Chocolate: Particle Characterization from Bean to Bar

AN138

Chocolate. The champagne of culinary treats. Long craved by anyone with a sweet tooth, it has blossomed into thousands of products, all to share that sweet flavor.

Chocolate has a rich history. Fermented beverages made from chocolate in Mesoamerica date back to 350 BC. The Aztecs believed that cacao seeds were the gift of Quetzalcoatl, the god of wisdom, and the seeds once had so much value that they were used as a form of currency.

Particle size characterization is critical to the quality of manufactured chocolate products.

Introduction

The chocolate production process today is a marriage between technology and craftsmanship. Generally, the presence of particles above 30 µm leads to poor mouth-feel. It is perceived as gritty. Meanwhile, particles below 20 µm feels silky and smooth [1]. Below 30 µm, 2-3 µm of a particle size difference can be detected by the tongue as a different level of smoothness [1]. Therefore, monitoring, standardizing and setting specifications for particle size testing is a critical and necessary measure for quality control across chocolate plants.

Careful control of particle size is what sets premium chocolate apart from lower quality chocolate candies. It also allows scaling, while maintaining a consistent taste profile. In this note, we will compare chocolate bars from small, medium, and large scale chocolate manufacturers from the perspective of particle size and shape.

Production Process

It all starts with cocoa beans. After beans are harvested, fermented and roasted, beans are de-shelled into nibs. Nibs, chocolate in its purest form, are then ground and heated to free up cocoa butter into 100 percent chocolate liquor. Next, sugar, milk, and more cocoa butter are added to the chocolate liquor before it is aerated. That results in the further reduction of particle size, a very important process known to the industry as conching.

The primary purpose of conching, just as milling of nibs, is to elevate mouth-feel. And depending on a bean's origin, batch size, and cocoa content, conching time for one batch of beans may differ from a few hours to a few days just within one manufacturer. It may also differ even more greatly between high and low-end chocolates.

Before liquid chocolate is molded and scored into bars, it undergoes a tempering process where repeated heating and cooling allows fat to crystallize into several different sizes and shapes. The tempering step affects the surface glossiness and snap of the final product – for example, the white spots on chocolate bars are those larger, less stable crystals of cocoa butter. By tightly controlling how fat crystals re-align and interlock into a chainlike structure, and subsequently, its fat particle shape during tempering, it is possible to control the flow behavior of chocolate suspensions. And therefore create a low fat chocolate without jeopardizing taste [2].

Analysis Techniques

The traditional method of measurement, the Hegman gauge or grindometer (Figure 1), is a simple device that measures the degree of dispersion in viscous suspensions. The Hegman gauge only detects the presence of the largest particles or aggregates, and does not determine particle size or particle size distribution. It therefore, only applies to sizes above 12.5 µm or 7 Hegman [4].

The device requires users to manually spread a suitable amount of suspension by means of a scraper on a calibrated tapered groove. At a constant speed with sufficient pressure, users will then inspect the result at a 20-30 degree angle from the gauge surface. As a result, the operation is highly subject to user errors and variability. So while a Hegman gauge is a low-priced option for chocolate particle sizing, the device accuracy (±2 µm) is merely as sensitive as the human tongue [1, 4].

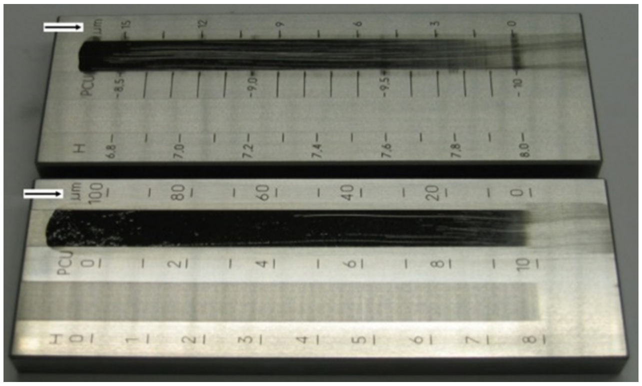


Figure 1. Hegman Gauge (grindometer) is a simple device that measures particle size of viscous suspensions [3] in a qualitative approach.

On the contrary, laser diffraction (for size) and digital image analysis (for size and shape), are techniques with little manual input. Laser diffraction is a mature and established sizing technique due to its ease of use, high throughput and high reproducibility.

Users begin with melted chocolate and a solvent, such as Isopar G or sunflower oil for pre-dispersion. After basic sample information is entered in the software, the pre-dispersed chocolate sample is then added drop by drop into the laser diffraction chamber (already filled with the dispersion medium) until an appropriate volumetric concentration (approximately 85-95% of laser transmittance) is reached. This process is guided in real time by the user interface. The report is displayed within seconds. The size analysis result offers a good correlation with consumer acceptability at various stages of the production process, and is thus a step up from Hegman gauge.

The image analysis technique, on the other hand, is an excellent tool to identify processing (e.g. tempering) or compositional (e.g. sugar, fat or milk powder content) differences due to its ability to "see" and record particle shape parameters. Similar to laser diffraction, pre-dispersed chocolate samples are slowly added until a good concentration is reached before measurement. Users can visualize individual particles flowing through the measurement cell in real time. For R&D, this is a natural step up from the laser diffraction technique.

Case studies are presented below.

Materials and Methods

Laser Diffraction

Three brands of chocolate from large, medium, and small chocolate factories were acquired and tested to showcase useful information provided by a HORIBA **Partica mini LA-350 Compact Routine Laser Diffraction Analyzer**. The following analytical test method was used:

Refractive Index: 1.59 | Imaginary (absorption): 0.1i
Dispersant fluid – Isopar G
Circulation speed – 3, without ultrasonic

Once the analytical test method was determined to be repeatable, reproducible and robust, a standardized chocolate "sequence" file was saved into the LA-350 software. The sequence then was used as a one-button auto operation for all chocolate measurements.

Image Analysis

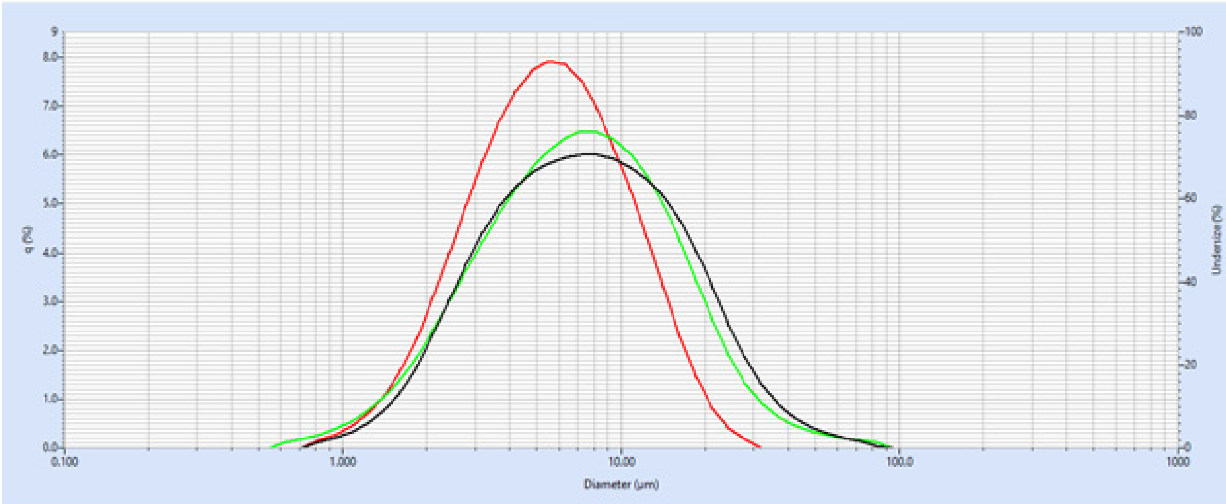
Researchers tested a baking chocolate and a regular dark chocolate of the same brand with the **CAMSIZER X2 Dynamic Image Analysis System Particle Size and Shape Analyzer** with the following analytical test method:

Hardware	With X-flow
Nominal covered area	0.1%
Image rate	100% (1:1)
Stop measurement after	20,000 images
Fill transparent particle	On
Pump speed	5, without ultrasonic
Dispersion Fluid	Isopar G

Results and Discussion

Laser Diffraction – Size Comparison

Three plain dark chocolates with the same cocoa concentration manufactured by different size chocolatiers were analyzed. "Large chocolate factory" (**red**) is a HORIBA laser diffraction user, whereas small (**black**) and medium (**green**) chocolate factories' sizing methods were unknown.

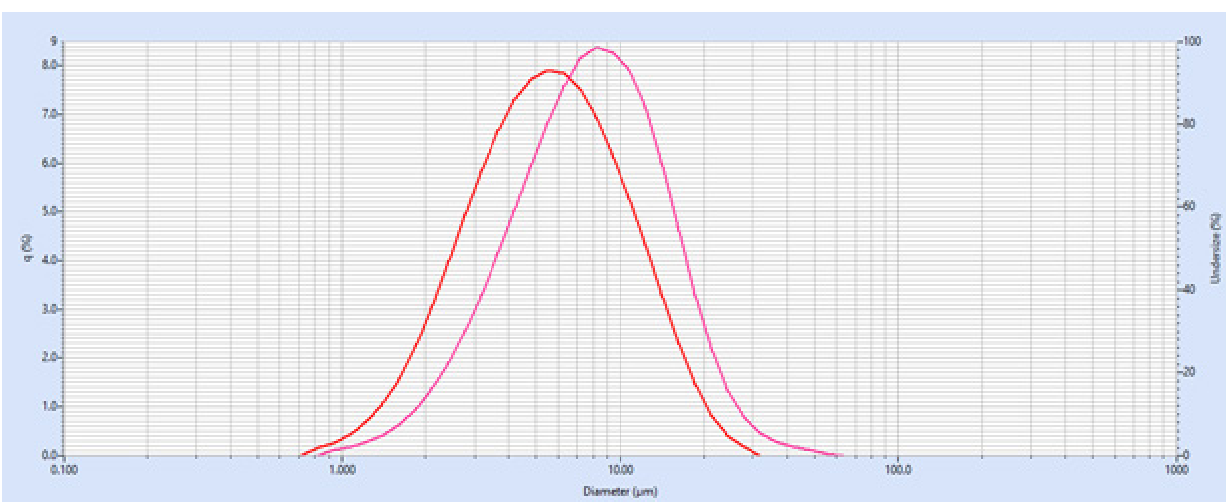


Data name	Graph type	D(v,0.1)	D(v,0.5)	D(v,0.9)	St. Dev.
Large Chocolate Factory		2.31211 (µm)	5.50367 (µm)	12.56218 (µm)	4.3014 (µm)
Medium Chocolate Factory		2.38831 (µm)	7.03215 (µm)	18.97867 (µm)	8.7815 (µm)
Small Chocolate Factory		2.59905 (µm)	7.40903 (µm)	21.13812 (µm)	9.0413 (µm)

Figure 2. The particle size distributions of dark chocolates from three different manufacturers display different Dv90 values and distribution widths.

As seen in Figure 2, dark chocolates from the three separate factories have similar Dv10 (2.31 µm to 2.60 µm), but differ greatly at the coarse end of the distribution (Dv90 of 12.56 µm to 21.14 µm). This disparity is likely due to different milling conditions. In addition to size, the "large chocolate factory" displayed the narrowest (St. Dev. 4.30 µm) particle size distribution, indicating that the particle size is more tightly controlled during production process compared to its counterparts. Chocolate with a narrower distribution width and fewer particles sized above 30 µm [2] tend to be perceived as luxurious and smooth, as it "hugs" the tongue*.

**Our very own HORIBA Scientific team had a blind taste of the chocolate samples. While no one was a trained chocolate connoisseur and that the sampling pool was relatively small, the verdict was unanimous: chocolate from the small chocolate factory was rated the worst. An interesting finding!*



Data name	Graph type	D(v,0.1)	D(v,0.5)	D(v,0.9)	St. Dev.
Large Chocolate Factory		2.31211 (µm)	5.50367 (µm)	12.56218 (µm)	4.3014 (µm)
Chocolate Liquor-Large Chocolate Fa		3.18514 (µm)	7.79116 (µm)	16.62825 (µm)	5.9672 (µm)

Figure 3. An overlay of two size distributions (chocolate liquor (pink) and final chocolate product (red)) before and after conching and tempering.

The effect of conching and tempering from the "large chocolate factory" can also be observed by the LA-350 in Figure 3 when the particle size distribution of chocolate is shown adjacent to its predecessor, chocolate liquor. This chocolate liquor was collected from the same processing batch, provided by the "large chocolate factory." Note that the entire distribution shifted towards the fine end with conching and tempering, but still retained the same particle size distribution shape.

Dynamic Image Analysis – Size and Shape Comparison

Next, one baking (**green**) and one regular dark chocolate (**red**) from the same chocolate factory were tested to compare processing and composition.

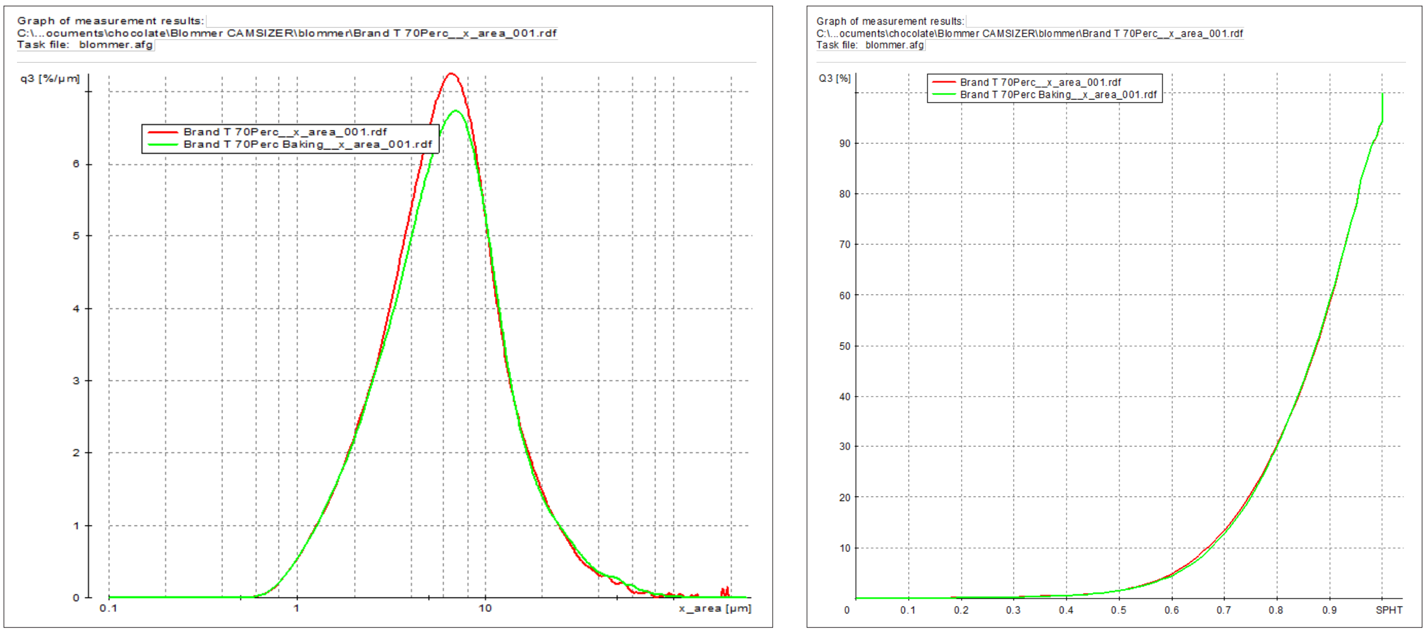


Figure 4. Particle size (left) and shape (right) comparison for two chocolates, one marketed and priced as baking chocolate (green) and the other marketed and priced as regular chocolate (red).*

	Regular chocolate	Baking chocolate
Dv10	4.2 µm	4.2 µm
Dv50	9.6 µm	9.6 µm
Dv90	34.6 µm	35.4 µm
Mean value SPHT3	0.841	0.846

An overlay of the particle size distributions, expressed in volume weighted, is shown on the left in Figure 4 and shape distribution, expressed in degree of particle roundness (SPHT), is shown on the right. One chocolate was marketed and priced lower as baking chocolate while the other was marketed as the regular chocolate. Yet the size analysis result showed little to no difference between the two. Both samples also had the same spherecity, which suggests analytically that the packing strength of the chocolates do not differ much from one another. In this case, the data concluded that baking and regular chocolate were identical.

So, what went wrong? The result contradicted the original intent and assumption that baking chocolate differed from regular chocolate.

We reached out to the manufacturer representative. They later confirmed after the analysis that both their baking and regular chocolates are indeed, exactly the same!

Conclusion

In this study, both laser diffraction and image analysis have proven to be useful in answering the question why one brand may be preferred over another. The LA-350 compact routine laser diffraction analyzer is ideal in determining mouth-feel by scrutinizing particles above 20 µm (silky) and above 30 µm (gritty). Its built-in sequence measurement can also be used as a standardized test method across different chocolate plants, thereby, removing operator errors and making quality control possible for scaling. The CAMSIZER X2 dynamic imaging system, on the other hand, is suitable for identifying formulation differences and the packing strength of final chocolate products.

References

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