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Nanotechnologies — Characterization of individualized cellulose nanofibril samples

Nanotechnologies — Caractérisation d'échantillons de nanofibrilles individualisées de cellulose

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

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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 of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

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

Cellulose nanomaterials derived from naturally occurring cellulosic fibres are renewable advanced materials with unprecedented properties. They are of wide variety in morphology, e.g. different shapes, branching and networking. Basic research related to cellulosic nanomaterials has been increasingly conducted worldwide. At the same time, manufacturing industries have already started to deliver cellulose nanomaterials to the market. Application industries are also becoming more and more interested in these new materials.

All native cellulosic fibres are composed of bundles in which the smallest fibril unit is an elementary fibril originating from a cellulose terminal enzyme complex. An elementary fibril is made of a certain number of cellulose molecules and contains crystalline regions predominantly. The size of an elementary fibril is specific to the native cellulose source. In wood pulp, the cross-sectional dimension of an elementary fibril is about 3 nm and its aspect ratio can reach more than 200. In native cellulose fibres, elementary fibrils do not exist as single fibrils but adhere to each other through hydrogen bonding and are densely packed to form a bundle of fibrils. Very recently, however, some novel methods to extract and separate these elementary fibrils, through chemical modification of the outer surface of the fibrils followed by mechanical treatment, were developed. The chemical modification methods include TEMPO-mediated oxidation and phosphorylation. Using the above treatments, each native elementary fibril can be converted to an individualized cellulose nanofibril (iCNF) with charges at its surface. An iCNF has the functional groups on the outer surface of the fibril, and iCNFs can be separated from each other, one by one, by the static repulsion due to the electrostatic charge of newly introduced functional groups. Refer to Annex B for more explanations on iCNFs.

Several manufacturing companies have already begun producing iCNFs. iCNFs are now delivered increasingly to the worldwide market for applications in the industrial fields of polymer composites, adhesives, additives, gels, etc. Some examples of iCNF-containing commercial products are diapers with deodorant performance and gel ink for ballpoint pens. In all applications, appropriate characterization of the iCNF samples is necessary so that desired products can be manufactured.

This document provides a sound basis for the commercialization as well as the research and development of iCNF materials.

Nanotechnologies — Characterization of individualized cellulose nanofibril samples

1 Scope

This document specifies characteristics to be measured of individualized cellulose nanofibril (iCNF) samples in suspension and powder forms and their measurement methods. In addition, it provides sample preparation, measurement and data analysis procedures.

This document does not apply to the characterization of iCNFs that have been modified after they are manufactured.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO/TS 80004-2, *Nanotechnologies — Vocabulary — Part 2: Nano-objects*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO/TS 80004-2 and the following 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/

3.1
elementary fibril
structure, originating from a single terminal enzyme complex, having a configuration of cellulose chains specific to each cellulose-producing plant, animal, algal and bacteria species

[SOURCE: ISO/TS 20477:2017, 3.2.5]

3.2
cellulose nanofibril
CNF
cellulose nanofibre composed of at least one *elementary fibril* (3.1), containing crystalline, paracrystalline and amorphous regions, with aspect ratio usually greater than 10, which may contain longitudinal splits, entanglement between particles, or network-like structures

[SOURCE: ISO/TS 20477:2017, 3.3.6, modified — The notes to entry have been deleted.]

3.3
individualized cellulose nanofibril
iCNF
discrete *cellulose nanofibril* (3.2) composed of one *elementary fibril* (3.1) with ionic functional groups on its surface