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Representation of results of particle size analysis —

Part 6: Descriptive and quantitative representation of particle shape and morphology

Représentation de données obtenues par analyse granulométrique

Partie 6: Description et représentation quantitative de la forme et de la morphologie des particules

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 9276-6 was prepared by Technical Committee ISO/TC 24, *Particle characterization including sieving*, Subcommittee SC 4, *Sizing by methods other than sieving*.

ISO 9276 consists of the following parts, under the general title *Representation of results of particle size analysis*:

— *Part 1: Graphical representation*

— *Part 2: Calculation of average particle sizes/diameters and moments from particle size distributions*

— *Part 3: Adjustment of an experimental curve to a reference model*

— *Part 4: Characterization of a classification process*

— *Part 5: Methods of calculation relating to particle size analyses using logarithmic normal probability distribution*

— *Part 6: Descriptive and quantitative representation of particle shape and morphology*

Introduction

A variety of different methods for the descriptive and quantitative representation of particle shape and morphology are known. Even for the term particle size, there is no single definition. Different methods of size analysis are based on the measurement of different physical properties. In ISO 9276-1, the particle size is defined as the diameter of a sphere having the same physical property. This is known as the equivalent spherical diameter. So-called property functions help to correlate it with the property of primary interest, which may, for instance, be flowability, taste or dissolution time.

Broad application of sizing methods in particle characterization shows that particle size is often an important factor. But particle size alone is not sufficient to allow particle phenomena such as powder flow, mixing, abrasion or biological response to be understood. Particle shape and morphology play an important role in particle systems and therefore it is also necessary to characterize and describe these characteristics quantitatively.

Including additional shape parameters in property functions is supposed to give a better correlation with the particular property of the particle system. For instance, knowledge of the size of grinding particles and of the sharpness of their edges will make it possible not only to distinguish between fresh and used grinding particles but also to predict their abrasive effect quantitatively by means of a property function.

ISO 13322-1 and ISO 13322-2 give guidance on the measurement, description and validation methodologies used when determining particle sizes by static and dynamic image analysis, respectively. Broad industrial use of image analysis techniques requires standardized methods of measurement for the characterization of the size, geometrical shape and morphology of particles.

A particle's shape is the envelope formed by all the points on the surface of the particle. Particle morphology represents the extension of a simple shape description of this kind to more complex descriptions including characteristics such as porosity, roughness and texture.

Various glossaries of terms giving descriptions, in words, of particle shape and morphology already exist (see Clause 5). These descriptions may be useful for the classification or identification of particles but, at the moment, there is insufficient consensus on the definition of particle shape and morphology in the quantitative terms necessary for them to be implemented in software routines. A future revision of this part of ISO 9276 may cover this.

Representation of results of particle size analysis —

Part 6:
Descriptive and quantitative representation of particle shape and morphology

IMPORTANT — The electronic file of this document contains colours which are considered to be useful for the correct understanding of the document. Users should therefore consider printing this document using a colour printer.

1 Scope

This part of ISO 9276 specifies rules and nomenclature for the description and quantitative representation of particle shape and morphology. To achieve a more comprehensive description of a particle or particle system, particle size information can be used together with other information but, in most cases, the particle size information cannot be replaced.

The averaging of shape over all particles in a sample has been shown to be an ineffective approach. Distributions of other particle characteristics are required in addition to particle size distributions (see ISO 9276-1).

The relevance, to technological applications, of any method of representing particle shape is the deciding factor in its use. Therefore this part of ISO 9276 is restricted to methods which can be correlated with physical properties in industrial applications.

The aim of particle analysis is to determine the most appropriate characterization method for a particular application. This implies a profound understanding of the relationship between particle characteristics and macroscopic product and process properties (or at least a database of broad empirical data).

Problems of shape and morphology would normally be three-dimensional problems, but most definitions in this part of ISO 9276 are in fact given for two dimensions because of the widespread use of image analysis methods.

With the help of the evaluation criteria given in Clause 4, a minimum set of shape descriptors is derived in Clause 8 from the various descriptors and methods in Clause 5, enabling a direct comparison of different shape analysis equipment or methods to be made within the limits discussed in Clause 6.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 9276-1:1998, *Representation of results of particle size analysis — Part 1: Graphical representation* (and its Technical Corrigendum ISO 9276-1:1998/Cor.1:2004)

ISO 13322-1:2004, *Particle size analysis — Image analysis methods — Part 1: Static image analysis methods*

3 Symbols and abbreviated terms

For the purposes of this document, the symbols given in ISO 13322-1 and ISO 9276-1 and the following apply.

In ISO 9276-1, the symbol x is used to denote the particle size or the diameter of a sphere. However, it is recognized that the symbol d is also widely used to designate these values. Therefore, in this part of ISO 9276, the symbol x may be replaced by d wherever it appears.

Symbols for the particle size other than x or d shall not be used.

A projection area

A_{box} Feret box area

A_{c} area of the convex hull (envelope) bounding the particle

b intercept on graph for fractal dimension

C circularity

CI global surface concavity index

D_{F} fractal dimension

d_{cmin} diameter of the minimum circumscribed circle

d_{imax} diameter of the maximum inscribed circle

d_{L} spacing of a series of parallel lines [for use in the Cauchy-Crofton formula (see Clause A.1)]

E thickness

I_{α} number of intercepts [for use in the Cauchy-Crofton formula (see Clause A.1)]

L_{G} geodesic length

N number

P length of perimeter

P_{C} length of the perimeter of the convex hull (envelope) bounding the particle

Rn roundness

S surface area

V volume

x_{A} area-equivalent diameter of particle

x_{E} thickness of a very long particle

x_{Fmax} maximum Feret diameter

x_{Fmin} minimum Feret diameter

x_{LF} Feret diameter perpendicular to the minimum Feret diameter, normally known as “length”

x_{LG} geodesic length of a very long particle

x_{Lmax} length of major axis of Lengendre ellipse of inertia

x_{Lmin} length of minor axis of Lengendre ellipse

x_P perimeter-equivalent diameter of particle

x_S surface-equivalent diameter of particle

x_V volume-equivalent diameter of particle

α angle or direction

Ω_1 robustness

Ω_2 largest concavity index

Ω_3 concavity/robustness ratio

ω number of erosions

Ψ Wadell's sphericity

Ψ_{FP} average concavity

4 Criteria for the evaluation of shape description methods

A common problem in shape description is how to judge the quality of a shape description method. Not all methods are suitable for every kind of shape and application. Until now, consistent evaluation criteria have not existed for shape description methods.

Criteria for the evaluation of shape description methods:

— *accessibility*, which describes how easy it is to compute a shape descriptor in terms of memory requirements and computation time;

— *scope*, which refers to the classes of shape that can be described by the method;

— *uniqueness*, which describes whether a one-to-one mapping relationship exists between shapes and shape descriptors;

— *stability* and *sensitivity*, which describe how sensitive a shape description is to “small” changes in shape.

Each method shall use descriptors with a specific degree of complexity. In general, descriptors can be described as sets of numbers that are produced to describe a given shape. The shape may not be entirely reconstructable from these descriptors, but the descriptors for different shapes shall be sufficiently different to make it possible to discriminate between the shapes.

Criteria for shape descriptors:

— *invariance with respect to rotation and reflection* — for a given shape, the values of the descriptors shall be the same irrespective of the orientation of the particle;

— *invariance with respect to scale* — for a given shape, the values of the descriptors shall be the same irrespective of the size of the particle;

— *independence* — if the elements of the descriptors are independent, some can be discarded without the need to recalculate the others;

— *economy* — it is desirable that the descriptors be economical in the number of terms used to describe a shape.

The above three invariance conditions (concerning rotation, reflection and scale) guarantee that the result of a shape analysis is not affected by the parameters of the analysis and is independent of the particle size. It should, however, be stressed that the particle size at which certain shape information is obtained may be of practical relevance, as in the case of surface roughness, and size shall therefore be included in the shape analysis.

The robustness of shape descriptors with respect to the density, translation and rotation of the sampling grid can indicate whether it is acceptable to compare measurement results from different algorithms or different image analysers [1].

5 Classification of methods and descriptors

5.1 General classification

Methods of shape description, as well as the various shape descriptors, can be classified according to different criteria. An obvious way of classifying shape descriptors is to determine whether they are qualitative or quantitative in nature:

a) Qualitative description, i.e. in words: expressions such as “needlelike particles” and “oblate shape”. Examples of this type of shape characterization are given in the US Pharmacopoeial Convention [2], in ASTM F 1877 [3] and in the glossary made available by the NIST Center for Analytical Chemistry [4].

b) Quantitative description: in the following text, shape descriptors will be understood as numbers that can be calculated from particle images or physical particle properties via mathematical or numerical operations.

5.2 Levels of shape

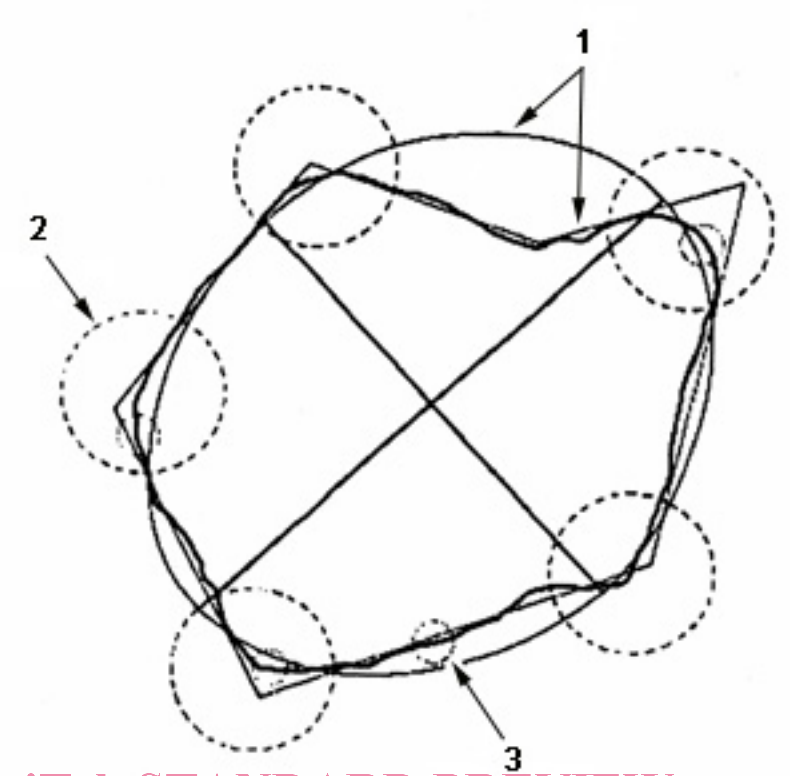
For a better understanding of shape description, it is important to establish definitions regarding the basic characteristics of an arbitrary object. The shape of an arbitrary object can be defined in many ways. One such definition describes shape as a binary image representing the extent of the particle. This can be understood as the silhouette of the particle. Barrett [5] recognizes three potentially independent particle shape properties (see Figure 1):

— *form*, which reflects the geometrical proportions of a particle;

— *roundness*, which expresses the radius of curvature at the particle corners;

— *surface texture*, which is taken as defining local roughness features at corners and at edges between corners only.

These particle shape properties may not suffice for a complete description of the shape of a particular particle and may be defined differently by different authors. But they give us a good idea of how shape parameters can be measured at different levels of size. Three corresponding levels of shape can thus be distinguished: macroshape, mesoshape and microshape.



Key

1 form

2 roundness

3 surface texture

Figure 1 — Illustration of form, roundness and surface texture [6]

Macrodescription is a description of the overall form of a particle defined in terms of the geometrical proportions of the particle. In general, simple geometrical descriptors calculated from size measurements made on the particle silhouette are used. Low-order Fourier descriptors can also be regarded as macrodescriptors.

Mesodescription provides information about details of the particle shape and/or surface structure that are in a size range not much smaller than the particle proportions, like Barrett's [5] roundness and concavity.

The following mesodescriptors can be defined:

a) morphological mathematical descriptors, computing robustness and largest concavity index;

b) a concavity tree, providing general insight into the organization of concavities and their complexity;

c) angularity descriptors, determining those parts of the boundary that are active in the abrasion process:

   1) an angularity factor, selecting the apices of corners which are coincident with the convex hull because it is these points that will make contact with the surface of another particle,

   2) a quadratic spike parameter, taking into account those spikes that are outside a circle, of area equal to that of the particle, centred over the particle centroid,

   3) slip chording, generating information on the number of cutting edges and their sharpness in the facet signature waveform;

d) fractal dimension, providing data on the overall structural complexity by consideration of a larger measurement step;

e) Fourier descriptors, of higher order than macrodescriptors, specifying the smaller-scale components of morphology;

f) bending energy, measuring the overall complexity of contour lines.

Microdescription determines the roughness of shape boundaries using two of the descriptors mentioned above:

— fractal dimension, measured using a measurement step smaller than that used for structural description;

— higher-order Fourier descriptors/coefficients for surface-textural analysis.

5.3 Principles for deriving shape descriptors

The level of inspection used in a method is a very practical criterion for the classification of the method, since many methods provide shape information at different size levels. Another convenient way of classifying methods is to differentiate between those which derive shape descriptors from particle images and those which derive shape descriptors from physical properties:

a) Calculation of geometrical descriptors/shape factors:

Geometrical shape factors are ratios between two different geometrical properties, such properties usually being some measure of the proportions of the image of the whole particle or some measure of the proportions of an ideal geometrical body that envelops, or forms an envelope around, the particle. These results are macroshape descriptors similar to an aspect ratio.

b) Calculation of dynamic shape factors from physically equivalent diameters:

These shape factors are similar to geometrical shape factors except that at least one physical property is considered in the comparison. Usually, the results are expressed as a ratio of equivalent diameters, e.g. Stokes sedimentation velocity to volume-equivalent diameter $x_{\mathrm{Stokes}}/x_{\mathrm{V}}$.

c) Morphological analysis:

Morphological analysis descriptors give mean values of particle shape that are not much smaller than the proportions of the whole particle. A typical example is concavity analysis.

d) Analysis of the contour line (shape boundary):

Multiple operations on the contour line of a particle can produce a set of shape descriptors. This set of shape descriptors contains information on the particle shape at different size levels. Methods belonging to this group include fractal dimension analysis and the use of Fourier analysis.

e) Analysis of grey-level images:

Multiple operations on the grey-level pixel image of a particle can produce a set of shape descriptors which can be correlated with the topology or surface texture of the particle.

f) Analysis of physical spectra:

Multiple operations on, or the mathematical treatment of, the physical spectra of a single particle can extract the shape information as a set of descriptors. Such a procedure has been described for shape analysis by azimuthal light scattering and diffraction spectroscopy.

