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Nanotechnologies — Polymeric nanocomposite films for food packaging with barrier properties — Specification of characteristics and measurement methods

Nanotechnologies — Films de polymères nanocomposites pour emballages alimentaires avec les propriétés barrières — Spécification des caractéristiques et méthodes de mesure

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Foreword

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This document was prepared by Technical Committee ISO/TC 229, *Nanotechnologies*.

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Introduction

The rapid market growth of emerging packaging containing nano-objects is due to the effects this packaging has on improving food shelf life and decreasing food waste. In addition, the increasing export and import of food and food products is creating a growing future demand for nano-enhanced packaging.

Typical plastics used for packaging are polyethylene, polypropylene, polyamide and polyester. The presence of nano-objects in packaging can enhance various characteristics of the polymeric films such as gas/water vapour barrier properties, UV-Vis light transparency, thermal properties and mechanical strength. One of the key purposes of such packaging is to deliver longer shelf life by improving the barrier properties of food packaging to reduce gas diffusion, water vapour exchange and UV-Vis light exposure[1]. The effect of gas, water vapour and UV-Vis light permeability of food packaging on the shelf life is described in Annex A. Various types of nano-objects, such as clay nanoplates, zinc oxide nanoparticles/nanorods, titanium oxide nanoparticles, have been incorporated into the polymeric matrix to improve the above-mentioned barrier properties.

In contrast to glass or metal packaging materials, polymeric materials are permeable to small molecules of gas(es) and water vapour as well as UV-Vis light. The possibility to improve the barrier properties of polymer packaging by the application of nanocomposites is a very attractive field. The principal factors affecting the permeability of the original polymer matrix and the nanocomposite are the crystallinity and crystal phases of the polymer, the state of dispersion and orientation of nano-objects in the nanocomposite, etc. (see Annexes B and C).

In general, for a successful application of nano-enhanced barrier food packaging, it is required:

— to define the relationship among composition, structure and properties;

— to identify characteristics and their measurement methods.

This document specifies the characteristics including barrier properties to be measured of polymeric nanocomposite films. It also recommends the relevant measurement methods for the characteristics. This document is expected to promote communication and mutual understanding of polymeric nanocomposites for food packaging application between buyers and sellers.

Nanotechnologies — Polymeric nanocomposite films for food packaging with barrier properties — Specification of characteristics and measurement methods

1 Scope

This document specifies characteristics including barrier properties to be measured of polymeric nanocomposite films used for improving food packaging. The barrier properties cover gas (oxygen), water vapour transmission and UV-Vis light transparency. This document also describes the relevant measurement methods.

This document addresses neither safety and health issues related to the food packaging nor environmental aspects.

2 Normative references

There are no normative references in this document.

3 Terms, definitions and abbreviated terms

3.1 Terms and definitions

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/

3.1.1
glass transition temperature
characteristic value of the temperature range over which the glass transition takes place

Note 1 to entry: Glass transition is a reversible change in an amorphous polymer or in amorphous region of partially crystalline polymer between a viscous or rubbery condition and a hard and relatively brittle one.

[SOURCE: ISO 11357-2:2020, 3.1, modified — Note 1 to entry has been replaced.]

3.1.2
melting temperature
temperature range over which crystalline or *semi-crystalline polymers* (3.1.7) lose their crystalline characteristics or particulate shape to produce a liquid, when heated

[SOURCE: ISO 472:2013, 2.584, modified — The definition has been reworded.]

3.1.3
nanocomposite
solid comprising a mixture of two or more phase-separated materials, one or more being nanophase

Note 1 to entry: Polymer matrix nanocomposite is referred to nanocomposite with at least one major polymeric phase.

[SOURCE: ISO/TS 80004-4:2011, 3.2, modified — Note 1 to entry has replaced the original Notes 1 and 2 to entry.]

3.1.4
nano-enhanced
exhibiting function or performance of materials intensified or improved by nanotechnology

[SOURCE: ISO/TS 80004-1:2015, 2.16, modified — “of materials” has been added.]

3.1.5
oxygen transmission rate
volume or amount of oxygen gas passing through a plastic material, per unit area and unit time, under unit partial-pressure difference between the two sides of the material

Note 1 to entry: The oxygen transmission rate in terms of volume is generally expressed in cubic centimetres per square metre, per 24 h and per atmosphere [$cm^3/(m^2 \cdot 24\ h \cdot atm)$], the volume of the gas being converted to standard conditions under a pressure difference of one atmosphere.

Note 2 to entry: The oxygen transmission rate in terms of amount is expressed in moles per square metre, per second and per pascal [$mol/(m^2 \cdot s \cdot Pa)$].

[SOURCE: ISO 15105-1:2007, 3.1, modified — “oxygen” has replaced “gas” in the term and “or amount of oxygen gas” has replaced before “of gas” in the definition. Notes 1 and 2 to entry have replaced the original note.]

3.1.6
packaging
product to be used for the containment, protection, handling, delivery, storage, transport and presentation of goods, from raw materials to processed goods, from the producer to the user or consumer, including processor, assembler or other intermediary

[SOURCE: ISO 21067-1:2016, 2.1.1]

3.1.7
semi-crystalline polymer
polymer containing both crystalline and amorphous phases, which may be present in varying proportions

[SOURCE: ISO 3146:2000, 3.1]

3.1.8
tortuous path
path of the gas passing through a polymeric matrix via passive shielding

3.1.9
UV-Vis transmittance
ratio of the radiant flux of a UV-Vis beam going through a film sample to that of the UV-Vis beam without the film sample

3.1.10
water vapour transmission rate
mass of water vapour transmitted through a unit area in a unit time under specified conditions of temperature and humidity

Note 1 to entry: Water vapour transmission rate is expressed in grams per square metre and per 24 h [$g/(m^2 \cdot 24\ h)$].

Note 2 to entry: Adapted from ISO 15105-1:2007, 3.1.

3.2 Abbreviated terms

AFM	atomic force microscopy
DLS	dynamic light scattering
DSC	differential scanning calorimetry
GC	gas chromatography
ICP/AES	inductively coupled plasma atomic emission spectroscopy
ICP/MS	inductively coupled plasma mass spectrometry
OTR	oxygen transmission rate
PTA	particle tracking analysis
SAXS	small angle X-ray spectroscopy
SEM	scanning electron microscopy
TEM	transmission electron microscopy
TGA	thermogravimetric analysis
UV-Vis	ultraviolet-visible
WVTR	water vapour transmission rate
XRD	X-ray diffraction
XRF	X-ray fluorescence

4 Essential and optional characteristics to be measured and their measurement methods

4.1 General

The characteristics to be measured of polymeric nanocomposite film are classified into two groups: essential characteristics and optional ones. The essential characteristics listed in Table 1 shall be measured. The optional characteristics listed in Table 2 are provided for information. These characteristics may be useful to measure depending on specific applications.

Table 1 — Essential characteristics to be measured and their measurement methods

Item	Characteristics		Measurement method
Nano-object	Size and size distribution		See 4.2.1
	Chemical composition content		See 4.2.2
Nanocomposite	Total luminous transmittance		See 4.3.1
	Ash content		See 4.3.2
	Barrier properties	OTR	See 4.3.3.2
		WVTR	See 4.3.3.3
		UV-Vis transmittance	See 4.3.3.4

Table 2 — Optional characteristics to be measured and their measurement methods

Item	Characteristics	Measurement method
Nano-object	Colour	See 4.4.1
	Morphology	See 4.4.2
Nanocomposite	Appearance	See 4.5.1
	Mechanical properties	See 4.5.2
	Melting temperature	See 4.5.3.1
	Glass transition temperature	See 4.5.3.2
	Crystalline phase type and crystallinity	See D.1
	Morphology	See D.2

4.2 Nano-object (essential characteristics)

4.2.1 Size and size distribution

4.2.1.1 General

The barrier properties of polymeric nanocomposite film are sensitive to the size of nano-objects incorporated into the polymeric matrix.

Nano-objects are three-dimensional objects with different shapes. It is impossible to represent the size of nano-object using a single number. Consequently, in most techniques it is assumed that the shape is spherical because a sphere is the shape that can be represented by a single number, its diameter (see ISO 19430:2016).

A test specimen for measurements of size and size distribution is taken from the nano-object raw material sample and a suspension is prepared.

The average size of a nano-object shall be measured using an appropriate measurement method and, if possible, specifying if the nano-object measured is primary or secondary (agglomerate). The measurement results shall be expressed in the unit of nm.

An appropriate measurement method from among SAXS, electron microscopy (TEM and SEM), DLS, AFM and PTA is recommended to be taken for measuring the average diameter of nano-objects.

NOTE 1 In most cases, the measured size can be of a secondary nano-object because of agglomeration. To inhibit agglomeration, an appropriate sample preparation is necessary.

NOTE 2 Ultra-sonication of the suspension containing a nano-object is an appropriate method before size measurement by the above mentioned methods.

4.2.1.2 Small angle X-ray spectroscopy

The size of nano-objects in liquid medium can be measured via SAXS. The SAXS technique is used to measure the primary and secondary nano-object size distribution, and primary and secondary nano-object average size.

NOTE ISO 17867:2015 specifies a method for the application of SAXS to the estimation of average nano-objects sizes in dilute dispersions where the interaction between the nano-object is negligible. Both number- and volume-based size distribution is measured via the SAXS method.

4.2.1.3 Electron microscopy

The size of nano-objects can also be measured by electron microscopy. TEM and SEM are used for size measurement of nano-objects (see ISO 21363 and ISO 19749, respectively). TEM and SEM methods provide two-dimensional images of the nano-object, which are number-based size distribution.

4.2.1.4 Particle tracking analysis

The size of nano-objects in a liquid can also be measured by PTA where the Brownian motion of nano-objects is traced optically. ISO 19430:2016 describes the evaluation of the number-based nano-object size distribution in liquid dispersions (solid, liquid or gaseous particles suspended in liquids) using the PTA method for diffusion velocity measurements.

4.2.1.5 Dynamic light scattering

The size of nano-objects in a liquid can also be measured by DLS. Particle size analysis is performed using the DLS method, which probes the hydrodynamic mobility of the particles. ISO 22412:2017 provides estimates of the average particle size and size distribution.

4.2.1.6 Atomic force microscopy

The size of nano-objects in dry form on a flat substrate can also be measured by AFM using height measurement (z-displacement). AFM provides a three-dimensional surface profile. While the lateral dimensions are influenced by the shape of the probe, displacement measurements can provide the height of nanoparticles with a high degree of accuracy and precision (see ASTM E2859-11).

4.2.1.7 Laser diffraction

The size of nano-objects and size distribution in many two-phase systems (e.g. powders, sprays, aerosols, suspensions, emulsions and gas bubbles in liquids) can be measured through the analysis of their light-scattering properties. In laser diffraction measurement, a laser beam is passed through a well-dispersed particle sample and particle size is measured by detecting the intensity of the scattered light produced (see ISO 13320).

The laser diffraction method is often applied to nanoplates the lateral size of which is larger than 100 nm.

4.2.2 Chemical composition content

4.2.2.1 General

A test specimen for measurements of chemical composition content is taken from the nano-object raw material sample and a dried powder is prepared. The chemical composition of nano-objects, i.e. elemental and compound compositions, of a nano-object raw material sample is one of the essential characteristics because it can influence the final products properties. The chemical composition includes major composition and minor composition (impurities). The chemical composition content is the ratio of the mass of an element or compound contained in a dried powder sample of nano-object raw material to that of the dried powder sample.

The chemical composition content shall be measured using an appropriate method. XRD, XRF, energy dispersive X-ray analysis, inductively coupled plasma/optical emission spectroscopy (ICP/OES) and /mass spectroscopy (ICP/MS) are recommended to be used for chemical composition content measurements.

The results of chemical composition content measurements shall be expressed in the form of element type, element content (kg/kg), chemical compound type and chemical compound content (kg/kg).

4.2.2.2 X-ray diffraction

XRD can identify the chemical compound type for a nano-object raw material sample.

The XRD technique, by way of the study of the crystal structure, can be used to identify the crystalline phases present in a material and chemical composition. Identification of phases is carried out by comparison of the achieved data to that in reference databases. The chemical compound content is measured via the ratio of the intended chemical compound's peak intensity to the base material chemical compound's peak intensity.