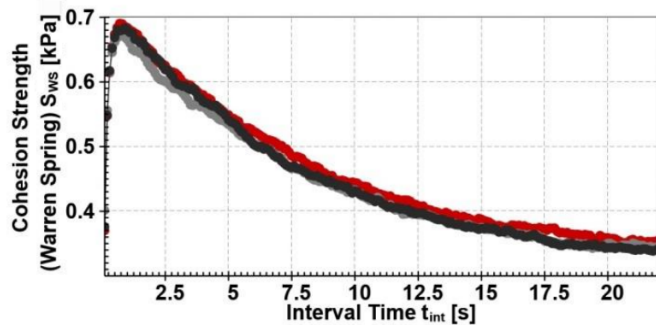
Figure 13: Warren-Spring cohesion measurements of flour in triplet determination at 3 kPa consolidation stress (pre-treatment: sample drying at 90 °C overnight).

The increase in cohesion strength due to water adsorption can be studied with Warren-Spring measurements after the powder has been exposed to different environmental conditions in a climate chamber. For this purpose, the flour was preconditioned at a relative humidity of 95% RH and a temperature of 35 °C for different periods of time (Figure 14).
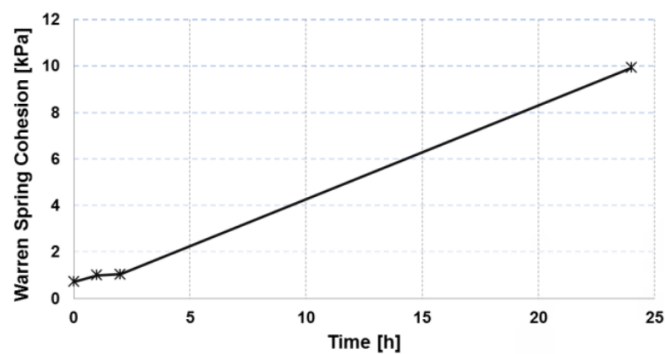
Figure 14: Warren-Spring cohesion of flour after 0 h, 1 h, 2 h and 24 h in the climate chamber at 95 %RH and 35 °C.

The increased water content in the flour sample promotes the formation of capillary bridges between the particles and thus an increased cohesion strength value. The

test results show that exposing the flour to high relative humidity values for a short period of time (1 and 2 h) does not significantly change the flow behavior. However, if the exposure time is increased to 24 hours, a significant increase in cohesion strength is observed. This undesirable and uncontrolled agglomeration of powders is called "caking" and is one of the main problems during transport and storage of powders containing salt or sugar. The caking effect can not only cause problems during the processing of the powder, but also affect the quality of the final product.

Yield locus and time yield locus
The yield point of a consolidated bulk solid is called the yield locus function and indicates the boundary between "solid" and "liquid" behavior at a well-defined consolidation state. More information on the test procedure can be found in the application report "Introduction to Powder Rheology".

The powder shear cell can be used together with a convection temperature device and a humidity generator to condition and measure the sample under controlled environmental conditions without the need for an additional climate chamber. The change in flowability is observed via an initial reference measurement and subsequent tests after application of defined humidity and temperature levels over a defined period of time. The effect of caking based on humidity was observed here on a protein powder (Figure 15). During the test, the temperature was kept constant at 40 °C and a relative humidity of 18% RH was selected. The protein powder was conditioned in the convection temperature oven for 24 h and the change of flow behavior was monitored.
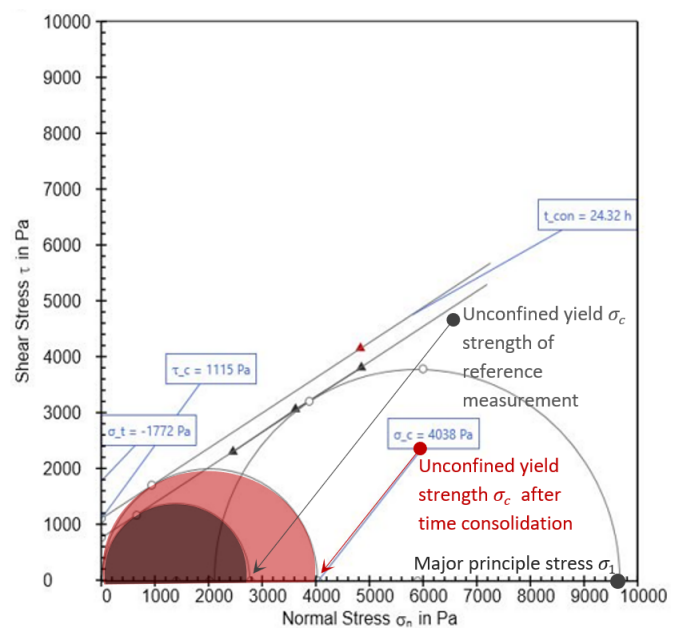
Figure 15: Yield locus function (black; before sample conditioning) and time yield locus at 6 kPa pre-shear (red; after exposure to 18% RH and 40 °C for 24 h) of a protein powder.

The unconfined yield strength of a powder is obtained by drawing a Mohr circle through the origin and tangential to the yield locus function. The higher the point of intersection on the x-axis, the higher the normal stress required to deform or "break" the powder as a function of the major principal stress σ_1 (consolidation stress). This means that a powder will flow if the force acting on the powder is greater than the unconstrained yield strength, and σ_c is therefore a measure of the flowability. From the relationship between the major principle stress and the unconfined yield strength, the coefficient of flowability ($ff_c = \sigma_1/\sigma_c$) can be determined. The time consolidation test results with variable humidity are shown for the protein powder in Table 5.

COEFFICIENT	REFERENCE SHEAR MEASUREMENT (BEFORE TIME CONSOLIDATION STEP)	SHEAR MEASUREMENT (AFTER TIME CONSOLIDATION STEP)
COHESION T_C	786 Pa	1115 Pa
UNCONFINED YIELD STRENGTH σ_C	2782 Pa	4038 Pa
MAJOR PRINCIPLE STRESS σ_1	9680 Pa	
COEFFICIENT OF FLOWABILITY ff_c	3.48	2.40

Table 5: Coefficients derived from the protein powder yield locus analysis before and after the time consolidation step (sample conditioning at 18% RH and 40 °C for 24 h).

The test shows that when the powder is exposed to moisture, a significant increase in the yield locus function and unconfined yield strength is observed and consequently higher forces are required to "break" the powder bed.

To determine what environmental conditions the powder can be exposed to until the caking effect inhibits the flow of the powder, several tests were carried out with increasing relative humidity. During the test, the temperature and the exposure time were kept constant compared to the previous test and only the humidity was increased step by step. At the point where σ_c exceeds σ_1, the powder loses its flowability. Figure 16 shows that the protein sample starts to clump when a water activity of more than 0.25 is reached.
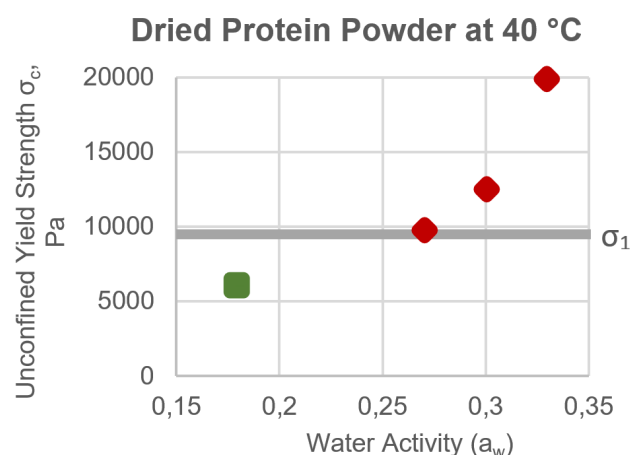
Dried Protein Powder at 40 °C

Figure 16: Evolution of time consolidation unconfined yield strength with increasing water activity/humidity level in the temperature chamber.

INSTRUMENTS SUITABLE FOR THESE MEASUREMENTS
MCR Evolution Series
Powder flow cell
Powder shear cell with CTD 180 and humidity generator

3 Packaging and Storage of Food Powders

Environmental conditions, such as temperature and humidity, and mechanical forces, including gravity, exert influence on how a powder behaves.

In this unit, different measurements with specific purposes are proposed for food powder characterization: density and oxidation stability. These measurements enable producers to optimize the powder's packaging process and storage conditions to ensure product quality at all times.

3.1 TAPPED AND TRUE DENSITY OF FOOD POWDERS

Fine powders and granular materials are widely used in industrial processes in a variety of applications. The performance of the final product depends on the properties of the individual powdered components that make up the formulation. Morphology, cohesive strength, and the powder's tapped or true density are some of the properties that are used in formulation optimization and contribute to efficient packaging and transportation of the final blend. Therefore, they have become standard techniques for product development. Tapped density analysis offers the simplicity of characterization and provides an insight into a powder's cohesiveness and flowability. This value is obtained by mechanically tapping a volumetric vessel or a graduated cylinder containing a known amount of sample a fixed distance until no visible change in its volume is observed.

The material's compressibility index and Hausner ratio can then be calculated. Free-flowing powders generally show little difference between the bulk and tapped densities.

Determination of a material's true density begins with an application of Archimedes' principle of displacement. Because some samples interact with the usual displacement fluid (water), gases such as helium or nitrogen are substituted. Boyle's gas law is then applied to ascertain a material's true volume. The results of the two processes are used to calculate the true density, which can be used to directly assess the chemical composition and morphology of a material in a quality control step.

Measurement data and results

Four samples of ground coffee were added to 250 mL graduated cylinders (readable to 2 mL) to around two-thirds of their volumes. The weight of each was recorded. Before documenting the untapped volume (bulk density), each coffee was shaken and visually inspected to ensure it had no discernable air pockets in its uncompacted state. The ground coffee sample in the cylinder was then placed and secured on the tapping head of the Autotap. Volumes were recorded after 10, 500, and 1,250 taps. An additional 1,250 taps were used to check for any observable change in volume.

The final results are outlined in Table 6, which clearly shows that the tapped densities of the different types of ground coffees increase by around 0.04 g/ml. In Image 1.2, the four analyzed ground coffees are shown in a plot of volume versus number of taps. The differently roasted coffees exhibit similar trends with similar values. The blonde roast is the slight outlier with significantly higher bulk and tapped density values. Bulk and tapped density, the compressibility index, and the Hausner ratio are calculated following the formulas below.

Equation 1 – Bulk density (g/mL):
where: m = weight of the sample (g)
V_i = initial volume of the sample (mL)

$$\frac{m}{V_i}$$

Equation 2 – Tapped density (g/mL):
where: m = weight of the sample (g)
V_f = final volume of the sample (mL)

$$\frac{m}{V_f}$$

Equation 3 –
Carr's Compressibility index (%):
where: V_i = initial volume of the sample (mL)
V_f = final volume of the sample (mL)

$$\frac{(V_i - V_f)}{V_i} \cdot 100$$

Equation 4 – Hausner ratio (g/mL):
where: V_i = initial volume of the sample (mL)
V_f = final volume of the sample (mL)

$$\frac{V_i}{V_f}$$

COFFEE ROASTS	BULK DENSITY (g/mL)	TAPPED DENSITY (g/mL)	COMPRESS-IBILITY INDEX (CI) %	HAUSNER RATIO (HR)
DARK	0.33	0.37	12.6	1.14
MEDIUM	0.31	0.35	13.4	1.14
BLONDE	0.36	0.42	13.7	1.16
DECAF DARK	0.30	0.35	12.9	1.15

Table 6: Bulk & tapped density of coffee roasts
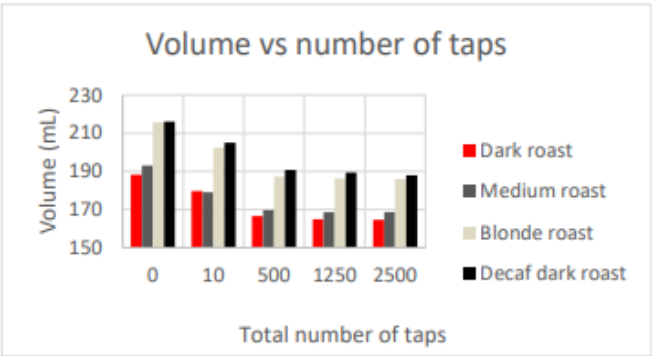
Figure 17: Volume versus number of taps

HAUSNER RATIO (-)	CARR INDEX (%)	FLOW CHARACTER
1-1.11	10	Excellent
1.12-1.18	11-15	Good
1.19-1.25	16-20	Fair
1.26-1.34	21-25	Passable
1.35-1.45	26-31	Poor
1.46-1.59	32-37	Very poor
>1.60	>38	Very, very poor

Table 7: Scale of flowability according to Hausner ratio and the Carr index

Interpretation of results

Figure 17 shows how volume decreases as the number of taps increases. The initial volume of the same mass of coffee is larger for dark coffee than blonde. This can be attributed to reduced exposure to heat, causing more volatiles (moisture) to be retained in the coffee bean.

Results show that the compressibility indices are approximately 13% and Hausner ratios are near to 1.15. These ground coffees exhibit good flow characteristics according to the flowability scale (Table 7).

Conclusion

Food powder manufacturers can use the information gleaned by tapped and skeletal density measurement to optimize packaging and storage processes. Knowing a material's compressibility index and Hausner ratio can influence container design, transport requirements, and storage necessities.

> INSTRUMENTS SUITABLE FOR THESE MEASUREMENTS
> **Autotap Series**
> **Ultrapyc Series**

3.2 OXIDATION STABILITY OF FOOD POWDERS

Food powders in the form of final product or as an ingredient typically have a long shelf life, however it depends on their physical and chemical properties as well as on the packaging and storage conditions. A conventional test method for analyzing the oxidation stability of powder is e.g., the sensory analysis or the determination of the peroxide value after a certain storage time. Compared to these test methods the RapidOxy 100 provides an easy, fast, and safe determination of oxidation and storage stability without any sample preparation.

Measurement data and results

In contrast to the sensory analysis or the investigation of the peroxide value, the RapidOxy 100 determines the oxidation stability in a fully automatic manner. The measurement proceeds under accelerated conditions by increasing the temperature and the exposure to excess of pure oxygen. The sample in the test chamber is set under pressure with pure oxygen and heated up. During the measurement the temperature is kept strictly constant and the pressure is continuously traced. Due to the oxygen consumption the pressure drops accordingly. Since the consumption of oxygen by a powder is a rather slow process, the measurement is stopped after a defined time (e.g. 20 hours). The pressure drop is sample-specific. The greater the amount of oxygen consumed within the set time, the less oxidatively stable is the powder. The pressure drop can simply be compared to other samples to investigate different batches, formulations or packages. The higher the pressure drop the lower is the oxidation and storage stability.

A test of corn flour under the conditions below shows the following data:

PARAMETER	SETTING
PROGRAM	Temperature-sensitive sample
TEST TEMPERATURE	60 °C
FILLING PRESSURE	700 kPA
TEST DURATION	1200 min

Table 8: Test conditions

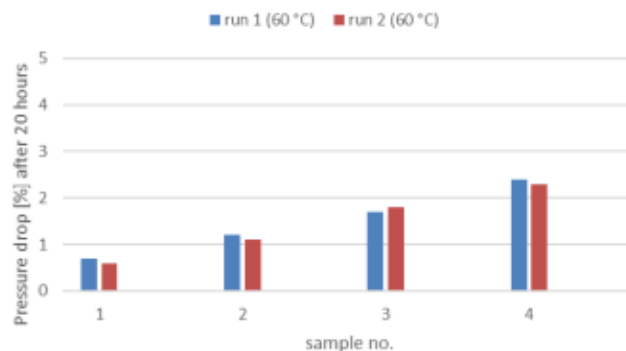| SAMPLE NO.	PRESSURE DROP [%] AFTER 20H: RUN 1	PRESSURE DROP [%] AFTER 20H: RUN 2	STANDARD DEVIATION [%]	REPEAT-ABILITY [%]
1	0.7	0.6	0.05	15.4
2	1.2	1.1	0.05	8.7
3	1.7	1.8	0.05	5.7
4	2.4	2.3	0.05	4.3

Table 9: Oxidiation stability results

Figure 18: Pressure drop of the two measurement runs

Interpretation of results

As the results show in Table 9 and Figure 18, the oxidation stability decreases from sample 1 to sample 4. Sample 1 indicates the highest oxidation stability because the lowest pressure drop is observed whereas sample 4 indicates the lowest oxidation stability because the highest pressure drop is observed. Furthermore, the standard deviation of each run is calculated to evaluate the quality of the test results. The small standard deviations show the measured test results are reliable so that different packaging, and storing conditions can be evaluated.

Conclusion

Measurement of oxidation stability of food powders without sample preparation is repeatable and reliable using the proposed method in order to identify the most appropriate packaging and storage conditions. Packing materials can also be evaluated as well as the shelf life that results from the usage under specific conditions.

INSTRUMENTS SUITABLE FOR THESE MEASUREMENTS
RapidOxy 100

4 Final Product Analysis and Customer Experience

One of the most critical aspects of food powder development is ensuring that the final product is safe and enjoyable for the consumer. When opening a food powder package, there should not be any visible clumps or moisture and the powder should readily dissolve or mix into water or with other ingredients. Thus, the surface area and pore size of the final formulation must be optimized to ensure that the final product is stable and dissolves quickly. Additionally, along with surface area, the viscosity of suspended or dissolved powders should be analyzed to ensure that the product mouthfeel for the consumer is pleasant and neither too gritty nor too viscous. Finally, food powder products must be tested for the presence of heavy metal and trace element contamination to ensure that the final product is safe for consumption.

4.1 DISSOLUTION RATE OF FOOD POWDERS

The overall surface area and pore size of a food powder significantly affect its dissolution rate. Both surface area and pore size are typically measured by gas sorption analysis.

Surface area of milk powders

Once the solvent reaches the single particles of the powder, the surface area is critical, because it defines the area available for the interaction between the solvent and the solute particles. The surface area is a function of particle size and shape, can differentiate porosity from the bulk sample, and can be measured using vacuum volumetric methods. This technique measures the amount of an adsorbate (e.g. gas or vapor) adsorbed onto the solid surface of an adsorbent (e.g. milk powder) at a defined absolute temperature and pressure. Measurements of the gas or vapor uptake amount versus pressure at a constant temperature is known as the adsorption isotherm. This isotherm is used to determine the accessible surface area by applying the Brunauer Emmett Teller (BET) method.

The calculated BET-accessible surface area values for the experimental Kr adsorption isotherm (Figure 19) were 0.041 m^2/g and 0.074 m^2/g for infant and toddler milk powder formula, respectively. With the low surface areas

of the milk powders, low values of the BET C constant (4.14 and 7.05) were consistent with similar organic compositions that display weak adsorbate-adsorbent interactions.
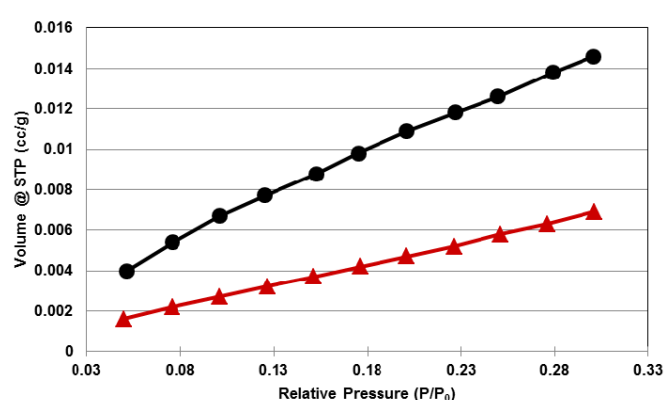
Figure 19: Kr adsorption isotherms at 77 K for toddler (black circles) and infant (red triangles) formula.

These results show clear particle size and morphologic differences between the samples, leading to the conclusion that the toddler formula has a higher solubility overall, but is prone to form clump during dissolution due to its low permeability. This is corroborated subjectively through their behavior in the subsequent preparation steps in the following liquid analyses.

INSTRUMENTS SUITABLE FOR THESE MEASUREMENTS
Nova Series

Permeability of milk powders

Permeability is determined by how well the solvent (in this case water) is able to access the individual particles of the powder. The higher the permeability, the more accessible is the sample matter.

This parameter was measured using the combination of an Anton Paar rheometer and a powder flow cell, by pumping a defined air flow across the particle bed at a fixed pressure gradient. This indicates how much access the water has to the product which influences the process of dissolution. A product with high permeability in this sense prevents the undesired formation of knots and nodules of undissolved powder while dissolving. These lumps of undissolved powder are the result of a barrier layer forming when water is taken up within mostly secondary agglomerates, which slows down the process of dissolution. Since formula should be prepared quickly, this is highly undesirable, making this an important parameter in regards to the quality of the product.

Figure 20 and Table 10 show the permeability results, representing the powders' air flow resistances under different compaction forces. The infant formula is not only notably more permeable, it is also much more dependent on the compaction, showing a significant change.
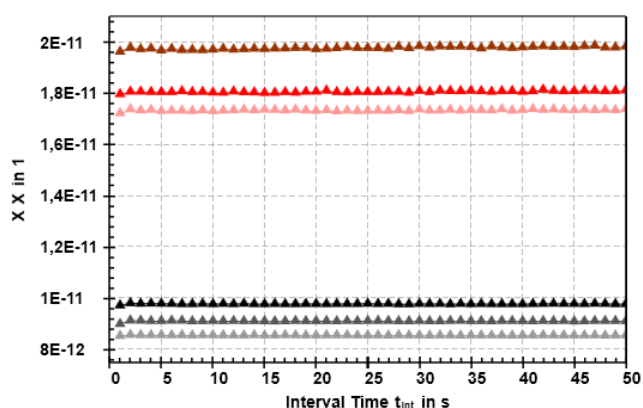

Figure 20: Permeability – toddler formula (black to gray curves) and infant formula (red curves) measured at compressions of 3 kPa, 6 kPa and 9 kPa (from darkest to lightest)

Generally, the permeability decreased with increasing normal stress during compression, and the infant formula showed significantly higher permeability (by a factor of more than two) than the toddler formula. In itself, this could be an indication of higher particle size diversity. However, this could also be a function of chemical composition. A higher amount of carbohydrates in the composition (as is the case with the toddler formula) would naturally change the morphology of the particles, in this case leading to less free volume and therefore lower permeability.

The dependence on compression (Figure 20) shows the vulnerability of some powders to compaction. It can be expected that the infant formula will be more likely to produce clots after prolonged storage compared to fresh infant formula. However, the toddler formula shows worse dissolution behavior irrespective of applied stress and thus will exhibit clotting either way. This was also observed subjectively in the dissolution steps.

SAMPLE	PERMEABILITY [10⁻¹² m²] AT 3 kPa CONSOLIDATION	PERMEABILITY [10⁻¹² m²] AT 6 kPa CONSOLIDATION	PERMEABILITY [10⁻¹² m²] AT 9 kPa CONSOLIDATION
INFANT	19.8	18.1	17.4
TODDLER	9.8	9.1	8.5

Table 10: Results of the permeability measurements

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MCR Evolution Series
Powder flow & shear cell

Vapor sorption

Water vapor sorption is a property or phenomenon related to a powder's ability or propensity to absorb water molecules from the atmosphere. Producing materials in large quantities and shipping them to distributers for sale can involve long storage times with significant water vapor absorption, which producers must ensure does not affect the substance's quality. Appropriate packaging is essential to avoid influences from outside environmental conditions. But before the product is packaged into its final wrapping, it has already interacted with the environment. In addition, the customer may not use the whole product at once, but rather store it after opening.

An especially influential environmental factor for powder properties is the relative humidity. It varies at different geographic locations and between different buildings and outdoors.

Furthermore, the shelf life of materials such as milk powders can be influenced by both relative humidity and temperature.

Determining the amount of water adsorbed into a material such as milk powder can be measured using a static volumetric vapor sorption analyzer. This type of instrument can provide specific fixed or variable temperatures and relative humidities (RH) that mimic storage conditions and reveal how much water uptake or release occurs throughout a specific time period. The vapor sorption experiments below were carried out on dry milk powder samples at 95 % RH at temperatures of 25 °C, 35 °C, and 45 °C. With increased exposure time to 95 % RH, the

sample mass increased. The mass change in percent is depicted in Table 11.

TIME	MILK POWDER MASS CHANGE		
24 h	21.72 % (25 °C)	24.72 % (35 °C)	24.78 % (45 °C)

TEMPERATURE	MASS INCREASE IN %
25 °C	24
35 °C	24.72
45 °C	24.78

Table 11: Milk powders' % mass change at 95 % relative humidity at given temperatures

All samples increased by 20 % to 25 % of their original mass after 24 hours of exposure to 95 % RH at different temperatures. Higher uptake amounts were achieved with higher temperature, suggesting that storage of milk powders at lower temperatures reduces their likelihood of adsorbing excess moisture.
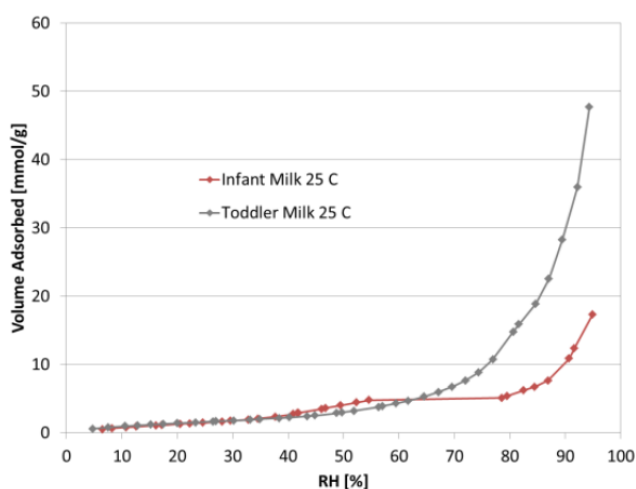

Figure 21: Water vapor adsorption isotherms of milk powders at 25 °C

The full water sorption isotherms of the infant and toddler formulas were measured at 25 °C and are shown in Figure 21. Clear differences are seen between the two formulas with the toddler formula adsorbing more water at higher RH. In addition, the infant formula isotherm shows an unusual step in the adsorption branch starting between 50 RH% – 60 RH% that is most probably related to a lactose phase transition, confirming that the two formulas have different compositions.

INSTRUMENT SUITABLE FOR THESE MEASUREMENTS
VSTAR Series

4.2 VISCOSITY AND MOUTHFEEL OF FOOD POWDERS

The final infant/toddler milk needs to meet various criteria concerning quality. Apart from nutritional aspects, quality is further reflected in the texture and stability of the milk. Additionally, its flow behavior when being filled into a bottle and especially when it is sucked out of the bottle by the child is of great importance when judging the experience and effort needed to draw the liquid. This, in turn, is dependent on the particulates present within the dissolved formula which can change according to the nutritional needs of the child. The liquid's flow through the bottle into the child's mouth, as well as the intensity of sucking required, differs depending on the viscosity and flow behavior of the milk product. The viscosities at shear rates which are representative of the milk flowing out of the small hole in the feeding bottle provide important information. Furthermore, viscosity in general plays a role in the formula's texture and its dispersion stability.

The methods for characterization of powder and liquid dispersion presented in this report will guide users to describe the characteristics of milk powder and its final milk product. The flow curves in Figure 22 reveal different flow behavior for the two liquid milk samples. Infant milk showed only slight shear-thinning flow behavior while the toddler milk was significantly shear-thinning (more than an order of magnitude decline in viscosity).

Further, the absolute viscosity values of the toddler milk were consistently higher than those of the infant milk.
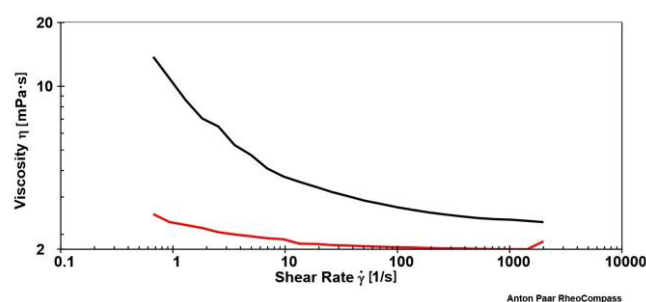

Figure 22: Flow curve of infant milk (red) and toddler milk (black) at 37 °C

The almost Newtonian flow behavior of the infant milk can be explained by the uniform particulate size distribution in the solution. The highly shear-thinning behavior of the toddler milk is likely due to the insoluble carbohydrate ingredients like maltodextrin in the milk powder.

Considering the geometry of a baby bottle, the tip of the nipple is the most sensitive part where most of the shear rate is applied.

Considering the high shear rate applied, designing formula to be shear-thinning (advertently or inadvertently) is a prudent decision. Also considering the stronger muscular development of toddlers versus infants, a higher viscosity overall might be desirable as well.

INSTRUMENT SUITABLE FOR THESE MEASUREMENTS
ViscoQC Series
MCR Evolution Series

4.3 FOOD SAFETY

Acid digestion of food powders to prepare samples prior to ICP-MS testing for metal contamination

Industrially produced food products and their primary agricultural products are amongst the best-characterized and continuously monitored substances of daily life. Because of the impact of food quality on human health, chemical analysis is required by law in most countries. Trace element analysis, e.g., for toxic heavy metals like lead, arsenic, cadmium, or mercury, is a routine task in industrial and governmental food testing labs.

In order to demonstrate the excellent suitability of Multiwave 7000 for sample preparation of food and other organic samples prior to element analysis the recovery rates of certified reference materials were determined. The described conditions can be used as a starting point for different kinds of organic samples.

Approximately 0.5 g of the sample were multiply weighed into the 18 mL quartz and PTFE-TFM vials.

The vials were put into Rack 18 and 1 mL of H_2O and 4 mL of conc. HNO_3 (65 %) were added. After closing the vials with the plug-on caps the rack was put into the liner already filled with load solution (150 mL of water and 5 mL of conc. HNO_3).

In two consecutive runs (one with quartz vials only and the other one mixed with quartz and PTFE-TFM vials) the liner was put into the Pressurized Digestion Cavity (PDC) and the digestion program was started. After cooling, 1 mL of conc. HCl was added for stabilization of Hg. Subsequently the samples were transferred into 50 mL tubes, filled up and analyzed. Prior to measurement the solutions were diluted 1 to 10 with distilled water.

The measured values are well-comparable with the certified values and the results were similar when the digestions were performed in either quartz or in PTFE-TFM vessels.

Element	Reference Value [mg/kg]	Measured Value [mg/kg]	Recovery Rate [%]
Ag	0.666 ± 0.009	0.688 ± 0.009	103 ± 1
As	7.65 ± 0.65	7.63 ± 0.12	100 ± 2
Ca	838 ± 20	887 ± 19	106 ± 2
Cd	2.48 ± 0.08	2.52 ± 0.02	102 ± 1
Co	0.371 ± 0.009	0.353 ± 0.008	95 ± 2
Cu	71.6 ± 1.6	70.5 ± 0.5	98 ± 1
Fe	205.8 ± 6.8	203.3 ± 1.4	99 ± 1
Hg	0.0371 ± 0.0013	0.0328 ± 0.0011	89 ± 3
K	6520 ± 90	6135 ± 70	94 ± 1
Mg	1085 ± 23	1110 ± 12	102 ± 1
Mn	18.5 ± 0.2	18.9 ± 0.2	102 ± 1
Na	3297 ± 53	3069 ± 34	93 ± 1
Ni	1.04 ± 0.09	0.975 ± 0.021	94 ± 2
Pb	0.308 ± 0.009	0.307 ± 0.005	100 ± 2
Rb	3.26 ± 0.14	3.02 ± 0.05	92 ± 1
Se	2.06 ± 0.15	2.11 ± 0.20	102 ± 10
Th	0.0367 ± 0.0043	0.0359 ± 0.0016	98 ± 4
V	0.577 ± 0.023	0.552 ± 0.025	96 ± 4
Zn	1424 ± 46	1441 ± 14	101 ± 1

Table 12: NIST1566b, Results and recovery rates, n=15 Quartz:10, TFM:5

The applied digestion method is not only suitable for the digestion of the mentioned reference materials (spinach leaves and oyster tissue) but also serves as a representative starting point for any kind of organic sample, whereas relevant parameters such as sample weight, time and temperature might be adapted accordingly.

INSTRUMENT SUITABLE FOR THESE MEASUREMENTS
Multiwave 5000
Multiwave GO Plus

5 Anton Paar Solutions

MCR Evolution Rheometer Series

The MCR Evolution series is the result of consistent thinking and rethinking, of continuous development based on decades of experience, and of the feedback of more than 10,000 satisfied customers. The combination of innovative and thousandfold field-proven technology with the modular design represents the benchmark in its class. With 200+ accessories, the MCR Evolution rheometer series gives you endless possibilities for rheological investigations and material characterization.

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The powder shear cell is ideal for determining the flow behavior of consolidated powders and their time-dependent behavior. Additional accessories provide full control over temperature and humidity.

The powder flow cell is an innovative and scientific approach to powder characterization offering a wide range of test methods. Powder behavior can be characterized under various processing conditions: simulating, adjusting, and optimizing, e.g., manufacturing processes.

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Nova Series

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Ultrapyc Series

Ultrapyc gas pycnometers measure the true and skeletal density of solids to track their purity and porosity. Measurements take less than 10 minutes, so they are perfect to control the quality of your solid materials throughout the manufacturing process. The PowderProtect mode enables measurements of fine powders without instrument contamination and the built-in Peltier temperature control ensures superior thermal stability with no external temperature control bath required.

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> www.anton-paar.com/eb-food-ultrapyc

Autotap Series

These one- and two-station tapped density analyzers for powders, granules, and small pellets are automated devices that comply with various internationally recognized standards. They provide high-level test method control with a user-selectable, lockable number of taps. A large range of sample sizes can be accommodated with different graduated cylinders.

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XRDynamic 500

This automated multipurpose powder X-Ray diffractometer drives unbeatable XRD data quality with maximum efficiency. It is a versatile platform covering a wide variety of applications with optimal solutions for powder XRD, non-ambient XRD, PDF analysis, SAXS, and more. Intuitive to use, with fully automated optics and alignment routines, it allows everyone, from novices to experts, to collect top-quality XRD data quickly while minimizing errors. XRDynamic 500: Driving XRD.

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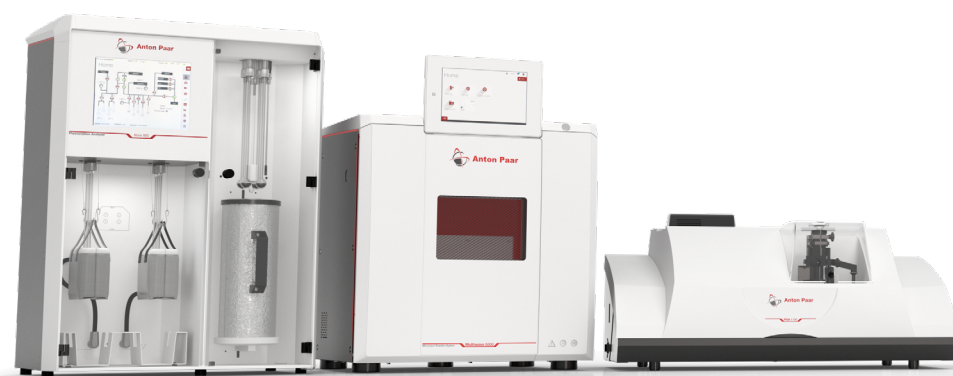
Multiwave 5000

This microwave digestion platform provides digestion of a broad array of samples (varying in difficulty or volume), acid leaching, solvent extraction, evaporation, microwave-induced O_2 combustion, sample drying, and synthesis. Furthermore, it comes with a comprehensive, interactive method library, a customizable, intuitive user interface, and modern sensor technology, together with easy-to-handle compact rotors.

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eb-food-multiwave-5000

Multiwave GO Plus

The Multiwave GO Plus microwave digestion system with the patented Directed Multimode Cavity (DMC) provides the best of both monomode and multimode microwaves. The microwave field adapts itself to the number of filled or empty positions and the filling state of the vessel – that's truly innovative. Due to the TURBO cooling process, unique cooling times as short as eight minutes for a fully loaded twelve-position rotor are possible (for EPA methods).

Find out more:
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eb-food-multiwave-go-plus

RapidOxy 100

Fully-automatic measurements 8x faster than other accelerated aging methods, at temperature ranges of up to 180 °C. Easy cleaning in less than 5 min. RapidOxy100 is the only Rapid Small-Scale Oxidation Test (RSSOT) on the market. Another bonus: the small 5 mL / 4 g sample size max. – especially helpful when costly samples are being investigated.

Find out more:
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ViscoQC rotational viscometers ensure the quality of substances – from liquid to semi-solid. Eliminate the risk of measurement errors and free up time and budget with the simplest spindle exchange mechanism, automatic spindle detection, automatic speed searching function for new formulations, and more. All of these features have been developed to make the operator's life easier.

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With the laser diffraction technology of the PSA series, you can determine the particle size and particle size distribution of both liquid dispersions and dry powders from the nanometer up to the millimeter range. Since these two parameters have an impact on processability as well as the properties of the final product, understanding them is essential for product development and quality control.

Find out more:
www.anton-paar.com/eb-food-psa

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