TECHNICAL SPECIFICATION

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Dispersibility of solid particles into a liquid

Dispersibilité de particules solides dans un liquide

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 24, *Particle characterization including sieving*, Subcommittee SC 4, *Particle characterization*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Dispersing particles is crucial for many end-use product properties such as colour, sensorial quality, polishing, film homogeneity, conductivity, therapeutic efficacy, opacity of paints and inks, as well as UV protection in cosmetics. Dispersions are evaluated over the life-cycle of a product, beginning in the design stage, through production and for the end product. Additionally, some type of dispersion process is a prerequisite for many routine particle characterization methods. Finally, the state of dispersion is an important issue for risk evaluation of fine particles and for classification of nano-enabled products[37].

However, the ease with which a particulate material is dispersed in a liquid phase to meet established or desired criteria, commonly referred to as dispersibility, is not well defined; and its common usage varies widely across different fields and applications. Most existing definitions are application or product specific. Therefore, the need exists for a document that summarizes the common practice, and which also clearly defines dispersibility in a generic manner that is not application dependent. This document addresses that stated need.

The definition for dispersibility is established based on a survey of existing standards of national and international organisations as well as standardized procedures of industry in different application fields. With respect to the final product in different applications, the state of dispersion can be characterized according to the uniformity of the dispersed phase over the entire volume and according to the mean particle size or size distribution with respect to application specific criteria.

This document does not address intermediate- or long-term alterations including aging of a suspension following processing, nor does it address secondary destabilization phenomena. Stability and shelf life are considered in specific standards such as ISO/TR 13097 and ISO/TR 18811.

Dispersibility of solid particles into a liquid

1 Scope

This document establishes a generally applicable (i.e. not application specific) definition for dispersibility. It identifies significant characteristics for evaluating dispersibility and lists examples of methods used to characterize dispersibility in various applications.

This document applies to processes that disperse powders into a liquid continuous phase while reducing the size of agglomerates or flocs down to the intended level, that homogenize an existing dispersed solid phase of a suspension or the mixture of two suspensions, or that exchange the original continuous phase in a suspension for another. Specific methods to disperse particles and to characterize the state of dispersion and/or homogeneity are only referenced, if necessary, for context.

This document is applicable to nano- and micro-sized particles across a range of product applications.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
agglomerate

loosely coherent assembly of *particles* (3.12) and/or *aggregates* (3.2) held together by weak physical interactions, with a total surface area virtually equal to the sum of the surface areas of the *constituent particles* (3.13)

Note 1 to entry: The transitive verb “agglomerate” means “to gather into a cluster”, and the process by which the cluster or assembly is formed is generally called “agglomeration”.

Note 2 to entry: Agglomeration can be a reversible process.

3.2
aggregate

assemblage of *particles* (3.12) into rigidly joined structures

Note 1 to entry: Formation of aggregates is usually an irreversible process.

Note 2 to entry: The forces holding an aggregate together are strong, for example covalent bonds or those resulting from sintering or complex physical entanglement.

Note 3 to entry: In common use, the terms aggregate and agglomerate (also aggregation and agglomeration) are sometimes applied interchangeably, but this practice is deprecated since the terms are not synonymous.

Note 4 to entry: The transitive verb “aggregate” means “to gather into a mass or whole”, and the process by which the structure is formed is generally called “aggregation”.

[SOURCE: ISO/TR 13097:2013, 2.2, modified — The term has been changed from "aggregation" to "aggregate"; notes 1 and 3 to entry has been updated; note 4 to entry has been added.]

3.3
comminution
operation of reducing *particle* (3.12) size by crushing, grinding or pulverisation

[SOURCE: ISO 12743:2021, 3.14]

3.4
dispersing agent
dispersant
substance capable of promoting the formation of a *dispersion* (3.7)

[SOURCE: ISO 862:1984, 82, modified — The admitted term "dispersant" has been added.]

3.5
disperse
distribute *particles* (3.12) homogeneously throughout a continuous phase, often by means of reducing the size of *agglomerates* (3.1)

3.6
dispersibility
qualitative or quantitative characteristic or property of a particulate source material assessing the ease with which said material can be *dispersed* (3.5) within a continuous phase

Note 1 to entry: Spatially uniform distribution (*homogeneity* (3.11)) of the dispersed phase is considered an integral part of the desired end point.

Note 2 to entry: *Particle* (3.12) size or particle size distribution is often used as an end point relative to defined criteria specific to the application.

Note 3 to entry: Dispersibility refers to a specific *dispersion* (3.7) process and specific process time.

Note 4 to entry: *Dispersion stability* (3.8), though a related phenomenon, should not be confused with dispersibility.

3.7
dispersion
multi-phase system in which discontinuities of any state (solid, liquid or gas) are homogeneously distributed in a continuous phase of a different composition or state

Note 1 to entry: If solid *particles* (3.12) are *dispersed* (3.5) in a liquid, the dispersion is referred to as a suspension. If the dispersion consists of two or more immiscible liquid phases, it is termed an emulsion.

Note 2 to entry: This term can also refer to the act or process of producing a dispersion, but in this context the term “dispersion process” shall be used.

[SOURCE: ISO/TR 13097: 2013, 2.5, modified — The words "in general, microscopic" have been removed from the begining of the definition; "discontinous phase" has been remove from the within the parentheses; "dispersed" has been replaced by "homogeneously distributed"; note 1 to entry has been updated; note 2 to entry has been added.]

3.8
dispersion stability
ability to resist change or variation in the initial properties (state) of a *dispersion* (3.7) over time, in other words, the quality of a dispersion in being free from alterations over a given time scale

Note 1 to entry: In this context, for instance agglomeration or segregation represents a loss of dispersion stability.

[SOURCE: ISO/TR 13097:2013, 2.6, modified — In note 1 to entry, "creaming" has been replaced by "segregation".]

3.9

energy density

amount of energy per unit volume of sample applied to a material

3.10

floc

ensemble of *particles* (3.12) that form a loosely coherent structure with high void fraction

Note 1 to entry: Flocs are held together by weak particle-particle attraction or by the osmotic pressure of a continuous phase containing polymers not adsorbing to *dispersed* (3.5) particles (loss of configurational entropy of polymers, depletion flocculation).

Note 2 to entry: The term floc may be used to denote an *agglomerate* (3.1) produced by addition of a flocculating agent (e.g., a polyelectrolyte), but flocs can also form spontaneously.

3.11

homogeneity

degree to which a property or a constituent is uniformly distributed throughout a quantity of material

[SOURCE: IUPAC Gold Book[38]]

3.12

particle

minute piece of matter with defined physical boundaries

Note 1 to entry: A physical boundary can also be described as an interface.

Note 2 to entry: A particle can move as a unit.

[SOURCE: ISO 14644-5:2004, 3.1.7, modified — The original note to entry has been replaced by 2 new notes to entry.]

3.13

constituent particle

identifiable, integral component of a larger *particle* (3.12)

[SOURCE: ISO/TS 80004-2:2015, 3.3, modified — Note 1 to entry has been removed.]

3.14

primary particle

original source *particle* (3.12) of *agglomerates* (3.1) or *aggregates* (3.2) or mixtures of the two

Note 1 to entry: Although a primary particle is often a *constituent particle* (3.13), a constituent particle is not necessarily a primary particle.

[SOURCE: ISO 26824:2013, 1.4, modified — The 2 original notes to entry have been replaced by a new note to entry.]

3.15

sedimentation

settling (separation) of the *dispersed* (3.5) phase due to the higher density of the dispersed *particles* (3.12) compared to the continuous phase

Note 1 to entry: The accumulation of the dispersed phase at the bottom of the container is evidence that sedimentation has taken place.

Note 2 to entry: Particles with a lower density compared to the continuous phase float rather than sink.

Note 3 to entry: Sedimentation leads to a loss of *homogeneity* (3.11).

[SOURCE: ISO/TR 13097:2013, 2.13, modified — The second sentence in the definition has been moved to note 1 to entry; the original note 1 to entry has been removed; notes 2 and 3 to entry have been added.]

3.16
surfactant
substance that lowers the interfacial energy of a material in contact with a liquid

Note 1 to entry: This term is a contraction of surface-active agent.

[SOURCE: ISO 18115-1:2013, 4.464, modified — The words "or the surface energy of that liquid" have been removed at the end of the definition; the 2 original notes to entry have been replaced by a new note to entry.]

4 Basic aspects of dispersion process

Dispersing particulate matter into a liquid continuous phase in the context of processing is a common method to produce suspension-based products in many industrial fields and to prepare samples for analysis (e.g. particle size analysis). In this document, the term “dispersion process” is used in the context of processing (dispersing a material)1). A dispersion process may start from dry source material or a pre-existing suspension. Dispersions can be created by mixing particles into a liquid to achieve a uniform distribution throughout the entire volume, and additionally tailoring the size or size distribution by mechanical or chemical means. Comminution, the size reduction of aggregates or primary particles by milling, for example, is not considered here. Furthermore, the dispersion process results in a multiphase system and not a solution.

The performance of a dispersion process, and therefore the quality/state of the final suspension, depends on the source materials, the liquid continuous phase, the type and conditions of processing, and is application-dependent and often quantified by reference specifications or criteria.

There are numerous textbooks, scientific papers, national and international standards, etc., dealing with different aspects of dispersing particles and characterization of achieved results (see ISO 8780-1, ISO 14887, ISO/TS 276872) and References [39] to [43]). Relevant ISO documents are included in the Bibliography and Annex A.

Various terms are introduced to compare or to quantify the success of the dispersion process, such as “dispersibility”, “level of dispersion”, “ease of dispersion”, “dispersibility index”, “degree of dispersity”, “re-dispersibility”, focussing often on a specific, mostly application driven aspect. These broad views and discussions reflect the practical need to characterize the suspension with regard to different sample preparation or manufacturing processes, as well as the supplied raw materials of different suppliers or batches from the same supplier. On the other hand, comparison or evaluation of the outcome is difficult due to the use of different terms and definitions. To arrive at a generic definition for material “dispersibility”, it is appropriate to define the starting point of a (liquid-based) dispersion process.

To disperse powder, the process starts with powder consisting of particles, aggregates and/or agglomerates and has the following objectives (see ISO 14887):

- wet the powder (source material) with the liquid (continuous phase);
- obtain a uniform distribution of mass throughout the liquid volume (primarily for mixing, not size reduction);
- decrease (reduce) the size of agglomerates of the source material to the application specific criteria for size or size distribution, or down to constituent particles or primary particles if desired.

In practice, these procedures are often combined intentionally with additional appropriate measures to prevent the occurrence of spatial inhomogeneities in volume concentration (e.g. due to separation or sedimentation) and any re-association of dispersed particles impacting the specified size or size distribution, respectively. These latter phenomena concern the stability of the dispersion state (ISO/

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1) In literature the term “dispersion” and its derivatives (disperse, dispersed, dispersing) can be used both as an active verb (as in “to disperse” something) and in reference to the material itself (as in “a colloidal dispersion”).

2) Withdrawn.

TR 13097 and ISO/TR 18811) with respect to time, which is beyond the scope of this document. ISO/TR 13097 provides guidelines to characterize dispersion stability.

To disperse pre-existing suspensions, the process starts with a liquid suspension of the source material that has degraded over time due to destabilization phenomena or requires, for its ultimate purpose, an exchange or alteration of the continuous phase or further processing to achieve product-specific criteria. In this case the objective may be one or more of the following:

— re-disperse agglomerated particles and sediments of a destabilized (e.g. aged) suspension;

— disperse the particles into a different liquid matrix or formulation (e.g. exchange of solvents);

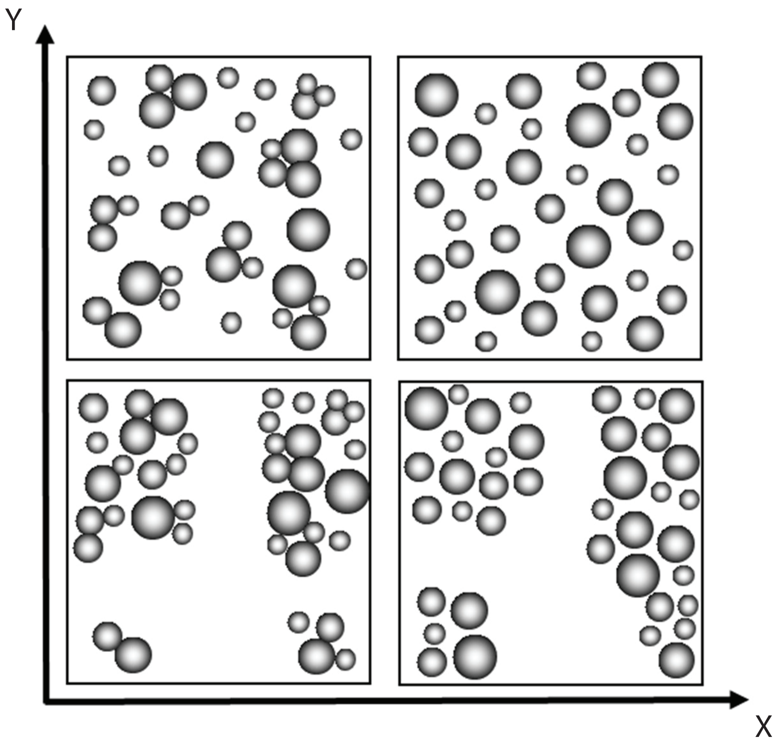
— disperse two or more different types of source material to form a single suspension;

— reduce the size of agglomerated particles to the required size or size distribution for a specific application or end-use.

According to the above description, dispersion processes may be theoretically divided into distributive and disruptive processes, as depicted in Figure 1. These processes often occur in parallel.

Distributive processing involves the mixing of particles throughout the continuous phase volume, often called homogenisation, to obtain uniformly distributed particles throughout the entire suspension volume. This process may not involve size reduction, if the energy input is insufficient to break apart existing agglomerates. In this case, the cohesive forces between particles are stronger than the applied disruptive forces.

Disruptive processing involves the reduction in size of the source material (such as agglomerates, flocs) and is often called dispersive processing, deagglomeration or simply dispersion. Success is attained when the disruptive forces necessary to separate an agglomerate's constituent particles overcome the attractive forces between them. It should be noted that achievement of product specified fineness (endpoint of disruptive processing) does not necessarily correspond with attainment of constituent particles, nor does it necessarily mean that a uniform spatial distribution has been achieved.



Key

X level of disruptive process

Y level of distributive process

Figure 1 — Schematic illustration of distributive and disruptive processes[40]

Three independent factors influence the success of the distributive or disruptive process:

a) the inherent chemical and physical properties of the source materials, for example, dry or wet, particle size and size distribution, shape, brittleness or state of agglomeration;

b) the interaction between the particle surface and the continuous phase; this factor depends, for instance, on wettability, electrostatic interactions, depletion forces, van der Waals forces, polarity interaction, and hydrogen bonding;

c) the dispersion process itself; time, energy, intensity and mechanism (mechanical principle) have an influence on the success of the distributive or disruptive process.

Therefore, to describe dispersibility all three factors shall be considered (multidimensional approach). Each of these independent factors can be characterized by a number of different attributes and quantified by suitable metrics.

5 Definition of dispersibility

5.1 Dispersibility definitions in existing standards

The concept of dispersibility has been used across a wide range of applications and industries for a substantial period of time; however, its interpretation and usage vary widely even among existing international and national standards. Therefore, a survey of existing standards and published references was conducted in order to identify commonalities and differences.

Annex A consists of an annotated listing of relevant standards. According to those standards, dispersibility can refer to the ease with which one can incorporate a powder into a suspension or