Column

Technology That Uses Light to Measure Powder Size – Why Measure Particle Diameter? To What Point Can It Be Measured? –

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Introduction

The Japanese character for "powder" is made up of two parts that mean "separate" and "rice". Originally, grains like rice were crushed into small pieces. There are many types of powder around us, not limited to the "powder" that referred to crushed grains a long time ago. Some types of powder are suspended powders that are deemed to be harmful, such as fine particulate matter 2.5 ($PM_{2.5}$). More familiar types of powder would be in the form of medicine or food. We get benefits from many types of powders in our daily lives, from flour to home appliances and even space-related technologies. A wide variety of powders are used in various industries. There are powders that can be seen with the naked eye, as well as micrometer-size (0.001 mm) fine particles, and nanometer-size (0.001 μm) nano particles, all of which have different material characteristics and other characteristics that are based on size, and improve our daily lives. Powders that have a size of 100 nanometers or less are called nanoparticles. Nanoparticles can be organic or inorganic and are the technological elements that support various technologies. The HORIBA Group has been manufacturing and selling particle size distribution measuring equipment that uses light since the 1980s, which is one of the technologies that are used to evaluate these powders. There are many different types, such as the centrifugation method, laser diffraction/scattering method, and dynamic light scattering method (photon correlation method), and it is now possible to take measurements more quickly for more objectives and applications than before.

Particle diameter distribution analyzers are is used in many different fields, and this paper will discuss some of those applications. There are many different words related to "powder", such as "particle", "particulate", or "particulate matter", or "grains of" a certain substance. The way these are expressed vary based on the situation and industry. Particle diameter distribution is also called particle size distribution, but here we will call it particle diameter distribution.

Why measure Powder (Particle) Size?

When measuring particle diameter distribution using the laser diffraction/scattering method, the particle diameter distribution is measured based on the intensity of scattered light as a function of angle. With this method, there is a wet measurement method where the particles are dispersed into a dispersion medium like water, and measurements are taken after the particles disperse. There is also a dry measurement method where measurements are taken on

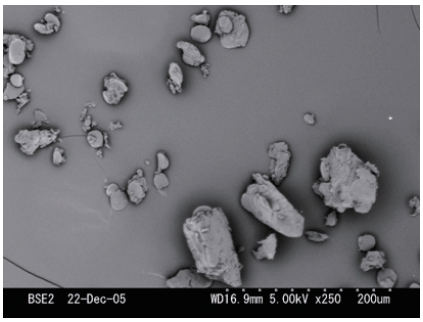
Figure 1 LA-9501/2

dry particles. LA-950V2 (see Figure 1) has a wide measurement range, from 10 nm to 3 mm. Why do you think we measure the size of powder particles in food and medicine?

Particle size of flour and why flour is in powder form

In English, words like “particle” or “powder” are often used in fields where particle diameter distribution is measured, but the word “flour” seems to often give an image of a “powder”. Flour is a major type of powder that has a wide variety of applications, and is required for daily life. Rice has a structure in which the hull can simply be separated from the inside, but with wheat, the hull is separated after it is pulverized. When wheat is made into powder, it is easier to process, and can be used for many different types of foods, such as bread, pasta, and cake, as well as Japanese foods like udon, ramen, and okonomiyaki. Depending on the size, color, and components, some flours have strong or weak particles, and are used for different applications, and understanding particle diameter is very important in assuring product quality.

For this paper, we measured flour sold in the market using the LA-950V2, using the wet method and dry method. With the wet method, the flour is put into a dispersion medium and measurements are taken. With the dry method, the powder is measured as-is. Flour also ends up swelling in water, so we used ethanol as the dispersion medium. The particle diameter distributions for the wet method and the dry method are shown in the graph below. An SEM image of the flour is also shown (see Figure 2).



SEM image

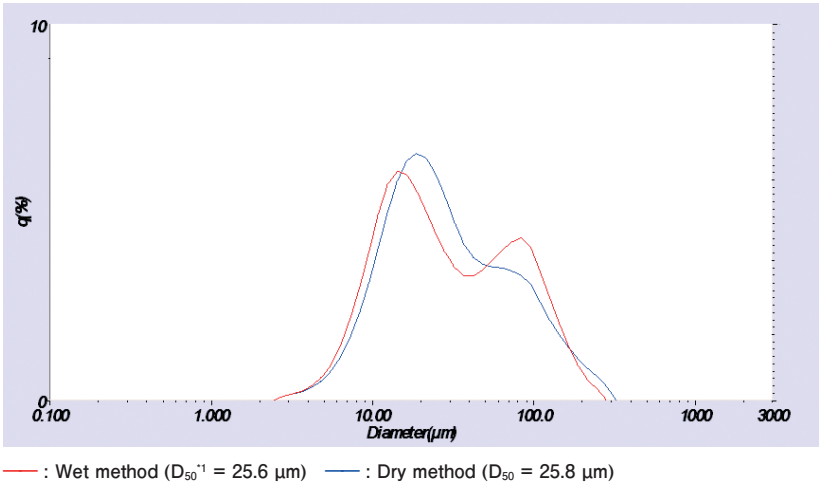


Figure 2 The particle diameter distributions for the wet method and the dry method and SEM image

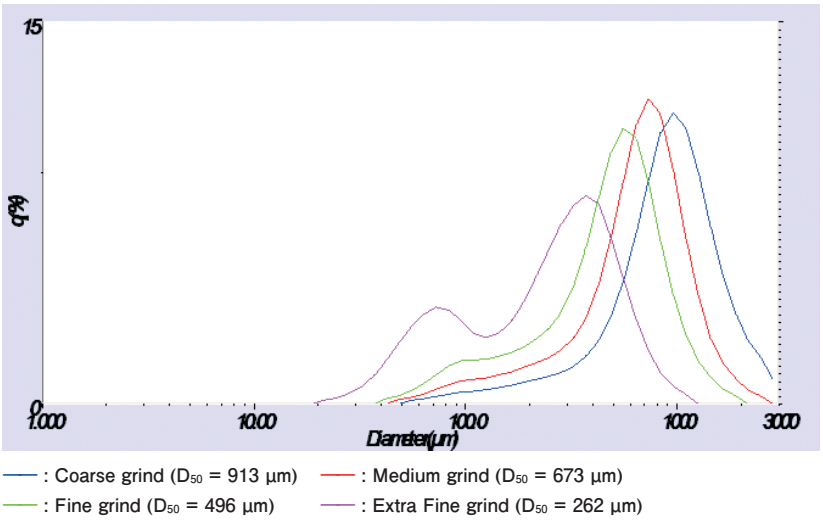
From both the SEM image as well, you can see that flour has mixed sizes. This time, we used the LA-950 to take measurements using the wet method and the dry method, and the results showed that both got the same wide particle diameter distribution. The wet method has stable dispersion, and produced more stable results.

*1: Particle diameter for 50% of the cumulative value. Also called the median diameter in the particle diameter distribution.

Particle Size and Aroma and Taste of Coffee

Coffee is said to have originated in Ethiopia, and was taken to the Arabian Peninsula in the 10th and 11th centuries, where records were found indicating that it was used as a medicine. The custom of drinking coffee began in the 14th and 15th centuries, and roasting methods were discovered around the same time. In modern times, coffee is a favorite around the world, and is a familiar drink for many people. There are various methods for extracting coffee, such as the filter method, the siphon method, and pressure extraction like espresso machines. With the espresso method, the coffee is extracted in a short time, so a coarse grind does not bring out the full taste and aroma. Therefore, as fine a grind as possible is preferable when the espresso method is used. However, when the filter method is used, if the grind is too fine, the extraction will be excessive, which will have an adverse effect on taste and aroma, so a medium grind is preferable for the filter method. A coarse grind would be suitable for a slow extraction at lower temperatures.

For this paper, we used the dry measurement method on ground coffee beans in the market to see what kind of particle diameter distribution they have, and how the particle diameter distribution is different based on the grinding method (see Figure 3).



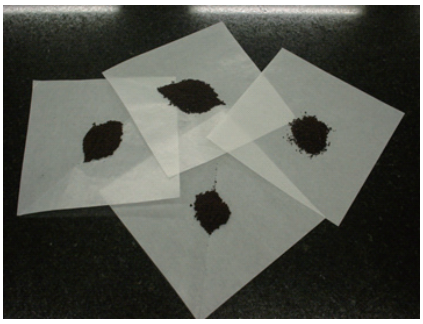


Figure 3 The coffee particle diameter distribution is different based on the grinding method

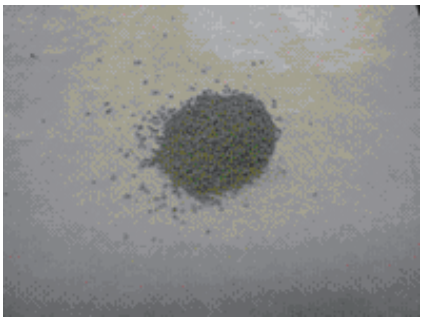
If we look at the measured distribution, you can quickly see the respective size characteristics. One objective of making a solid into a powder is to increase reactivity and solubility by increasing the surface area. As you can see, sorting out the particle size and getting rid of variances are important factors in controlling taste and aroma.

Particle Size and Effectiveness of Medicine

In the field of medicine, particle diameter is a very important point for ensuring the effectiveness of medicine as well as safety. Many medicines are toxic, and the amount ingested needs to be strictly controlled to avoid side effects. For excellent treatment effectiveness, a technology is needed that only delivers the necessary amount of medicine molecules to the necessary area. This is called a Drug Delivery System (DDS). Drug delivery systems use

granulation technology (size), coating technology, and encapsulation to control the effective area (stomach, intestines, etc.), time until the medicine dissolves, and time until the medicine takes effect.

Medicines dissolve in various dissolving agents, like water, so in some cases, it is difficult to use the wet measurement method for the laser diffraction/scattering method. Also, different effects are achieved by different sizes of granulated powder such as stomach medicine, and these need to be measured as-is. In cases like these, how do we measure particle diameter distribution? Dissolving agents can be investigated, but for this paper, we measured the particle diameter distribution of stomach medicine using the dry measurement method that doesn't use a dispersion medium. If we look at the results, we can see that there is a wide distribution, and that large and small particles were detected at the same time (see Figure 4).



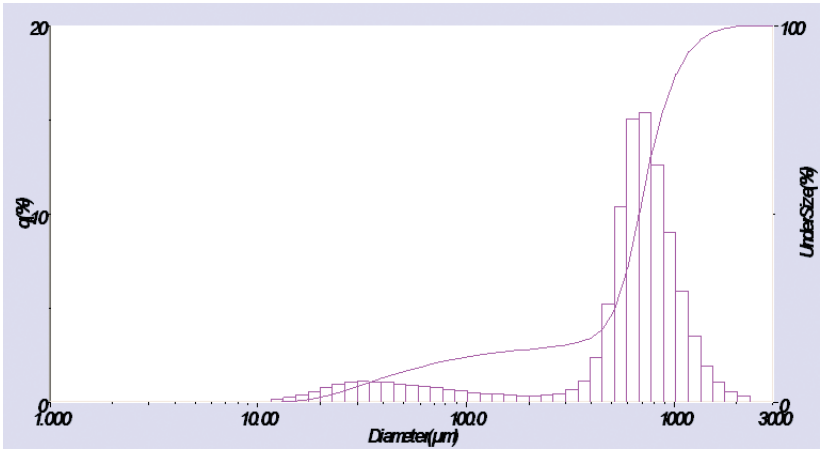


Figure 4 The particle diameter distribution of stomach medicine using the dry measurement method

In recent years, research on nanoparticle-sized DDS has progressed, with the goal of achieving pinpoint treatment with a higher accuracy. One of the subjects of this research is measuring the particle diameter of drug carriers like liposomes*2 and dendrimers*3.

*2: Artificial fine particles composed of phospholipids. The particle wall is a film made of 2 molecules, one that is hydrophilic and one that is hydrophobic.

*3: As a rule, this refers to macromolecules with a highly branched structure.

Particle Size and How It Feels to eat Chocolate (Sweetness and how it melts in your mouth)

Cacao, the main raw ingredient in chocolate, originated in the tropical regions in Central and South America and comes from the cacao tree, an evergreen tree in the Sterculiaceae family. The fruit of the cacao tree is large oval-shaped (depending on the type, may be elliptical shaped, partially oval shaped, or triangular shaped) cacao pods that directly hang down from the trunk, with a length from 15-30 cm and a diameter of 8-10 cm. Inside each cacao pod are 20-60 cacao beans. The substance produced by immersing these cacao beans in water and fermenting them becomes the raw material used for cocoa and chocolate. The typical raw ingredients that go into chocolate are cacao mass (a paste made from pulverizing the cacao beans), cocoa butter (the fat of the cacao bean), sugar, milk, spices, etc. The texture when eating chocolate

depends on how much the cacao mass was pulverized. The larger the particles, the faster the chocolate melts in your mouth. If the particles are too large, the texture seems rough. But the chocolate will seem smooth if the particles are small, but the chocolate won't seem to melt in your mouth. The smallest particle size that will seem rough to the human tongue is about 20 µm.

For this paper, we measured and compared the particle distribution measurement results for Japanese chocolate and American chocolate. If we look at the results, we will probably find that both will feel different when someone eats them. Chocolate dissolves in water, so we used Isopar H (a paraffin-based solvent) as the dispersion medium (see Figure 5).

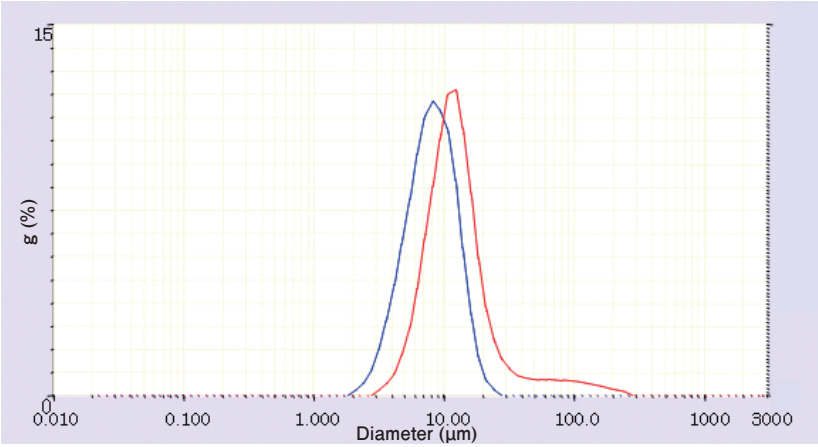


Figure 5 The particle diameter distribution of chocolate

Even if we just look at one food example, we can see that technology for measuring particle diameter distribution is required.

*4: Particle diameter (max. value) is 100% of the cumulative value.

To what point can powder (particle) size be measured?

If particles are small and mostly not visible to the naked eye, how do we go about measuring particle size? When using the dynamic light scattering method to measure particle diameter distribution, the particle size can be determined based on the diffusion coefficient. The SZ-100 (see Figure 6) can measure smaller particle diameters than the laser diffraction/scattering method, with a measurement range from 0.3 nm to 8 µm. If it's this small, it may not seem like "powder".

Figure 6 SZ-100

Vitamin size

Vitamin E is a fat-soluble vitamin with some of the strongest antioxidant effects among the vitamins, and is famous as a vitamin for preventing aging. Vitamin E is also called Tocopherol. Tocopherol acetate, an acetic acid ester of tocopherol, is one vitamin E derivative that is widely used for medicine, cosmetics, food, animal food, treating illnesses, nutrition supplements, and as an agent to prevent oxidation in food additives. How large is the size of the

vitamin?

For this paper, we dissolved d-α-tocopherol acetate[*5] in water, dispersed it, and took measurements. The results showed an average diameter of 45 nm. 45 nm is a small size, Also, it is probably a particle made up of several d-α-tocopherol acetate molecules (see Figure 7).

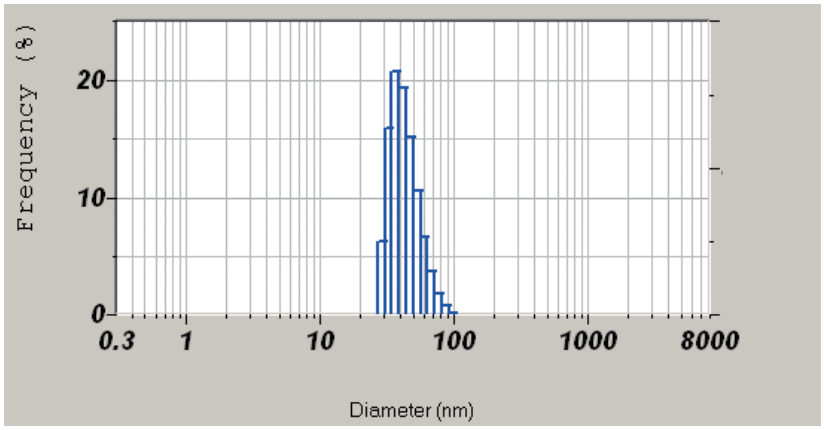
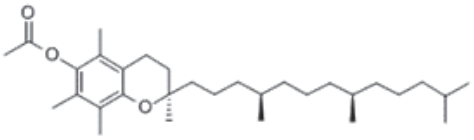


Figure 7 The particle diameter distribution of d-α-tocopherol acetate molecules



Structure of d-α-tocopherol acetate molecules
(from Wikipedia)

*5: d-α-tocopherol acetate 10% lysate (viscosity 1.6 mPs·s): Provided by Eisai Food & Chemical Co., Ltd.

Size of Colloidal Silver

A colloid is a particle with a size between 1-100 nm that is not observed using optical microscopes and doesn't pass through semi-permeable membranes, and has unique characteristics such as Brownian movement[*6] and the Tyndall phenomenon[*7]. Colloidal silver is a solution that has silver nanoparticles with a size of 100 nm or less uniformly dispersed in the liquid. Normally, colloidal silver is created using the liquid phase reduction method. It is possible to create colloidal silver with a desired particle diameter by changing the reduction agent type, conditions, etc. This process is stabilized by modifying macromolecules on the surface, creating three-dimensional barriers, and using

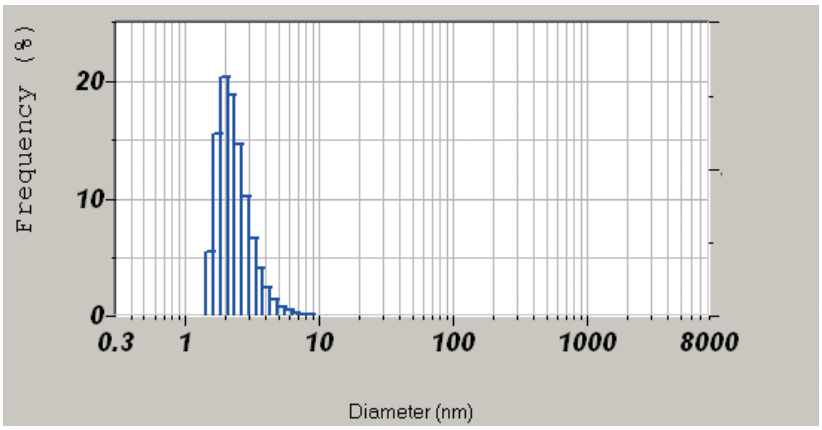


Figure 8 The particle diameter distribution of colloidal silver

electrostatic potential to ensure repulsive force between particles. Colloidal silver nanoparticles has optical, electrical, and heat characteristics not found in other substances and is used for microwiring material (conductive ink), paste, and filling material. This is used in a wide range of products, from solar cells to sensors and antibacterial agents.

These results show measurements from a colloidal silver solution dispersed in hexanes. We got an average diameter of about 2.5 nm. This is 1/1000 of 1/1000 of 2.5 mm. Of course, this particle cannot be seen by the naked eye (see Figure 8).

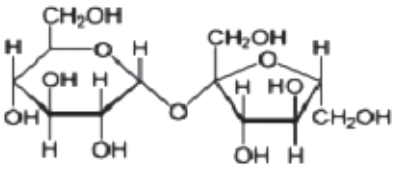
*6: Phenomenon when fine particles dispersed in a liquid, etc. move randomly
*7: Phenomenon when light is shone onto a liquid with fine particles dispersed in it to show the path of the light

Size of Sucrose (main component in sugar)

When water is added to salt or sugar crystals, they quickly dissolve into a transparent aqueous solution. Salt (NaCl) is an electrolyte, and can be ionized and broken down into atomic-scale Na ions and Cl ions when it comes in contact with water, and as such it is difficult to measure the particle size. Sugar keeps the shape of the molecules when dissolved into water, so the molecule size/order of the particles can be measured. Sucrose, the main component in sugar, is a type of sugar that is a blend of 2 sugars: glucose and fructose. Both glucose and fructose are made of sweet, colorless crystals, and dissolve in water, and these are widely used as sweeteners.

Actual measurements showed an average particle diameter of 1 nm or less. This is almost the size of one molecule. Can you imagine one molecule? It has a structure similar to the one below.

Here, to get scattering strength, we took measurements in a high concentration



Structure of sucrose
(From Wikipedia)

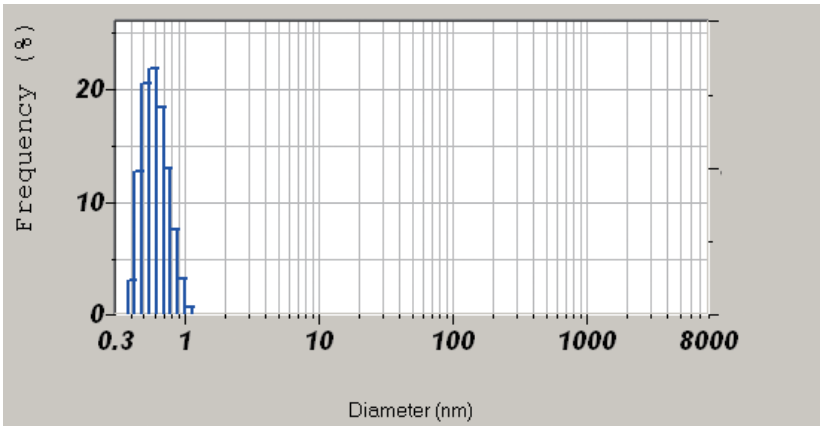


Figure 9 The particle diameter distribution of sucrose

(From Wikipedia)

of 40%, but it is important to pay attention to aggregates. Also, the viscosity was somewhat high, with an actual measurement of 3.5 MPa·s[*8]. Measurement results for a solution of 40% sucrose in water are shown below (see Figure 9).

Conclusion

As we have mentioned in this paper, particle diameter distribution is measured for many types of particles, from things like flour and coffee that are visible to the naked eye, to the molecules in vitamins and sugar that can't be seen with the unaided eye. Measuring particle diameter distribution is a major factor in the quality of many products, such as food, medicine, ceramics, ink, paint, cosmetics, battery materials, semiconductor materials, and products that rely on nanotechnology. Here is no more convenient method in which particle diameter can be measured, and a very important measurement technology.

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