Comparing granulation measurement methods for grain materials

Stanisław KAMIŃSKI, Dorota KAMIŃSKA – Kamika Instruments, Warsaw

Please cite as: CHEMIK 2012, **66,** 5, 406-411

Unknown properties of laser devices

Measurement method: **laser diffraction** does not measure the size of particles directly, but only the diffraction angle of the laser beam from the particle surface. Based on this angle, the size, i.e. some dimension of particles, is determined. What do we know about this dimension? We know nothing. We do not know whether this is the diameter, length or diagonal of the particle. Is it the average maximum or minimum dimension of the particle? Or maybe it is a dimension dependent on the optical properties of the device and not on the particle shape.

The "measurement range of tested particles" presented in offers of various devices employing laser diffraction (so-called laser devices), e.g. 0.02 μm – 2000 μm, should be distinguished from the regulated measurement range of the device.

Apart from the measurement range of tested particles, there is a relatively limited measuring range of the device, i.e. the distance between the minimum and maximum dimension possible to obtain during one setting of optical-electronic systems. This is the determination of the so-called measurement dynamics.

In order to determine it, one should learn the parameters of the A/C (analogue-to-digital) converter which measures the intensity of a luminous flux on the photoelements of the laser device.

Current is generated on each illuminated photoelement which may be converted into voltage measured by an A/C converter.

The A/C converter is a system for measuring voltage, with a specified number of measuring ranges / channels which correspond to a gradual increase of voltage. The lowest range should correspond to the illumination of a photoelement by one particle, while the highest range – to the illumination of a photoelement by a specified maximum number of particles. A larger number of particles are not differentiated by the A/C converter. If only one particle "is included" in the first measurement class of the A/C converter, then the highest class should "contain" as many particles as there are classes in the A/C converter. This is the resolution of the converter with a single measurement.

A single measurement of the maximum particle is very important because one 2-mm particle counterbalances 2000^3, i.e. 8,000,000,000 particles with a size of 1 μm. Failure to measure a single maximum particle undermines the sense of measuring the entire set of particles. If we assume a dimension of the minimum particle, then we should ponder over the dimension of the maximum particle with a specified optical setting and measurement capabilities of the A/C converter. For particles of different sizes, an equation may be created which describes and compares their volume:

$$\frac{\Pi}{6}D^3 \times 1 = \frac{\Pi}{6}d^3 \times n$$

after abridgements $$D = d \times \sqrt[3]{n}$$

where:

$\sqrt[3]{n}$ – is the so-called measurement dynamics

d – minimum dimension

D – maximum dimension

n – number of smaller particles which has to be the equivalent total resolution of the A/C converter

for	n =	10,000	D = 21 d
	n =	100 000	D = 46 d ≈ 50 d
	n =	1,000,000	D = 100 d

Taking into account the average total resolution of the A/C converter n = 100,000 and recalculating according to formula D ≈ 50 d for different measuring ranges of the device, we obtain:

for minimum dimensions	d = 0.02 μm	D = 1 μm
for popular dimensions	d = 1 μm	D = 50 μm
for maximum dimensions	d = 40 μm	D = 2,000 μm

For a repeatedly higher number of particles in a set than the converter's resolution, the adding and averaging of measurement results occurs. Then the existence of the largest single particles in a set fades, while the measuring range of the device becomes even more narrow. Why is this possible? Because every particle in the laser diffraction method is represented by an analogue signal and only the sum of analogue signals is converted by the A/C (analogue-to-digital) converter into a digital signal sent to the computer.

The sum of analogue signals is first the sum of light intensity on a given photoelement, converted into current, and then converted into voltage which may be measured using an A/C converter.

There are many conversions and the precision is small because the light is additionally reflected off the particle and if the particle is not circular, the measurement result does not always coincide with the actual size.

The measurement result depends also on the optical properties of the particle. The Mie theory, which people try to apply sometimes, is suitable only for ideally circular and transparent particles. If the particle has a mat surface and irregular shape, the measurement result is even less precise.

For this reason, it is difficult to compare measurement using sieve analysis with measurement performed using laser diffraction, while such comparison should be fairly obvious.

The result of a laser size analyser depends also on the number of particles which may be determined from the laser pattern intensity. For single particles, the diffraction pattern is "barely visible" (we do not know the sensitivity threshold from which it is measureable) according to [1]: "Patterns coming from specific groups overlap. The resulting pattern (...) will look like a blurred spot. From the size of expanding the diffraction maximum one may draw conclusions as to the spread of particle sizes, at least as long as the distances between them are much bigger than their sizes".

Very little is said about the blurred diffraction spot and it is measured rather freely using optical-electronic converters placed in laser devices. Test [2] published by NIST may be provided as an example. NIST is the American National Institute for Standards and Technology. The test included 21 devices, including 18 laser devices which measured the same material. As seen on the appended chart, a different setting of the devices' optical properties gives a broad distribution of the same particle spread. The distribution equals approx. 20% for 1 μm. It is still unknown how to precisely and correctly measure a **blurred diffraction spot**.

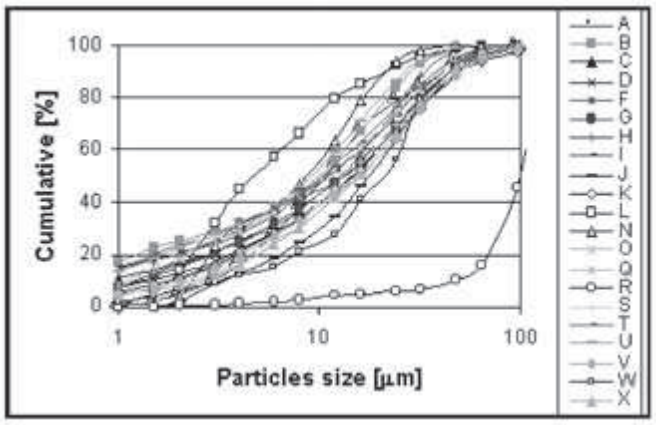

Fig. 1. Test results run by NIST – spread of measurements results of the same substance made mainly on laser size analyzers [2]

The critical argument in many tenders is referring to conformity with norm ISO no. 13320 [3]. This norm generally describes measuring using laser diffraction for particles with special shapes and properties. The shapes and properties are described separately. These are "optically homogenous", "isotropically spherical" particles. For particles other than circular, the norm does not provide the manner of measurement. If the actual particles are different, the measuring result may be obtained only through a comparison with an earlier intended similar set, determined by other measurement methods. For this, different devices and analysers are needed in order to determine the dimensions and shape of particles before performing measurement using a laser device. As an example, we present photographs from page 38 of the described norm. Figure 2 presents three different particle shapes and their diffraction patterns. Sphere – is unambiguously and precisely determined optically and may be measured well.

Cuboid – is well measurable if its geometric axes coincide with the optical system's axes. If we tilt the rectangle to the side, the diffraction pattern becomes more complicated and is not unambiguously measurable. Such cuboid may also be tilted to the front or back which gives a diffraction pattern which is hard to anticipate because the pattern depends also on the sharpness of the cuboid's edge. Irregular particle – the presented diffraction pattern allows for freely interpreting the dimensions of the particle. Generally, an interpretation is used which is most favourable to the user, but is it coherent with actual dimensions?

ISO 13320:2009(E)

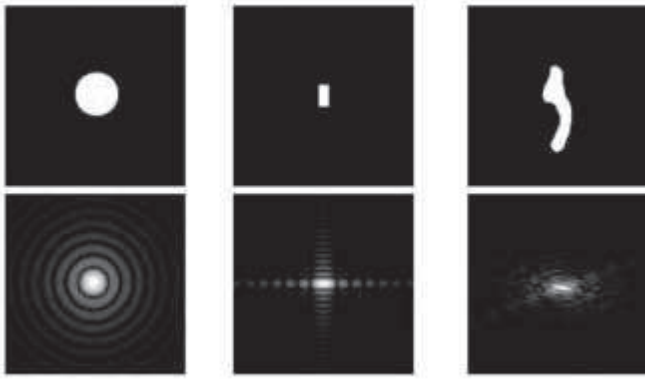

Fig. 2. Circular, rectangular and irregular particles and their scattering patterns [3]

Laser devices may specify only one dimension, but what are digital devices – those using the most modern ways of optical-electronic digital measuring – capable of?

Using a recorded scanning time, with a known speed of the particle's movement across a known measuring space, the second dimension of the particle may be determined.

In devices which use digital technology for measuring single particles it is possible to add different geometrically configured measuring paths and to perform a 3D measurement of particles.

Laser devices vs. digital measurement methods

In the age of computers and electronic (digital) measurement methods, the use of analogue measurement methods is an anachronism. Even if you use a laser for this purpose, the currently used laser diodes lack precise stabilization of operating in time and a geometrical uniformity of radiation intensity. This is sufficient for analogue measurements. Currently, with very fast electronic elements, each particle may be measured separately, while the multimillion set of particles may be counted, measured and divided into fractions within a few minutes [5]. Modern measurement methods do not share some of the important disadvantages of the laser devices. By scanning every particle separately, its shape may be precisely determined during measurement, thanks to which it is possible to automatically adopt a suitable algorithm for calculating the particle volume. In laser devices, the identification of particles in terms of shape and optical properties should be performed before the measurement. If you do not do this, the measurement precision will be easy to anticipate...

This involves comparing what we see under the microscope or what we can measure using any classic method, e.g. on sieves, with the result obtained using a laser device. The biggest success of laser devices is that many have believed that the measurement may be performed within 20s. In specific cases, it is possible that the substance will perfectly fit such measurement. It is impossible to separate all pudding stones and conglomerates as well as to dry measure the entire representative sample. The actual measurement requires much more time. In digital devices, the entire representative sample should be measured which lasts a few minutes, although the entire set of particles can be measured even within 10s. A large measurement problem of laser devices is multimodal (i.e. "multi-humped") separations. Obtaining such separation on a laser device is a difficult thing. Generally popular bimodal separations result from errors of lowering particle measurements and from adding background and noise to the separation. In this case, the typical first "hump" is generally located at approx. 1 μm and spreads below this dimension.

Quick scanning of voltage flow in digital methods automatically sets the zero level. Always one particle is measured; therefore, the entire measuring range of the A/C converter is at its disposal. It may be precisely measured without the participation of many other particles and may be digitally recorded in the computer's memory. Storing in its memory the dimensions of all particles, there are no problems with multimodal results.

Optical-electronic devices for measuring particles using the method of digital measurement of particle recording (digital devices)

In the measurement method described below, each single particle is measured and counted using an optical-electronic system. This occurs differently, most often through measuring radiation dispersion; sometimes, dispersion is affected by diffraction. The measurement of two particle dimensions is described in [6] and takes place using a 12 A/C converter with a sampling frequency of 12 MHz. The analogue electric impulse corresponding to the particle size is measured by an A/C converter and the impulse amplitude corresponds to the maximum particle dimensions, while the impulse width corresponds to the particle's thickness. The maximum measured particle dimension depends on the way of particle's movement in the measuring space and this may be controlled using a suitable dosing device.

The measurement results of single impulses in digital form are directly transferred to the computer, where they are stored on a disc. These measurements are not influenced by the particle's

optical properties because a specific gravity other than characteristic for air or water is sufficient for dispersing light through a perfectly transparent particle. What is important, measurement takes place in a parallel radiation beam, in specific measurement space dimensions selected depending on the size of measured objects. Measuring spaces are available with a cross-section from a few mm^2 up to 60,000 mm^2 (areas in different devices) [7]. Proper particle dimensions are obtained through its scanning analysis. For the purpose of dosing grain materials mainly air is used, but for materials in the form of suspensions water or aqueous solutions may be used with a changed device structure.

Measurement in air for all dry materials simplifies the preparation and performing of measurements. Such measurement may be applied to sticking or moist materials.

There is a strict link between the maximum grain dimension and the electric impulse amplitude as well as the minimum grain dimension specified by the impulse width. Measured and counted impulses enable an unambiguous, precise and repeated determination of the grain set in electric units, i.e. in converter channels, which may be stored in a computer's memory.

The grain set stored in the computer's memory in the form of a statistical distribution of quantity and size, after recalculating into a volume distribution, may be compared with actual measurements performed according to classic measurement methods. From each comparison, calibration characteristics may be obtained for the optical-electronic measuring device.

The digital device's calibration is attributed to a specific measuring method or grain shape. For the same measurement, results may be obtained for particle size distribution according to the following calibration: spherical, sieve and e.g. depositional.

The following question may arise: which of these calibrations is the best? All of them are good if they are consequently applied in the control of the industrial process. The problem is not with calibrations but with an unambiguous, but at the same time clear and precise manner of measuring grains which is ensured by digital devices, i.e. devices which measure single grains one by one.

With such performed measurements which correspond to actual dimensions, a sieve or areometric analysis may be simulated 100%.

Usually, the total measuring range of the digital device is divided into several sub-ranges because of the simple optimization of measurements. If there are no large particles, the measuring range may be narrowed. A proper measuring range may be selected for suitable particle dimensions. For each of these ranges, the converter works with a maximum resolution which sets new standards in measuring particle sizes.

Summing up

The method of digitally recording particles enables:

1. With simultaneous, multidirectional measurements one may obtain information about the shape of each particle, considerably exceeding the capabilities of a flat microscopic analysis.
2. For a device which measures only in one direction, using also the time of the particle's transition through the measuring space, a two-dimensional particle image is obtained without additional microscopic devices.
3. Because of the dosing manner, a morphologic image of the tested substance may be obtained, not only due to the shape, but taking into account also the specific gravity of the particle.

Literature

1. JR Meyer- Arendt „Wstęp do optyki", PWN 1979
2. Ferraris, C.F, Hackley V.A., Aviles A.I., Buchanan C.E., Analysis of the ASTM Round-Robin Test on Particle Size Distribution of Portland Cement: Phase I" NISTIR 6883, May 2002
3. Norma ISO 13320-1:2009 "Particle size analysis - Laser diffraction methods"
4. Jillavenkatesa A., Dapkunas S. J., Lum L.-S. H., "Particle Size Characterization"; NIST Special Publication 960-1, 2001
5. Kamiński S., Kamińska D., 2007. Porównanie optyczno-elektronicznych metod pomiaru granulacji. Aparatura Badawcza i dydaktyczna, XII, 2-3, Warszawa, s. 85-93.
6. Kamiński S. Dwuwymiarowa analiza uziarnienia popiołów, materiały konferencyjne „Popioły z energetyki", Zakopane 19-21 października 2011, 233-245
7. Kamiński S., Kamińska D Pomiar granulacji surowców w mineralurgii przy użyciu nowoczesnych elektronicznych urządzeń pomiarowych „Górnictwo i Geoinżynieria T.33/4 (2009), 135-139

Stanisław KAMIŃSKI – Ph.D. (Eng), the founder of KAMIKA Instruments (formerly ZEAP), has developed a unique patented measuring method, which allows measuring grain-size distribution in one, two or even three dimensions, the designer of many measuring systems dedicated to fast control of industrial processes, for many years, researcher at the Institute of Aviation , now as an expert is a member of Polish Committee for Standardization and European Committee for Standardization

KAMIKA Instruments: info@kamika.pl

Dorota KAMIŃSKA – M.Sc., a graduate of the University of Warsaw, for many years engaged in grain size measurements using KAMIKA analyzers.

KAMIKA Instruments: info@kamika.pl