

Application News

Laser Diffraction Particle Size Analyzer SALD-2300
Dynamic Particle Image Analysis System iSpect DIA-10

Evaluation of Particle Size in Chocolate —Evaluation of Particle Size Distribution and Coarse Particles that Affect Texture—

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User Benefits

- ◆ The solid particle size distribution in chocolate can be easily measured using the SALD-2300.
- ◆ The iSpect DIA-10 enables not only the measurement of particle size distribution but also, the number concentration of particles in a specified particle size range.

■ Introduction

Particle size distribution is one of the physical properties that describe the characteristics of materials and products containing particulate matter. In some cases, the difference in characteristics can be described simply by the average particle size. In other cases, the amount of particles above a certain size is important. The values to be noted vary depending on the type of material, product, and its intended use.

Here, chocolate is taken as an example of a product where particle size control is important. An example is presented of measuring the particle size distribution and the quantity of particles within a specific size range to assess the texture of chocolate.

When performing chocolate quality control, the particle size distribution is generally evaluated using a laser diffraction-type particle size distribution analyzer. In this article, in addition to using the SALD-2300 Laser Diffraction Particle Size Analyzer, the iSpect DIA-10 Dynamic Particle Image Analysis System is also employed to show the possibility of a slightly different evaluation approach to investigating the relationship between particle size/concentration and texture.

■ Measurement of Particle Size Distribution by SALD-2300

Chocolate is solid at room temperature. Therefore, it must be dissolved (dispersed) in a solvent to measure the particle size distribution of the solid particles contained within it. Samples must also be diluted to a concentration suitable for the particle size distribution measuring analyzer.

Isopropanol was selected as the dispersant because it can dissolve oil and fat and disperse the solid particles to measure the particle size distribution of just the solid particles in the chocolate.

The dispersion and measurement conditions are shown in Tables 1 and 2.

Table 1 Dispersion Conditions

Dispersion medium:	Isopropanol
Dispersant:	None
Dispersing Procedure:	About 0.1 g of thin slices of chocolate were scraped from a chocolate block. Then, they were placed in a 50 mL beaker, and 20 mL of isopropanol heated to about 40 °C was added, and the sample was irradiated for 5 minutes in a 240 W ultrasonic bath.

Table 2 Measuring Conditions

Equipment:	SALD-2300
Accessories:	Batch Cell (Sample Suspension is stirred while measurement is performed)
Refractive Index:	1.70-0.02i

Two commercial milk chocolate samples were used: sample F and sample UF. Taste tests were conducted by several individuals (including the author), and comparisons were made prior to measurement. The taste tests focused on texture differences and the smoothness on the palate and tongue.

The results showed that although there were differences among individuals, most perceived sample UF as smoother than sample F, while sample F felt slightly rough.

Fig. 1 shows the SALD-2300 particle size distribution measurement results. The particle size distribution curves of the two types of chocolate are superimposed. Four curves are drawn since each sample is shown by two curves representing the cumulative and the differential distributions. Sample F is drawn in black, and sample UF is drawn in red.

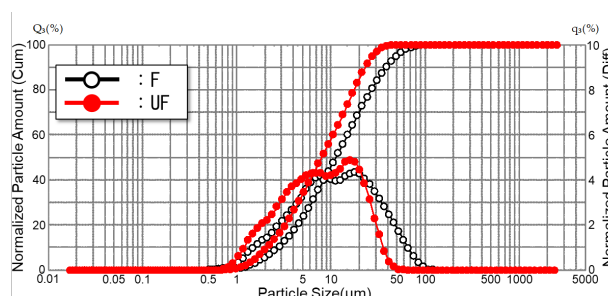


Fig. 1 Measurement Results of 2 Types of Chocolate by SALD-2300

There is a clear difference in particle size distribution between the two samples, and therefore, there is a difference in their representative particle size, such as median diameter. The lower limit of particle size distribution is about 0.7 μm for both samples, but the upper limit is about 90 μm for sample F and about 40 μm for sample UF.

In general, when food contains solid particles, it is said that people feel roughness in their mouth when the particles are larger than 20 μm . However, the feeling of roughness is not solely determined by particle size; other components also have a significant influence. In the case of chocolate, the oil and fat content and its melting characteristics are said to also affect the roughness.

Since this sample is milk chocolate with high fat content, we focused on the ratio of particles larger than 65 μm , which is slightly larger than 20 μm . For sample F, it was 1.77 %, and for sample UF, it was 0.00 %, which seems a reasonable correlation between the number of coarse particles and the roughness in the mouth.

■ Particle Size and Number Concentration Measurement by iSpect DIA-10

So far, the particle size distribution of two types of chocolate has been measured using the SALD-2300, and the relationship between the results and the texture has been examined. Next, we considered the measurement results obtained using the iSpect DIA-10.

The iSpect DIA-10 is different from general image analysis type particle size measurement equipment, and it has the advantage of being able to detect almost all particles within the measurement range. As a result, it is possible to accurately calculate the number concentration in the measurement solution.

From the SALD-2300 measurement results, it is clear that the sample contains about 20 to 30 percent particles that are below 5 μm , which is the minimum measurement size of iSpect DIA-10. Therefore, it is not possible to capture the number concentration of all particles in the sample.

On the other hand, since no particles exceed the measurement limit of 100 μm and almost all particles in the coarse particle region can be detected, it is possible to calculate the number concentration of particles above a certain size.

The results of measurements of samples F and UF using the iSpect DIA-10 are presented below. The dispersion procedure is the same as that for the SALD-2300 shown in Table 1, but the final concentration is equivalent to that of 0.1 g of chocolate dispersed in 1000 mL of isopropanol due to a further 50 times dilution during measurement.

The measurement conditions are shown in Table 3.

Table 3 Measuring Conditions

Equipment:	iSpect DIA-10
Frame Rate:	8 fps (Shooting Efficiency 97 %)
Binarization Threshold:	110
Total Amount of Liquid:	0.1 mL

Fig. 2 shows thumbnail images of several particles from sample F and sample UF arranged in order of maximum length. Sample F appears to contain coarser particles.

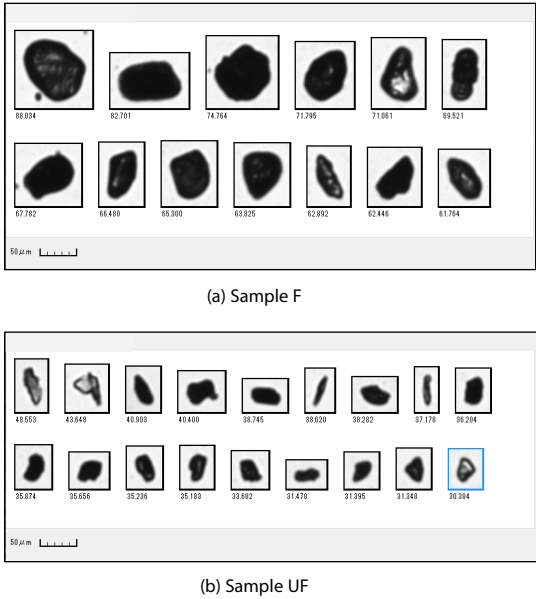


Fig. 2 Measurement Results of 2 Types of Chocolate by iSpect DIA-10 (Thumbnail Images)

Fig. 3 is a scatter plot showing the difference between the two samples in the coarse particle region. The maximum particle length is on the horizontal axis and the aspect ratio is on the vertical axis.

The upper part (a) is sample F, and the lower part (b) is sample UF. The total number of particles detected was 14746 for sample F and 6861 for sample UF.

The difference in the total number of particles is more than a factor of two, so it is somewhat difficult to make a direct comparison, but it is clear that sample F probably has a larger number of coarse particles. Also, the difference is especially noticeable in the number of particles larger than 50 μm .

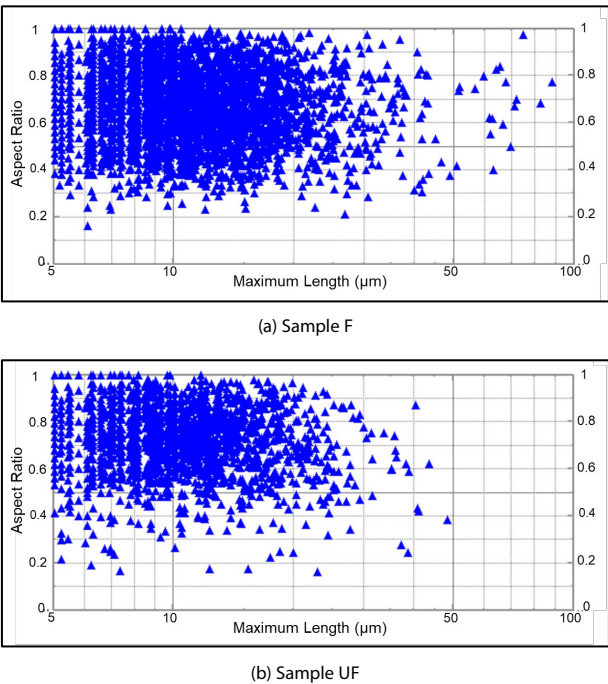


Fig. 3 Measurement Results of 2 Types of Chocolate by iSpect DIA-10 (Scatter Plot)

As previously mentioned, when the particle size exceeds 20 μm , people sense a roughness on the palate. Therefore, the number of particles with a maximum length of 20 μm or more per 1 g of sample was calculated from the number concentration. For comparison, the results of calculating the number of particles with a maximum length of 40 μm and 50 μm or more are summarized in Table 4.

While further investigations are needed to determine which values are appropriate as an indicator of the roughness or smoothness perceived when eating chocolate, the number of coarse particles contained could be one criterion.

Table 4 Concentration and Content of Particles Larger than a Specific Particle Size in Chocolate

	Sample	Maximum Length		
		20 $\mu\text{m} \leq$	40 $\mu\text{m} \leq$	50 $\mu\text{m} \leq$
Number Concentration (particles/mL)	F	308	36	19
	UF	128	5	0
Solid Particle Content (particles/g)	F	308×10^4	36×10^4	19×10^4
	UF	128×10^4	5×10^4	0

■ Conclusion

In this article, the particle size distribution of chocolate was measured using two instruments, the SALD-2300 and the iSpect DIA-10, and introduced the relationship between these results and the texture perceived when the chocolate was eaten. By looking at the results of the two measurement methods together, it is believed that a more in-depth analysis is possible.

In particular, the results of the number concentration of coarse particles measured using the iSpect DIA-10 are considered useful for evaluating texture based on the quantity of coarse particles. This can be expected to be effective in the food field and other fields where the abundance of small quantities of coarse particles is important.

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