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Functional pigments and extenders for special applications —

Part 1: Nanoscale calcium carbonate for sealant application

Pigments et matières de charges fonctionnels pour applications spéciales —
Partie 1: Carbonate de calcium nanométrique pour les enduits

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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 on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 256, *Pigments, dyestuffs and extenders*.

ISO 18473 consists of the following parts, under the general title *Functional pigments and extenders for special applications*:

— *Part 1: Nanoscale calcium carbonate for sealant application*

— *Part 2: Nanoscale titanium dioxide for sunscreen application*

Introduction

Sealants are widely used to prevent the penetration of air, gas, or liquid in many industries including construction, automobile, and electronics. Extenders are essential part of sealant formulation to reduce cost and improve their rheological and mechanical properties. Content of extenders vary significantly and can be as high as 50 % (mass fraction). The most common extender in sealants is calcium carbonate ($CaCO_3$) because it is readily available and comes in various sizes which act as a rheological modifier, a reinforcing agent, and opacifier. Commercial calcium carbonate can be divided into ground calcium carbonate (GCC) and precipitated calcium carbonate (PCC), possesses three crystal structures including calcite, aragonite, and vaterite, and exists in various morphologies such as cubic, spherical, spindle, fibrous, needle-like, etc. Calcium carbonates with calcite crystal structure and cubic or spherical morphology are most widely used in sealant applications.

Nanoscale calcium carbonate (NCC) provides various sealants with improved performance and additional functionalities including thixotropy, flame resistance and improved durability and recyclability, and has become a major component in sealant formulation. Nanoscale calcium carbonate in the powdered form is readily manufactured nowadays, mostly through precipitation route to control the size and morphology. Surface treatment is crucial for utilizing NCC in sealants. Native $CaCO_3$ is hydrophilic. As a result, it tends to agglomerate in organic polymers and plasticizers. NCC, in particular, has a greater propensity for agglomeration because of its small size and large specific surface area. NCCs are surface treated to render them hydrophobic and improve their dispersibility in hydrophobic systems. Surface treatment also improves polymer matrix compatibility, thus improving interfacial adhesion between extender and polymer.

It has been found that the particle size, specific surface area, mass fraction, morphology, pH value, magnesium content, oil absorption value, moisture content, and other characteristics of supplied nanoscale calcium carbonate all have impact on the performance of the sealant incorporating these nanoparticles. The need to specify the characteristics of NCC which relate to sealant performance comes from the following facts. First, the agreements between customers and suppliers do not always cover all material characteristics that have influences on performance and/or processability of sealants or they have been interpreted differently by the customers and suppliers. Second, nanomaterials are relatively new. Material properties can depend on the techniques to measure them. Therefore, providing information regarding characteristics of nanoscale calcium carbonate in sealants will facilitate the communication between customers and suppliers.

This part of ISO 18473 lists the properties, measurements, and characteristics of nanoscale calcium carbonate and intents to aid its acceptance and application in sealants.

Functional pigments and extenders for special applications —

Part 1:
Nanoscale calcium carbonate for sealant application

1 Scope

This part of ISO 18473 specifies requirements and corresponding methods of test for surface treated nanoscale calcium carbonate in powder form for sealant application.

2 Normative references

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

ISO 787-2, *General methods of test for pigments and extenders — Part 2: Determination of matter volatile at 105 °C*

ISO 787-5, *General methods of test for pigments and extenders — Part 5: Determination of oil absorption value*

ISO 787-9, *General methods of test for pigments and extenders — Part 9: Determination of pH value of an aqueous suspension*

ISO 3262-1, *Extenders for paints — Specifications and methods of test — Part 1: Introduction and general test methods*

ISO 3262-6, *Extenders for paints — Specifications and methods of test — Part 6: Precipitated calcium carbonate*

ISO 9277, *Determination of the specific surface area of solids by gas adsorption — BET method*

ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

3 Terms and definitions

For the purposes of this part of ISO 18473, the following terms and definitions apply.

3.1
nanoscale
size range from approximately 1 nm to 100 nm

Note 1 to entry: Properties that are not extrapolations from a larger size will typically, but not exclusively, be exhibited in this size range. For such properties, the size limits are considered approximate.

Note 2 to entry: The lower limit in this definition (approximately 1 nm) is introduced to avoid single and small groups of atoms from being designated as nano-objects or elements of nanostructures which can be implied by the absence of a lower limit.

[SOURCE: ISO/TS 80004-2:2015, 2.1]

3.2
precipitated calcium carbonate
synthetic calcium carbonate consisting of trigonal crystals (like those of calcite) or rhombic bipyramidal crystals (like those of aragonite)

[SOURCE: ISO 3262-6:1998, 3.1]

4 Requirements and test methods

Nanoscale calcium carbonate (powdered form) applied for industrial use of sealant shall comply with the requirements specified in Table 1.

Table 1 — Requirements for nanoscale $CaCO_3$ used in sealant

Characteristic/Property	Unit	Requirement	Test method
Average crystal size, mass based[a]	nm	≤100	XRD[b] method
Median particle size by mass distribution (particle size distribution is optional)[a]	nm	[c]	Centrifuge method[d]
Median particle size by number distribution (particle size distribution is optional)[a]	nm	[c]	TEM[e] method
Specific surface area (BET)[f]	m^2/g	≥16	ISO 9277
Mass fraction of calcium carbonate	% (mass fraction)	≥85	ISO 3262-1
Insoluble residue in hydrochloric acid	% (mass fraction)	≤0,5[g]	ISO 3262-6
$MgCO_3$ content	% (mass fraction)	To be agreed between the interested parties (if there is no agreement, it is not necessary to measure)	ISO 3262-1
DINP[d] or linseed oil absorption	ml/100 g	≥23	ISO 787-5
Moisture	% (mass fraction)	To be agreed between the interested parties	ISO 787-2
Whiteness	—	To be agreed between the interested parties	Clause 6
pH value	—	To be agreed between the interested parties	ISO 787-9

[a] Characteristic for particle size to be agreed between the interested parties.
[b] XRD — X-ray diffraction.
[c] A requirement can only be fixed as soon as a standardized test method is available.
[d] Standard under development.
[e] TEM — Transmission electron microscopy.
[f] BET — Brunauer-Emmett-Teller.
[g] The insoluble residue content is measured with the untreated $CaCO_3$.

Table 1 *(continued)*

Characteristic/Property	Unit	Requirement	Test method
Amount of organic treatment and general chemical type (surface treatment)	% (mass fraction)	To be agreed between the interested parties	To be agreed between the interested parties
a Characteristic for particle size to be agreed between the interested parties.			
b XRD — X-ray diffraction.			
c A requirement can only be fixed as soon as a standardized test method is available.			
d Standard under development.			
e TEM — Transmission electron microscopy.			
f BET — Brunauer-Emmett-Teller.			
g The insoluble residue content is measured with the untreated $CaCO_3$.			

NOTE The crystal type and particle shape are important, but not required for decision for a nanoscale material or not.

5 Sampling

Take a representative sample of the product to be tested as described in ISO 15528. Sample preparation shall always be carried out consistently so that repeated preparations based on replicate samples of a batch of powder (which was carefully mixed before being sampled or subdivided into samples) give closely comparable results.

6 Determination of whiteness

6.1 Apparatus

Use ordinary laboratory apparatus and glassware together with the following.

6.1.1 **Whiteness photometer or colour photometer**.

The apparatus shall have the following characteristics:

a) wavelength range from 380 nm to 780 nm, shall not exceed the range of 400 nm to 700 nm;

b) halfwidth of wavelength: the half-width of wave emitted from exit slit of the photometer shall be in the range of 10 nm;

c) luminous accuracy shall be in the 0,5 % of the full-scale luminous range;

d) deviation between the nominal wavelength and actual wavelength shall not exceed 0,5 nm.

6.1.2 **Powder sample press**.

6.2 Procedure

6.2.1 Sample preparation

Place a quantity of sample into the pressing sampling implement and press it into a surfacing sample board without veining, staining, and spotting. Three sample boards shall be pressed for each batch of products.