
Nanotechnologies — Clay nanomaterials —

Part 1: Specification of characteristics and measurement methods for layered clay nanomaterials

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Contents

Page

Foreword	iv
Introduction	v
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Abbreviated terms	3
5 Characteristics and measurement methods	4
5.1 General	4
5.2 Fundamental characteristics	4
5.3 Optional characteristics	5
5.4 Descriptions on characteristics and measurement methods	6
5.4.1 Chemical composition content	6
5.4.2 Mineral composition content	6
5.4.3 Interlayer distance	7
5.4.4 Thickness	8
5.4.5 Aspect ratio	9
5.4.6 Bulk density	9
5.4.7 Cation exchange capacity	9
5.4.8 Loss on ignition	9
5.4.9 Water absorption capacity	10
5.4.10 Moisture content	10
5.4.11 Brightness	10
5.4.12 Colour	10
5.4.13 Methylene blue adsorption capacity	11
5.4.14 Cohesion coefficient	11
5.4.15 Tap density	11
5.4.16 Specific surface area	11
5.4.17 Film formability	12
5.4.18 Electrical resistivity	12
5.4.19 Modifier type	13
6 Reporting	14
6.1 General	14
6.2 Information	14
6.3 Measurement results	14
6.4 Example of table format	14
Annex A (informative) Basic information on layered clay nanomaterials	16
Annex B (informative) Organo-modified layered clay nanomaterials (Organoclay)	18
Bibliography	20

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.

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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A list of all parts in the ISO/TS 21236 series can be found on the ISO website.

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.

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Introduction

Layered clay nanomaterials are a subgroup of clay materials with the external dimension (thickness) or the internal structural dimension (interlayer distance) in the nanoscale. Clay itself, as most important group of layered nanostructured silicates, refers to naturally occurring or synthetic material composed primarily of fine-grained minerals, which show plasticity through a variable range of water content and will harden when fired or dried. The minerals found in clay are generally silicates of less than 2 micrometres in lateral size. Clays are very abundant at the earth's surface; they form rocks known as shales and are a major component in nearly all sedimentary rocks. The small size of the particles and their unique crystal structures give clay materials special properties, including cation exchange capabilities, plastic behaviour when wet, catalytic abilities, swelling behaviour, and low permeability^[1].

Other than the structure and composition, there are several additional factors which are important in determining the properties and applications of clays and clay nanomaterials (see [Annex A](#)). These are the mineral impurities, the presence of organic materials, the type and amount of exchangeable ions and soluble salts, and the morphological aspects^[2].

Natural and modified clays as layered structured minerals are very important industrial materials. In pristine form, clay materials are normally subnano spaced layers, structured in bundles and in exfoliated state; they are nano-objects with thickness in the nanoscale while in intercalated form they are structured nanomaterials with interlayer space in nanoscale.

Modification of clay with change in its characteristic such as its hydrophobicity, interlayer distance, exchangeable ion, and surface connected groups leads to the extension of its applications e.g. for high performance nanocomposites, effective rheological modifier, or biomedical applications. A small quantity of well dispersed intercalated or exfoliated organo-modified layered clay nanomaterials in polymeric composites (see [Annex B](#)) is proved to show superior impacts on properties such as barrier, tensile modulus, mechanical strength, and flame retardancy.

There are numerous industrial applications for layered clay nanomaterials. Purified and modified clays are used as; coatings on paper to enhance whiteness and to allow the proper absorption of ink, the life time extender of rubber in tires, in concrete, as catalysts in many industries. Moreover, they can also be used in oil purification, pharmaceuticals, ceramic industry, soil stabilization, porcelains and barriers for nuclear and chemical wastes because of their cation-exchange capabilities, low permeability, and long-term structural stability. In addition, layered clay nanomaterials are utilized in purification industries, in agricultural and food engineering applications, polymeric nanocomposites, deodorizer, insecticide carrier, pesticides carrier, drilling fluids, desiccant, detergents, plasticizer, emulsion stabilizer, food additives, cosmetic applications, environmental remediation and many other miscellaneous applications^{[1][2]}.

For such a wide range of clay nanomaterial applications, various fundamental characteristics (as shown in [Table 1](#)) play undeniable roles. These characteristics are measured and reported by the provider of the layered clay nanomaterials. In fact, the determinations of these fundamental and basic characteristics will facilitate the communication between sellers and buyers of these nanomaterials for different applications. These characteristics are considered for all industrial layered clay nanomaterial applications such as nanocomposites, paper, ink, purification, and catalysts. In addition to fundamental characteristics, presented in [Table 1](#), some other optional characteristics of layered clay nanomaterials as shown in [Table 2](#) are measured and reported subject to the agreement between sellers and buyers.

Nanotechnologies — Clay nanomaterials —

Part 1: Specification of characteristics and measurement methods for layered clay nanomaterials

1 Scope

This document specifies characteristics to be measured of layered clay nanomaterials in powder form and chemically modified ones, and describes their relevant measurement methods.

This document does not deal with health, safety and environmental issues.

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 terminological 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/>

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3.1 aspect ratio

ratio of the sheet length to the sheet width

[SOURCE: ISO 8336:2017,3.13]

3.2

bulk density

ratio of the mass of an untapped powder sample and its volume including the contribution of the interparticulate void volume

3.3

cation exchange capacity

amount of exchangeable cations per defined mass of clay nanomaterial sample

3.4

clay

naturally occurring or synthetically manufactured material composed primarily of fine-grained minerals, which is generally plastic at appropriate water contents and will harden when dried or fired

Note 1 to entry: Taken from Reference [3].

Note 2 to entry: Although clay usually contains phyllosilicates, it may contain other materials that impart plasticity and harden when dried or fired. Associated phases in clay may include materials that do not impart plasticity and organic matter. Different disciplines have uniquely defined the size of clay particles, and it is for this reason that “fine grained” is used in the definition rather than a precise value. However, because of these size variations from discipline to discipline, it is important that the particle size be specified in the context of the application.

3.5

clay nanomaterials

material composed predominately of clay with any external dimension in the nanoscale or having internal structure or surface structure in the nanoscale

3.6

exchangeable ion

ions bearing in clays which can be exchanged with other ions

Note 1 to entry: See Reference [4].

3.7

exfoliated clay

state of separating clay layers and distributing individual layers

Note 1 to entry: Usually exfoliation of layered clay nanomaterials is conducted in liquid suspension by giving shear forces.

Note 2 to entry: See Reference [5].

3.8

film formability

ability of clay to form self-standing films (uniform and ordered lamination of clay layers)

Note 1 to entry: See Reference [6].

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3.9

gallery thickness

distance between clay layers

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3.10

intercalated clay

clay in which heterogeneous material (atoms, molecules and nanoparticles) is inserted into a host structure (crystal lattice or other macromolecular structure)

3.11

interlayer distance

distance between identical adjacent layers of clay which is sum of gallery thickness (height) and thickness of a single sheet of clay (d_s)

3.12

layer

discrete material restricted in one dimension, within or at the surface of a condensed phase

[SOURCE: ISO/TS 80004-11:2017, 3.1.2]

3.13

layered clay nanomaterial

clay nanomaterial composed of one or more structural layer

3.14

loss on ignition

dried sample's weight loss during a heat treatment up to 1 000 °C

3.15**moisture content**

ratio of the mass of water contained in a sample to that of the sample

3.16**nanocomposite**

solid comprising a mixture of two or more phase-separated materials, one or more being nanophase

[SOURCE: ISO/TS 80004-4:2011, 3.2]

3.17**nanostructured**

having internal or surface structure in the nanoscale

[SOURCE: ISO/TS 80004-11:2017, 3.1.8]

3.18**organoclay**

modified clay by exchanging the original interlayer cations for organic cations

3.19**phyllosilicate**

silicate mineral, such as mica, the tetrahedral silicate groups of which are linked in sheets

Note 1 to entry: See Reference [7].

3.20**iTeh Standards****smectite**

clay mineral (e.g. bentonite) which undergoes reversible expansion on absorbing water

3.21**Document Preview****specific surface area**

absolute surface area of the sample divided by sample mass

[SOURCE: ISO 9277:2010, 3.15]

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3.22**tap density**

mass of the powder divided by its volume after tapping the sample in powder form

3.23**total surface area**

sum of external and internal surface area

4 Abbreviated terms

AFM Atomic force microscopy

BET Brunauer-Emmett-Teller

FESEM Field emission scanning electron microscopy

FTIR Fourier transform infrared spectrometry

ICP-MS Inductively coupled plasma - mass spectrometry

ICP-OES Inductively coupled plasma - optical emission spectrometry

LOI Loss on ignition

SEM	Scanning electron microscopy
TEM	Transmission electron microscopy
TGA	Thermogravimetric analysis
UV-Vis	Ultraviolet-visible spectrophotometry
XPS	X-ray photoelectron spectrometry
XRD	X-ray diffraction
XRF	X-ray fluorescence

5 Characteristics and measurement methods

5.1 General

This clause provides both fundamental and optional characteristics of layered clay nanomaterials and their relevant measurement methods. Relevant standards describing measurement protocols for individual characteristics are also listed in this clause. However, it should be noted that these standards have not yet been fully validated for application to layered clay nanomaterials.

5.2 Fundamental characteristics

Table 1 lists the fundamental characteristics that are commonly used for material specifications of layered clay nanomaterials. The characteristics for measurements shall be selected from **Table 1** based on the agreement between sellers and buyers. **Table 1** additionally provides information on units suggested to be used for expressing the measurement results of individual characteristics, measurement methods recommended to be used, and other measurement method suggested to use when the recommended measurement methods are not available and existing standards for measurement protocols.

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Table 1 — Fundamental characteristics and the relevant measurement methods

Characteristic		Units	Recommended measurement method(s)	Other measurement methods	Relevant measurement protocols
1-1	Chemical composition content	kg/kg	ICP-MS	ICP-OES or XRF	—
1-2	Mineral composition content	kg/kg	XRD	—	—
1-3	Interlayer distance	Nm	XRD	TEM or FESEM	—
1-4	Thickness	Nm	AFM	XRD, TEM or FESEM	—
1-5	Aspect ratio	—	AFM	SEM, FESEM or TEM	—
1-6	Bulk density	kg/m ³	Gravimetry and volumetry	—	Japanese Pharmacopoeia: 3.01 ^[14]
1-7	Cation exchange capacity	cmol+/kg	Schollenberger method	—	ISO 23470:2018
1-8	Loss on ignition	kg/kg	Thermogravimetry	Heating and weighing method	ISO/TR 18230:2015
1-9	Water absorption capacity	kg/kg	Water absorption method	Enslin-Neff method	ISO 10769:2011
1-10	Moisture content	kg/kg	Thermogravimetry	Oven-drying method	ISO 10769:2011

5.3 Optional characteristics

In addition to the fundamental characteristics which shall be measured (Table 1), there are some other important characteristics which could be related to specific applications. The optional characteristics listed in Table 2 should be measured subject to the agreement between buyers and sellers. Table 2 additionally provides information on units suggested to be used for expressing the measurement results of individual characteristics, measurement methods recommended to be used, other measurement method suggested to use when the recommended measurement methods are not available and existing standards for measurement protocols.

Table 2 — Optional characteristics of layered clay nanomaterials and relevant measurement methods

Characteristic		Units	Recommended measurement method(s)	Other measurement methods	Relevant measurement protocols
2-1	Brightness	—	Reflectometry	—	TAPPI T646
2-2	Colour	—	Colorimetry	—	—
2-3	Methylene blue adsorption capacity	mmol/100g	Filter paper method	UV-Vis spectrophotometry	ASTM C837 – 09 (2014)
2-4	Cohesion coefficient	kPa	Direct Shear test	—	ISO 17892-7:2017/ASTM D3080/D3080M
2-5	Tap density	kg/m ³	Gravimetry and volumetry	—	Japanese Pharmacopoeia: 3.01 ^[14] and European Pharmacopoeia ^[15]