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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*.

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

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:

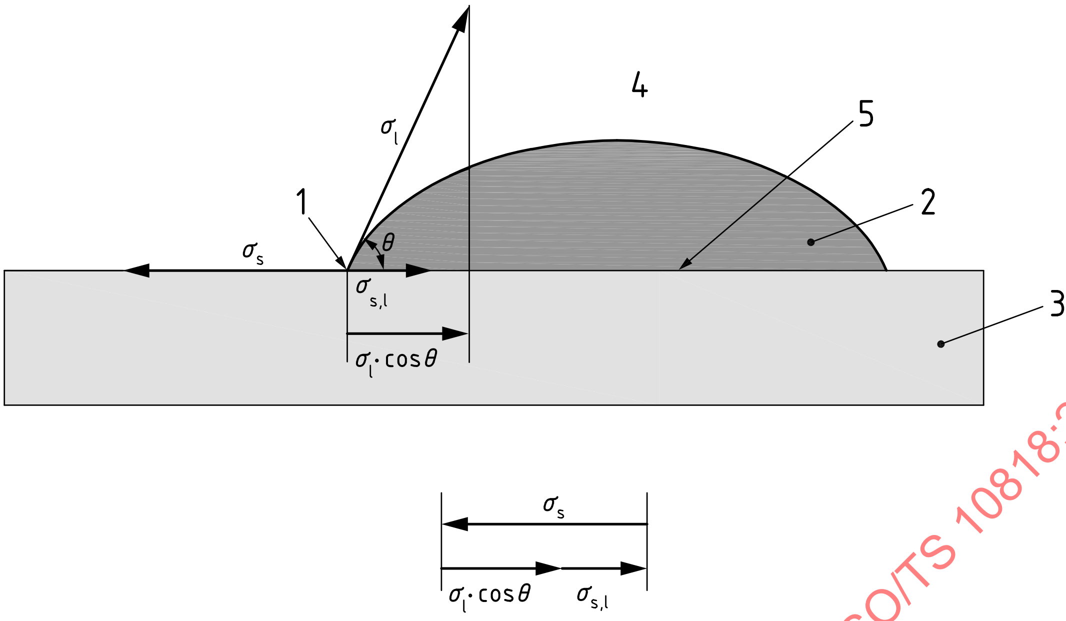
— ISO Online browsing platform: available at https://www.iso.org/obp

— 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) θ = 0° indicates a fully wetted surface and θ = 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.

4.3.2.2 Small angle X-ray spectroscopy

The size of nano-objects in solid 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 specifies a method for the application of SAXS to the estimation of average nano-objects sizes distributed in solid phase where the interaction between the nano-object is negligible. Both number- and volume-based size distributions is measured via the SAXS method.

4.3.2.3 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.

NOTE 1 For the case of nano-object incorporated in a fibre matrix of TCNNs, (cryo) ultramicrotomy can be utilized to prepare samples for TEM.

NOTE 2 SEM and AFM can be utilized for size measurement of nano-object coated on the fibres in TCNNs.

4.3.2.4 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).

4.3.3 Nano-roughness (recommended characteristics)

4.3.3.1 General

The superhydrophobic properties of TCNNs are sensitive to nano-roughness/nano-texture on fabric fibres. In order to observe the nano-roughness, scanning probe microscopy (SPM) methods should be utilized to evaluate nano-roughness of the superhydrophobic textiles. Surface microscopy should be employed to image test surfaces and fabric samples before and after ageing process/es. Both AFM and STM are appropriate for surface topography, however, the size of nanomaterials with 3D morphology are difficult to determine. It is impossible to represent the size of nanomaterials using a single number.

The measurement results shall be expressed in the form of graphical representation or surface porfilometry in nanometres (depth and width).

NOTE It can be assumed that the nano-roughness shape is cylindrical because a cylinder is the shape that can be represented by two numbers: its diameter (width) and its height (depth).

A test specimen for measurements of depth and width and morphology is taken from the TCNNs sample.

4.3.3.2 SPM

An appropriate method for graphical measurement is SPM. SPM is also recommended to be taken for measuring the average depth and width of the nano-roughness.

SPM 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 nano-roughness with a high degree of accuracy and precision (see the ISO 25178 series).

4.3.4 Morphology

Superhydrophobic TCNNs can contain nano-object and nanostructure. The nano-object can be in the form of nanofibre, nanoplate and nanoparticle. The morphology of nano-objects may affect superhydrophobicity and superhydrophobic durability of TCNNs. The morphology of nano-objects in a raw material is observed using SEM, TEM and SPM techniques. Microscopic images should have the scale bars. The number of images to be taken can be decided between the interested parties.

4.3.5 Chemical composition

4.3.5.1 General

A test specimen for measurements of chemical composition is taken from the TCNNs. The chemical composition of nano-objects (i.e. the elemental and compound compositions of a nanomaterials incorporated into or coated on the textile) is one of the mandatory characteristics because it can influence the final products properties.

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

NOTE For the determination of chemical composition, the TCNNs specimen is cut into small pieces and is used in techniques such as XRD, XRF and energy dispersive X-ray analysis and/or extracted with acidic artificial perspiration solution to be used in techniques such as ICP/OES and ICP/MS.

4.3.5.2 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 (phase analysis) present in a material and chemical composition. Identification of phases is carried out by comparison of the achieved data to that in reference databases.

4.3.5.3 X-ray fluorescence analysis

XRF analysis can identify the type of elements in a nano-object raw material sample.

XRF analysis can be used for a quantitative determination of major and trace element concentrations in homogeneous powder using a calibration with standard sample of same matrix (see ISO 18227).

4.3.5.4 Energy dispersive X-ray analysis

Energy dispersive X-ray analysis can identify the type of the elements in a nano-object raw material sample fitted to a scanning electron microscope or an electron probe micro-analyser (see ISO 22309).

4.3.5.5 Inductively coupled plasma

ICP/AES and ICP/MS (see ISO 17294-1) can measure the type of elements in a nano-object raw material sample.

NOTE 1 Solution test specimen is prepared using an appropriate solvent for the ICP technique.

NOTE 2 EN 16711-2 can be employed for determination of metal content of superhydrophobic textile. EN 16711-2 specifies a procedure for determination of antimony (Sb), arsenic (As), cadmium (Cd), cobalt (Co), copper (Cu), lead (Pb), mercury (Hg), nickel (Ni), in textile after extraction with acidic artificial perspiration solution.

4.3.5.6 Phase analysis (recommended characteristic)

XRD can identify the chemical compound type for a nanomaterial(s) incorporated into or coated on textile fibres.

The XRD technique, by way of the study of the crystal structure, should 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.

NOTE The powder for XRD test can be taken from digestion after incineration of the TCNNs sample.

The sample should be digested according to one of the procedures of digestion or microwave-assisted digestion agreed between seller and buyer. The goal of digestion is to completely decompose the solid matrix of TCNNs to transfer the nanomaterials into the solution for the further determination step. The choice of the digestion method depends on the instrument availability and agreement between the concerned parties.

The sample can contain various types of nano-materials for specific purposes. Therefore, a certain nano-material can be responsible for superhydrophobicity of TCNNs and the other nano-materials exist for other purposes. In this case, the nano-material for superhydrophobic properties should be determined by the manufacturer.

4.3.5.7 X-ray fluorescence analysis

XRF analysis can identify the type and content of elements in a nano-object incorporated into or coated on textile fibres.

XRF analysis should be used for a quantitative determination of major and trace element concentrations in homogeneous powder using a calibration with standard sample of same matrix (see ISO 18227).

NOTE The powder for XRF test can be taken from ash after incineration of the TCNNs sample.

4.3.5.8 Energy dispersive X-ray analysis

Energy dispersive X-ray analysis can make quantitative measurement and identify the type of the elements in a nano-object incorporated into or coated on textile fibres (see ISO 22309).

NOTE The powder for EDX test can be taken from ash after incineration of the TCNNs sample

4.3.5.9 Inductively coupled plasma

ICP/AES and ICP/MS (see ISO 17294-1) can measure the type and content of elements in a nano-object incorporated into or coated on textile fibres.

NOTE 1 A solution test specimen is prepared using an appropriate solvent for the ICP technique.

NOTE 2 The powder for ICP test can be taken from ash after incineration of the TCNNs sample

4.4 Superhydrophobicity

4.4.1 General

Superhydrophobicity of TCNNs can be possibly changed being subjected to ageing process. Therefore, superhydrophobic durability of fabrics shall be evaluated and reported before and after ageing process.

Contact angle, contact angle hysteresis shall be measured for superhydrophobic evaluation for the samples for which such measurement be applied.

NOTE In some exceptional cases, the measurement conditions for the surface texture of woven/nonwoven fabrics are not met.

4.4.2 Contact angle

Angle between a plane solid surface and the tangent drawn in the vertical plane at the interface between the plane solid surface (textile) and the surface of a liquid (water) resting on the surface can be evaluated according to ISO 15989.

A 5 µl water droplet was dropped to the five different location of fabric surface and average value is considered.

4.4.3 Dynamic contact angle

A dynamic contact angle measurement refers to techniques that measure the contact angle during movement. This is usually accomplished by adding liquid to a static droplet on a surface and thus pushing the front of the liquid across the unwetted surface. ISO 19403-6 specifies a method to measure the dynamic contact angle with an optical method. The advancing and the receding angles are determined. The contact angle is calculated from the forces of interaction, sample geometry and liquid surface tension. Both the advancing and receding angles can be measured. The difference is the contact angle hysteresis.

4.5 Superhydrophobic durability assessment

4.5.1 General

It is supposed that textile need to be washed and ironed 40 times, and dry cleaned 20 times in its lifetime. In addition, during utilization, the textile exposes to light radiation and mechanical abrasion (rubbing) which can decline the superhydrophobicity of TCNNs.

To evaluate superhydrophobic durability of TCNNs during service lifetime, both

a) grading level of superhydrophobicity and

b) index of durability performance approaches

shall be considered. According to grading and IDP-value the superhydrophobic durability can be assessed.

4.5.2 Grade of superhydrophobic durability

4.5.2.1 General

The grade of superhydrophobic durability (GSD) is defined as the resistance against losing superhydrophobicity during certain degree of ageing process. The GSD is defined as the grade in which the superhydrophobic textile does not cross the border of superhydrophobic limit. In Tables 3 to 6, the grading of superhydrophobic durability is summarized. The grading is according to the assumption that the textile undergoes 40 times washing, ironing and 20 times dry cleaning during its service time. In addition, it is supposed that the TCNNs subjected once to mechanical abrasion and light exposure according to ISO 105-X12 and ISO 105-A02, respectively.

Superhydrophobic durability in TCNNs should be in harmony with other performances of the fabrics such as colour stability, mechanical properties, etc.

NOTE Due to the different applications of TCNNs, therefore, it does not mean that a lower grade (below grade A) is not appropriate for that application. For instance, a grade C for washing and dry cleaning can be sufficient for the jeans.

4.5.2.2 Washing and dry cleaning

To assess the durability of superhydrophobic fabrics against laundering, they shall be subjected to several washing cycles. Superhydrophobic textiles may be graded according to their response to washing cycles, as shown in Table 3.

Table 3 — Superhydrophobic textile's level as a function of washing cycles

Level	In terms of washing	In terms of dry cleaning
Grade A	If $\theta \geq 150°$ and contact angle hysteresis less than 10° after above 40 times home washing	If $\theta \geq 150°$ and contact angle hysteresis less than 10° after above 20 times dry cleaning
Grade B	If $\theta \geq 150°$ and contact angle hysteresis less than 10° after above 20 times home washing up to 40 times home washing	If $\theta \geq 150°$ and contact angle hysteresis less than 10° after above 15 times dry cleaning and up to 20 times dry cleaning
Grade C	If $\theta \geq 150°$ and contact angle hysteresis less than 10° after above 10 times home washing up to 20 times home washing	If $\theta \geq 150°$ and contact angle hysteresis less than 10° after above 10 times dry cleaning and up to 15 times dry cleaning
Grade D	If $\theta \geq 150°$ and contact angle hysteresis less than 10° after above 5 times home washing up to 10 times home washing.	If $\theta \geq 150°$ and contact angle hysteresis less than 10° after above 5 times dry cleaning and up to 10 times dry cleaning
Grade E	If $\theta \geq 150°$ and contact angle hysteresis less than 10° after up to 5 times home washing.	If $\theta \geq 150°$ and contact angle hysteresis less than 10° after up to 5 times dry cleaning

4.5.2.3 Ironing

To assess the durability of superhydrophobic fabric against ironing, they were subjected to several ironing cycles. Superhydrophobic textiles may be graded according to their response to ironing cycles, as shown in Table 4.

Table 4 — Superhydrophobic textile's level as a function of ironing

Level	In terms of ironing
Grade A	If $\theta \geq 150°$ and contact angle hysteresis less than 10° after above 40 times ironing
Grade B	If $\theta \geq 150°$ and contact angle hysteresis less than 10° after above 20 times ironing up to 40 times ironing
Grade C	If $\theta \geq 150°$ and contact angle hysteresis less than 10° after above 10 times ironing up to 20 times ironing
Grade D	If $\theta \geq 150°$ and contact angle hysteresis less than 10° after above 5 times ironing up to 10 times ironing
Grade E	If $\theta \geq 150°$ and contact angle hysteresis less than 10° after up to 5 times ironing

NOTE For the case of winkle free fabrics, there is no need for assessment of superhydrophobic durability of fabrics against ironing.

4.5.2.4 Mechanical abrasion (rubbing)

To assess the durability of superhydrophobic fabric against rubbing, they were subjected to rubbing cycles according to ISO 105-X12. Superhydrophobic textiles may be regarded as has having passed or failed mechanical abrasion testing according to the criteria shown in Table 5.

Table 5 — Superhydrophobic textile's level according to ISO 105-X12

Level	In terms of rubbing
Pass	If $\theta \geq 150°$ and contact angle hysteresis less than 10° after 40 times rubbing (20 times to and 20 times fro)
Fail	If $\theta \leq 150°$ and contact angle hysteresis less than 10° after 40 times rubbing (20 times to and 20 times fro)

4.5.2.5 Light exposure

To assess the durability of superhydrophobic fabric against light exposure, they were subjected to light exposure cycles. Superhydrophobic textiles may be graded according to their response to light exposure, as shown in Table 6.

Table 6 — Superhydrophobic textile's level as a function of light exposure time

Level	In terms of light exposure
Grade A	If $\theta \geq 150°$ and contact angle hysteresis less than 10° after continuing light exposure until contrast between the exposed and unexposed portions of the test specimen is equal to grey scale grade 1 (see ISO 105-A02)
Grade B	If $\theta \geq 150°$ and contact angle hysteresis less than 10° after continuing light exposure until contrast between the exposed and unexposed portions of the test specimen is equal to grey scale grade 2 (see ISO 105-A02)
Grade C	If $\theta \geq 150°$ and contact angle hysteresis less than 10° after continuing light exposure until contrast between the exposed and unexposed portions of the test specimen is equal to grey scale grade 3 (see ISO 105-A02)
Grade D	If $\theta \geq 150°$ and contact angle hysteresis less than 10° after continuing light exposure until contrast between the exposed and unexposed portions of the test specimen is equal to grey scale grade 4 (see ISO 105-A02)
Grade E	If $\theta \leq 150°$ and contact angle hysteresis less than 10° after continuing light exposure until contrast between the exposed and unexposed portions of the test specimen is equal to grey scale grade 4 (see ISO 105-A02)

4.5.3 Index of durability performance

Index of durability performance (IDP) of superhydrophobic textile is defined as:

a) for the case of contact angle, η_{ageing}:

$$\eta_{\text{ageing}} = (\theta_{\text{i}} - \theta_N)/\theta_{\text{i}}$$

where

θ_{i} is the initial contact angle;

θ_{N} is the contact angle after N times ageing;

b) for the case of contact angle hysteresis, $\acute{\eta}_{\text{ageing}}$:

$$\acute{\eta}_{\text{ageing}} = (\varphi_{\text{i}} - \varphi_N)/\ \varphi_{\text{i}}$$

where

φ_{i} is the initial contact angle hysteresis;

φ_{N} is the contact angle hysteresis after N times ageing.

In contrast to the grading approach (see 4.5.2), the IDP approach is defined as a degree of decline below the border of superhydrophobic limit. The IDP value is a number between 0 and 1.

Table 7 shows the *N* values for the IDP measurement.

Table 7 — *N* values for the IDP measurement of TCNNs

In terms of	*N* value(s)
Washing and dry cleaning	40, 20, 10 and 5
Ironing	40, 20, 10 and 5
Mechanical abrasion	20
Light exposure	grade 1, grade 2, grade 3 and grade 4 of light exposure
NOTE The *N* value(s) can also be determined between seller and buyer.	
For the case of light exposure instead of N value, the grade of light exposure shall be determined.	

5 Reporting

5.1 General

5.1.1 Introduction

The manufacturer or provider shall report the following general information and measurement results of the mandatory characteristics of a TCNNs listed in Table 1. The recommended characteristics can also be reported in a similar manner.

5.1.2 General information

— Manufacturer's name;

— Product name;

— Batch and lot number;

— Type of TCNNs;

— Base of matrix material;

— Type and name of nano-objects;

— Manufacturing method of TCNNs (in case of nano-structure and nano-fibre).

5.1.3 Measurement results

5.1.3.1 Essential characteristics

— Roughness/ nano-roughness;

— Chemical composition of nano-objects;

— Morphology;

— Contact angle before and after ageing of TCNNs;

— Contact angle hysteresis before and after ageing of TCNNs.

5.1.3.2 Additional information

- Measuring organization; laboratory name and address;
- Measurement date.

5.2 Table format example for reporting

Table 8 is an example of the table format for reporting. The optional characteristics may be added to the reporting items and to Table 8.

Table 8 — Table format example for reporting

General		
Manufacturer's name:		
Product name:		
Batch no.:		
Lot no.:		
Method of making hydrophobicity:	( ) nanocomposite ( ) nanofinished (nano-coating / plasma) ( ) nanofibre	
Type of ageing before characteristic and measurement:	( ) washing ( ) dry cleaning ( ) ironing ( ) abrasion ( ) light exposure	
Characteristics and measurements		**Measurement date**
Type and name:		
Nano-roughness:		
Chemical composition:		
Nanomaterial type and content:		
Level of Superhydrophobicity durability:	( ) washing [grades: A to E]	
	( ) dry cleaning [grades: A to E]	
	( ) ironing [grades: A to E]	
	( ) abrasion [grades: Pass and Fail]	
	( ) light exposure [grades: A to E]	
Index of durability performance (IDP):	η_{ageing} $\acute{\eta}_{ageing}$	....
List of laboratory names and addresses:		

Annex A
(informative)

Safety, health and environmental issues

Extensive use of TCNNs for their functional properties raises concerns about health, safety and environment as such products are in contact with human body skin and thus may get absorbed and/or inhaled and get into the blood stream. In addition, washing TCNNs may release nanomaterials into the waste water and subsequently find their way into water sources.

Therefore, the user of this document is advised to refer to the relevant published standards and other publications about health, safety/occupational risk and environment. Table A.1 summarizes some publications on safety, health and environment which can be useful for the case of TCNNs.