Food characterization

Relevant for: Characterization of Powders and Granular Media,
Important for: Food Industry

Food powder formulation, manufacturing, and packaging processes require batch-to-batch consistency to ensure consumer safety and loyalty. Insights derived from experimentation using Anton Paar instruments that determine the density, particle size, cohesion strength, compressibility, and permeability can contribute to food powder quality and consistency. This application report focuses on milk powder and all-purpose flour because they are ubiquitous products on their own and crucial ingredients in many other food products and nutritional supplements.

1 Introduction

Consistency in food quality ensures consumer safety and loyalty. Repeatable quality is easily achievable by utilizing Anton Paar instruments that measure physical properties of foods. This report focuses on the physical characterization of powders because they are the basic ingredients of most foods. In fact, solid, liquid and even gelatinous foods can be transformed (e.g., crushed, pulverized, or dehydrated) to powders yet retain the essence of the food. Characterization of the powders helps ensure consistency from batch to batch and can lead to more confidence in food quality and savings in terms of formulation, reformulation, packaging, storage, and transport.

2 Density, Rheology, & Particle Size

Precise knowledge of food powder composition leads to dependable formulation — it is easy to identify ingredients; it is a challenge or trade secret to know how to mix ingredients together to produce a final product that will be enjoyed reliably. Anton Paar offers a versatile toolbox for characterization of food powders:

Table 1: Solid Density Analyzers

Instrument	Measures	Description
AutoTap	Tapped Density	Powder compressibility under constant force
UltraPyc 1200e	Skeletal/True Density	Mass to volume ratio

Table 2: MCR Rheometers

Instrument	Measures	Description
MCR 302 with powder flow cell	Compressibility	Percent change in powder volume when experiencing a load
	Cohesion Strength	Interaction between particles
	Flowability	Capacity of particles to flow
	Permeability	Resistance to air flow

Table 3: Particle Size Analyzers

Instrument	Measures	Description
PSA *1190*	Particle Size Distribution via Free-fall	Agglomeration analysis
PSA *990,1090,1190*	Particle Size Distribution via Venturi Mode	Primary particles
LiteSizer *500*	Particle Size Distribution via Dynamic Light Scattering	Stability Particle size of food ingredients

The Anton Paar Research Center conducted experiments using particle characterization instruments on dry food powders including milk powder (infant and toddler formula) and all-purpose flour.

3 Results

Using a representative quality control process, experiments were conducted to first measure the tap density with the Autotap. Next, skeletal density was determined using an UltraPyc 1200e. Results from these tests were supplemented with additional sophisticated particle size and rheology analysers.

3.1 Tapped and Skeletal Density

The initial filling densities for the milk powders were similar at 0.43 and 0.42 g/cm^3 for infant and toddler milk powder formulas. After tapping, following the ASTM 7481-18 standard procedure, they reached respective tapped densities of 0.56 and 0.55 g/cm^3. This change in tapped density allowed its compressibility to be determined, yielding 24.3 and 25.0. The all-purpose flour displayed an initial filling density of 0.56 g/cm^3 and a final tapped density of 0.71 g/cm^3. The compressibility equalled 21.1.

For the milk powders, the skeletal densities of 1.19 and 1.08 g/cm^3 were nearly twice the tapped density values for both. The ratio of tapped to skeletal density equalled 0.47 and 0.51, suggesting that these particles are far from spherical. The all-purpose flour skeletal density was determined to be 1.50 g/cm^3. The tapped to skeletal density ratio was 0.47.

These results suggest that both food powders have passable flowability and are materials conducive to uninterrupted process streams.

3.2 Particle Size

Milk powder: Figure 1 shows the differences in particle size distribution in the infant and toddler milk powder samples. The primary aggregates (measurements in Venturi mode) of the toddler formula are found in a smaller size range than the primary particles of the infant formula.

Interestingly, the analysis of D10, D50, and D90 in Free-fall mode indicated only small size differences between the aggregates of the two powders (refer to Table 4). Meanwhile, a shift towards a smaller size range for the primary particles in the case of the toddler formula was observed.

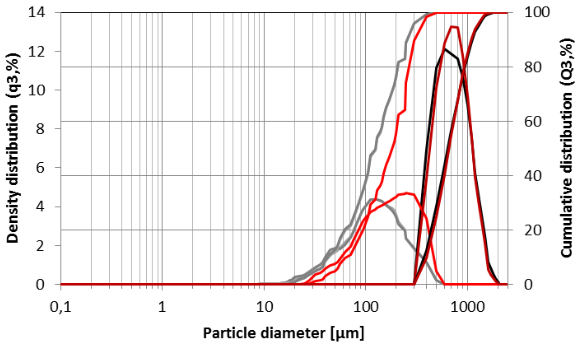

Figure 1: Particle density (q3,%) and cumulative distribution (Q3,%) of infant formula measured via the Venturi mode (light red) and Free-fall mode (dark red) & toddler formula measured by the Venturi mode (grey), and Free-fall mode (black).

Table 4: Summary of D-volume weighted data of laser diffraction for the infant (I) and toddler (T) formula

Infant (I) and Toddler (T) Formula				
		D10 [µm]	D50 [µm]	D90 [µm]
Venturi	I	72	189	402
	T	48	130	315
Free-fall	I	501	765	1193
	T	478	746	1222

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 will reconstitute faster in water. Moreover, the flowability will be also better than the toddler milk because of the bigger agglomerate size measured in Free-fall.

3.3 Rheology

Flour: The compressibility measurements provide information about the bulk density (consolidated and unconsolidated) of the sample after treatment/storage at increased humidity and different conditioning times. Figure 2, reveals that increased exposure to 95% RH at 35 °C decreases the bulk density. Thus, the longer the flour was exposed to 95% RH at 35 °C, the higher was the porosity of the flour. This is attributed to an increase in particle size.

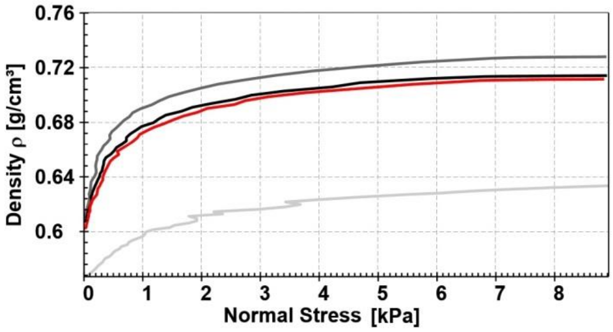

Figure 2: Compressibility measurements of flour samples after 0 (dark grey), 1 (black), 2 (red) or 24 h (light grey) at 35 °C and 95% RH.

Overall, an increased moisture content of the flour decreases the achievable bulk density upon compression. This is indicative of a higher intergranular friction as well as angle of repose increase. These phenomena are mirrored in the Cohesion analysis. This is true for different exposure times in the environmental chamber as well as for different pre-treatment procedures prior to exposure to high humidity and temperature.

3.4 Warren Spring cohesion

Flour: The Warren Spring method is applied to measure cohesion in especially cohesive powders such as flour. An example measurement (with multiple repetitions) is displayed in Figure 3. The curve maximum represents the value of the Warren Spring cohesion strength. A sharp decrease of cohesion strength after its peak indicates a fast breaking of the sample. In contrast, a wide peak and gentle decrease of cohesion strength over time indicates a slow-breaking powder. In this case, we see an intermediate behavior, with cohesion strength forming a peak neither sharp nor wide, confirming the applicability of the method.

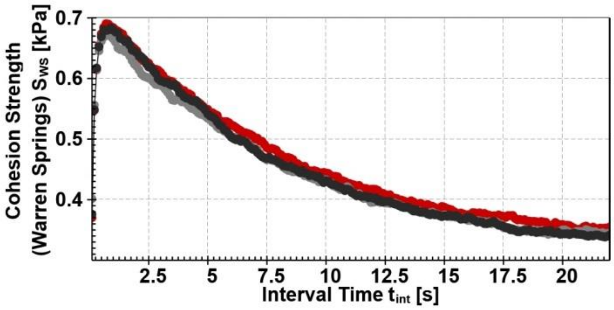

Figure 3: Reproducibility of Warren Spring cohesion measurements at 3 kPa of flour samples (pre-treatment: 90 °C overnight).

Longer pre-treatment of the flour sample in the environmental chamber raised the Warren Spring cohesion strength (refer to Figure 4). The increased moisture content of the sample promotes the formation of capillary bridges between the particles. Furthermore, the altered surface chemistry of the starch granules (water uptake) leads to a marked increase of internal friction and therefore to enhanced cohesion. Thus, the elevated Warren Spring cohesion is caused by multiple factors, all contributing to a marked change in flow behavior.

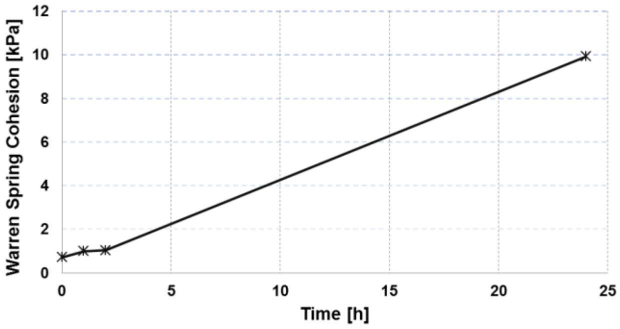

Figure 4: Warren Spring cohesion of flour samples after 0, 1, 2 and 24 h in the environmental chamber.

3.5 Permeability

Milk powder: Figure 5 and Table 5 show the permeability results, representing the powders' air flow resistances under different compaction forces. The infant formula is notably more permeable and more dependent on the compaction as shown by the significant change of permeability with different compactions.

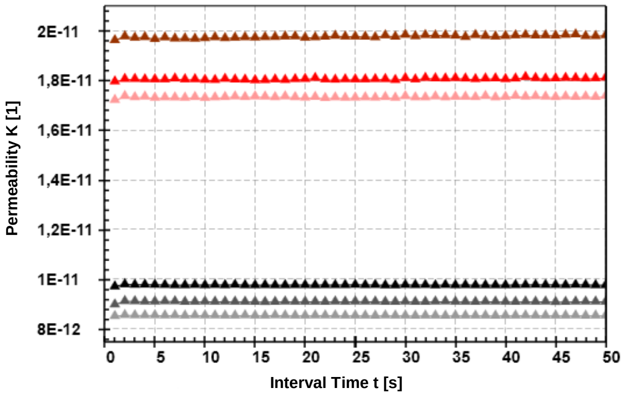

Figure 5: Permeability – toddler formula (black to gray curves) and infant formula (red curves) measured at compressions of 3 kPa, 6 kPa and 9 kPa (from darkest to lightest).

Generally, the permeability decreased with increasing normal stress during compression, and the infant formula showed significantly higher permeability (by more than a factor of 2) 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 shows the vulnerability of some powders to compaction (as seen with flour in Figure 2). 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.

Table 5: Results of the permeability measurements

Sample	Permeability $[10^{-12} m^2]$ at 3 kPa consolidation	Permeability $[10^{-12} m^2]$ at 6 kPa consolidation	Permeability $[10^{-12} m^2]$ at 9 kPa consolidation
Infant	19.8	18.1	17.4
Toddler	9.8	9.1	8.5

3.6 PSA on humid samples

Flour: In the case of flour samples, the purpose of particle size measurements is to investigate the process of agglomeration and how it impacts the material's flowability. Figure 6 shows the changes in particle size distribution (PSD) due to the formation of agglomerates during conditioning time. A shift of peaks towards a higher size range and a rearrangement of the peaks position occurred due to the increase in agglomeration events.

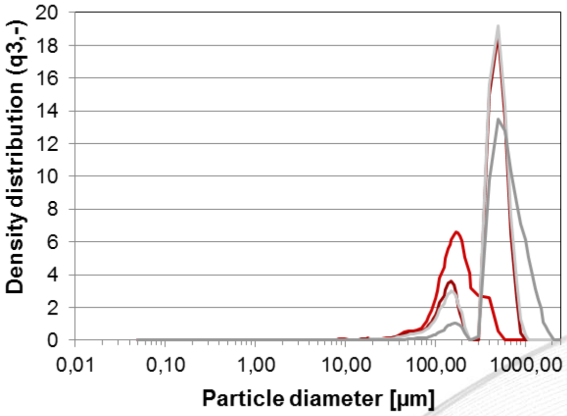

Figure 6: Particle density distribution (q3,-) of the unconditioned flour sample (dark red) and of the flour after 1 h (red), after 2h (light grey), after 24 h (dark grey) conditioning at 35 °C and the humidity to 95 % in the environmental chamber measured in Free-fall mode.

The peak detected around 300 µm in the unconditioned sample is due to starch-protein aggregates. After conditioning, the starch begins to dissociate from the proteins, swells and builds agglomerates with other single starch granules. This phenomenon is responsible for the formation of particles larger than 300 µm.

The values of D_{10}, D_{50}, and D_{90} are listed in Table 6 and, as expected, increase with conditioning time.

The D-weighted values and the PSD curves of the samples subjected to 1 or 2 h of conditioning do not appear significantly different. However, after 24 h of conditioning, the sharp increase in particle size clearly points to the formation of bigger agglomerates.

Table 6: Summary of D-volume weighted data of laser diffraction showing the agglomeration over time from Free-fall

Conditioning	D10 [µm]	D50 [µm]	D90 [µm]
None	92.3	181.9	388.4
1 h	132.2	509.6	696.7
2 h	148.1	519.1	726.9
24 h	418.4	640.6	1143.8

The particle size distributions returned by Venturi mode measurements (Figure 7) differ greatly from that of Free-fall measurements. These lack large (> 600 µm) particles and are essentially monomodal, which suggests that the Venturi unit promotes the dispersion of aggregates.

The distributions obtained for unconditioned and conditioned (24 h) samples are similar, but a slight shift towards larger particle sizes can be visualized in the cumulative distribution overview for the conditioned sample.

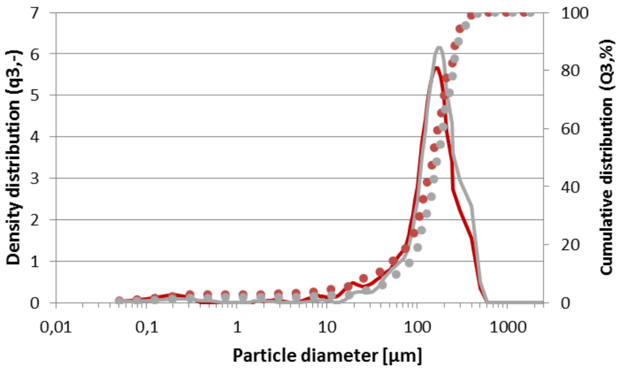

Figure 7: Particle density distribution (q3,-) and cumulative distribution (Q3,%) of the unconditioned flour sample (red) and after 24 h (grey) conditioning at 35 °C, 95 % RH in the environmental chamber measured in Venturi mode.

The increase in D_{10}, D_{50}, and D_{90} values, though clearly detectable (refer to Table 7), is also limited.

As the air pressure and the passage through the Venturi tube causes the dispersion of agglomerates, the effect of moisture and temperature on primary particles can be observed. Thus, the remaining shift is thought to be resulting from the swelling of single starch granules in response to water uptake.

Table 7: Summary of D-volume weighted data of laser diffraction in Venturi mode

Conditioning	D10 [µm]	D50 [µm]	D90 [µm]
None	38.1	157.3	340.9
24	67.6	178.8	376.3

3.7 Litesizer

Milk powder: Both formulas contain milk, plant oils and various minerals. The fat content causes the detection of peaks between 100nm and 10µm in both formulas. However, the higher content of lactose and the presence of maltodextrin determine the peak at 2179 nm in the toddler formula (refer to Figure 8).

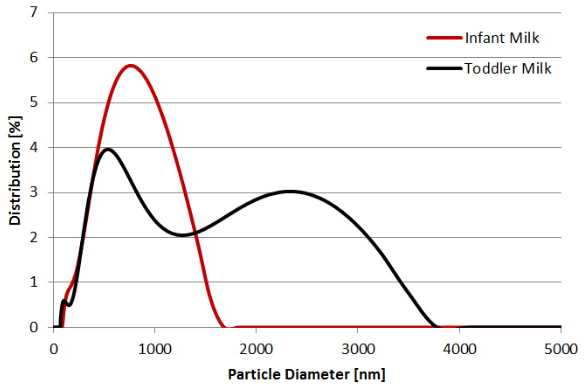

Figure 8: PSD of infant (red) and toddler (black) milk powder.

4 Conclusion

The Anton Paar Research Center investigated physical characteristics of milk powders and all-purpose flour using solid density, particle size, and rheology instrumentation. This multi-instrument methodology provides a reliable, repeatable quality testing process of food powders to help ensure batch-to-batch consistency leading to consumer safety and loyalty.

Foods are just one class of powders. These techniques can be relevant to other powders.

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