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Nanotechnologies — Textiles containing nanomaterials and nanostructures — Superhydrophobic characteristics and durability assessment

Nanotechnologies — Textiles contenant des nanomatériaux et des nanostructures — Caractéristiques superhydrophobiques et évaluation de la durabilité

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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

Recently superhydrophobic textiles (woven and nonwoven) have gained significant scientific and industrial interest for its potential applications in outdoor wear and protective clothing. The superhydrophobic textile surfaces refer to superior water repellency with a water contact angle exceeding 150° and low contact angle hysteresis of less than 10° (see Annex A). For this superhydrophobic textile, dirt and soils are loosely attached, and a rolling water drop can easily attach and remove them from the surface, giving self-cleaning properties. According to Young's, Wenzel and Cassie-Baxter Models superhydrophobicity of textile surface can be made by both the surface treatment with very low surface free energy materials and making nano-roughness (see Annex B).

Nanotechnology is employed to artificially change the surface free energy and/or cause nano- roughness on the surface. The following methods are normally utilized in this respect:

— using nano-objects such as silica, TiO_2, CNT, ZnO, etc., in various ways;

— surface etching, i.e. nano roughening (UV-laser or plasma), followed by grafting or physically/chemically attaching compounds with low surface energy;

— using nanofibres.

The establishment of superhydrophobic relies on

a) superhydrophobic (non-polar) surface chemistry, and

b) nanostructured surface texture (nano-roughness).

One of the most important obstacles affecting the market growth of textiles containing nanomaterials and nanostructures (TCNNs) showing superhydrophobic response is their relevant durability under different utilization and working conditions. This includes, laundering (washing), ironing, mechanical abrasion (rubbing) and light radiation exposure. If superhydrophobic properties are not durable, the TCNNs are useless in long term applications. Therefore, durability of superhydrophobic TCNNs over repeated use and wash are necessary.

In this regard, the durability and persistence of superhydrophobic behaviour of TCNNs needs to be assessed under above mentioned condition based on standard methods. Generally, from the consumer's perspective, the superhydrophobic durability of TCNNs is very important. However, there is no specific measurement method to evaluate the superhydrophobic durability. In fact, there is a lack of grading procedure for this characteristic.

This document both specifies the characteristics, performance and durability of the TCNNs subjected to laundry (washing), ironing, mechanical abrasion (rubbing) and light exposure. The superhydrophobic durability of such textiles are assessed and reported based on contact angle and hysteresis measurement of the samples before and after subjected to mentioned conditions. In fact, a specific grading method is established in this document. Further, this document also recommends relevant measurement methods to promote communication and mutual understanding of TCNNs for superhydrophobic application between buyers and sellers.

This document supports less water consumption and less waste water production. In addition, this document supports responsible production in terms of superhydrophobic durability of textile. Furthermore, this document can provide a potential for the economic growth for small and medium size enterprises. These items conform with several Sustainability Development Goals (SDGs) defined by United Nations.

Nanotechnologies — Textiles containing nanomaterials and nanostructures — Superhydrophobic characteristics and durability assessment

1 Scope

This document specifies the characteristics and performance(s) of the superhydrophobic textiles containing nanomaterials and nanostructures (TCNNs) based on contact angle measurement before and after being subjected to washing/drying (laundry), ironing processes, light sources and abrasion, that are to be determined by agreement between customer and supplier. This document solely covers woven and nonwoven fabrics.

This document does not address safety and health related issues.

2 Normative references

There are no normative references in this document.

3 Terms, definitions and abbreviated terms

3.1 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:

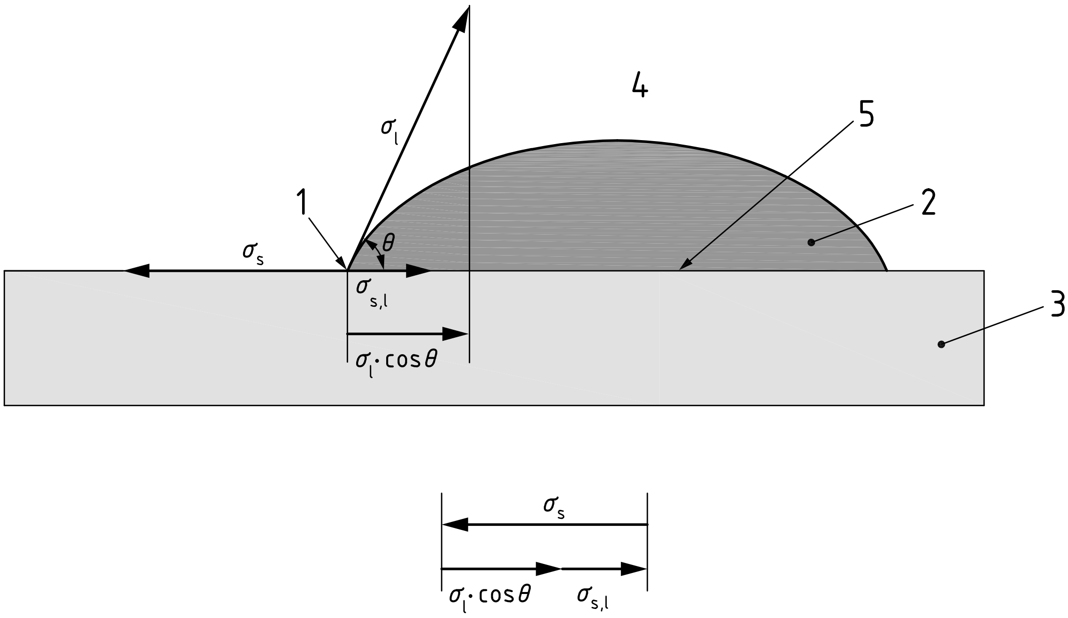
— ISO Online browsing platform: available at https://www.iso.org/obp

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

3.1.1
contact angle
θ
angle to the base line within the drop, formed by means of a tangent on the drop counter through one of the three-phase points

Note 1 to entry: See Figure 1.

Note 2 to entry: The contact angle is preferably indicated in degrees (°). 1° = (π/180) rad. If the system is in thermodynamic equilibrium, this contact angle is also referred to as thermodynamic equilibrium contact angle.



Key

1 three-phase point

2 liquid phase

3 solid phase

4 gas phase

5 base line

σ_l surface tension of the liquid surface

σ_s surface free energy of the solid surface

$\sigma_{s,l}$ interfacial energy between solid surface and liquid surface

θ contact angle

Figure 1 — Illustration of a contact angle in a wetting equilibrium

[SOURCE: ISO 19403-1:2022, 3.1.9, modified — "Illustration of a contact angle in a" has been added to the title of Figure 1.]

3.1.2
contact angle hysteresis
θ_{ar}
difference between the advancing angle and the receding angle

[SOURCE: ISO 19403-6:2017, 3.4]

3.1.3
nano-roughness
surface texture in the nanoscale

3.1.4
textile containing nanomaterials and nanostructures
TCNNs
textile products incorporated by nanotechnologies in the form of coatings, treatments, fibre material composites and nanoscale fibres

Note 1 to entry: TCNNs have been subdivided into three major types[1]:

— nanofinished textiles;

— nanocomposite textiles;

— nanofibrous textiles.

3.1.5
superhydrophobic surface
surface made from hydrophobic material for which the *contact angle* (3.1.1) with a water droplet exceeds 150° and *contact angle hysteresis* (3.1.2) is less than 10°

3.1.6
superhydrophobic durability
ability of superhydrophobic properties to withstand washing, ironing, abrasion and light exposure

Note 1 to entry: Durability means "ability to exist for a long time without significant deterioration in quality or value".

3.1.7
wettability
degree of wetting

Note 1 to entry: *Contact angle* (3.1.1) $\theta = 0°$ indicates a fully wetted surface and $\theta = 180°$ indicates a not wetted surface.

[SOURCE: ISO 19403-1:2022, 3.3.2]

3.2 Abbreviated terms

AFM	Atomic force microscopy
EDX	Energy dispersive X-ray analysis
ICP/AES	Inductively coupled plasma atomic emission spectroscopy
ICP/MS	Inductively coupled plasma mass spectrometry
ICP/OES	Inductively coupled plasma optical emission spectroscopy
SAXS	Small angle X-ray spectroscopy
SEM	Scanning electron microscopy
SPM	Scanning probe microscopy
TEM	Transition electron microscopy
XRD	X-ray diffraction
XRF	X-ray fluorescence

4 Mandatory and recommended measurement characteristics and their measurement methods

4.1 General

The characteristics to be measured of TCNNs are classified into two groups; mandatory characteristics and recommended ones. The mandatory characteristics listed in Table 1 shall be measured, and the recommended characteristics listed in Table 2 are provided for information. The recommended characteristics of TCNNs listed in Table 2 can be useful to measure depending on the application.

All measurements shall be carried out before and after ageing for durability assessment.

NOTE 1 The ageing for durability assessment has been explained in 4.2.

NOTE 2 Sampling method can be determined according to ISO 2859-1 or a procedure determined between the user and the manufacturer.

Table 1 — Mandatory measurement characteristics and their measurement methods for superhydrophobic durability

Item	Characteristics	Measurement method
Nanomaterials/nanostructure	Size and size distribution	See 4.3
	Morphology	See 4.3
	Chemical composition	See 4.4
Superhydrophobicity	Contact angle	See 4.5
	Contact angle hysteresis	See 4.5

Table 2 — Recommended measurement characteristics of TCNNs and their measurement methods

Item	Characteristics	Measurement method
Nanomaterials/nanostructures	Phase analysis	See 4.4
Superhydrophobicity	Nano-roughness	See 4.3

4.2 Ageing for superhydrophobic durability assessment

4.2.1 General

The durability of superhydrophobicity of TCNNs can be changed by ageing process. The ageing includes heat, abrasion, laundering and light exposure. In fact, the superhydrophobicity of the TCNNs depends on existence and quality of the nano-roughness on the fibres' surfaces. The ageing process may change or destroy the surface nano-roughness. Therefore, contact angle and contact hysteresis shall be measured before and after ageing process to evaluate the durability of superhydrophobicity of the TCNNs. The ageing process may be due to the processes listed in 4.2.2 to 4.2.5.

4.2.2 Washing and dry cleaning

As most textile fabrics undergo repeated laundering and dry cleaning during their lifetime, the washing and dry cleaning durability of such highly hydrophobic fabric is of significant importance. Domestic washing and dry cleaning shall be carried out in accordance with manufacturer instructions.

NOTE If the manufacturer does not give instruction, guidance can be taken from ISO 6330.

Different washing machine type, detergent type and type of drier can affect the test results. Therefore, the parties should agree on above mentioned parameters.

4.2.3 Ironing

Ironing can affect the superhydrophobic durability and performance of TCNNs for superhydrophobicity. Ironing/steam ironing procedure shall be performed under the conditions agreed between the user and the buyer.

4.2.4 Mechanical abrasion

Mechanical abrasion (rubbing) is one of the processes that can affect the superhydrophobic durability of TCNNs. In this respect, mechanical abrasion effect shall be applied in accordance with ISO 105-

X12 followed by assessment of superhydrophobic durability before and after being subjected due the abrasion process.

The rubbing finger shall exert a downward force of 9 N ± 0,2 N, moving to and fro in a straight line along a 104 mm ± 3 mm track.

4.2.5 Light exposure

Light exposure is one of the processes that can affect the hydrophobic durability of TCNNs. Light exposure is performed according to ISO 105-B01. The exposure device shall provide for placement of specimens and any designated sensing devices in positions that allow uniform irradiance from the light source. The relative spectral irradiance produced by the device should be a very close match to that of solar radiation, especially in the short wavelength UV region. Exposure devices shall be designed such that the variation in irradiance at any location in the area used for specimen exposure shall not exceed ±10 % of the mean. The configuration of the lamp with respect to the specimens on exposure, including the differences in distance between the lamp(s) and the samples can affect uniformity of exposure.

To simulate different environments, testing can be carried out under different conditions. The type of conditions should be agreed between parties. The chosen conditions shall be reported (exposure cycle A1, A2, A3 and B).

4.3 Nanomaterial and nanostructure evaluation

4.3.1 General

Size and size distribution, nano-roughness, morphology and chemical composition of nanomaterials and nanostructure in TCNNs can be evaluated.

4.3.2 Size and size distribution

4.3.2.1 General

The superhydrophobic properties and superhydrophobic durability of TCNNs are sensitive to the size and size distribution of nano-objects incorporated into or coated on the fibres as well as nanostructure (nano-roughness).

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).

Nanostructured materials have internal or surface structure in the nanoscale.

A test specimen for measurements of size and size distribution is taken from the TCNNs sample. The average size of a nano-object shall be measured using an appropriate measurement method. The measurement results shall be expressed in the unit of nanometres.

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