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Nanotechnologies — Particle size distribution for cellulose nanocrystals

Nanotechnologies — Distribution en taille des particules pour les nanocristaux 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 and www.iec.ch/national-committees.

Introduction

Cellulose nanomaterials, including cellulose nanocrystals (CNCs) and cellulose nanofibrils, are anticipated to have significant commercial impact. Cellulose nanocrystals are produced from naturally occurring cellulose, primarily from wood pulps and annual plants, by acid hydrolysis. Their production from readily available cellulose sources makes them a candidate for use as a potentially non-toxic, biodegradable and sustainable nanomaterial. The recent demonstration of the feasibility of large-scale CNC production and the availability of infrastructure for harvesting raw materials will facilitate their commercial development. CNCs and cellulose nanofibrils are produced in a number of countries on pilot, pre-commercial or commercial scales. Estimates of the market potential for cellulosic nanomaterials are as high as 35 million metric tons annually, depending on the predicted applications and the estimated market penetration[10],[11]. Standards for characterization of CNCs are required for material certification to facilitate sustained commercial and applications development.

Cellulose nanocrystals have high crystallinity and are nanorods with high aspect ratio, surface area and mechanical strength. They assemble to give a chiral nematic phase with unique optical properties and their surface chemistry can be modified to ensure colloidal stability in water and to facilitate dispersion in a variety of matrices. These properties, plus their biocompatibility, low cost and minimal toxicity, enable many potential applications. Industrial producers are working with receptor industries in various application areas, including nanocomposite materials, health and personal care products, paints, adhesives and thin films, rheology modifiers and optical films and devices. Standardization activities within ISO/TC 229 and ISO/TC 6 have focused on nomenclature and terminology, characterization methods in general and specific methods for determining surface functional groups, metal ion and dry ash content. Particle size distribution is also a key property for CNC characterization. Particle morphology and size distribution control some properties of individual CNCs and contribute in part to their organization in suspensions, dry films and matrices. These properties and chemical characteristics determine CNC colloidal stability, viscosity and self-assembly, as well as performance in applications (e.g. reinforcement of nanocomposites). Length distribution may also be used to differentiate among cellulose nanocrystal grades or products.

This document describes a method for reproducibly dispersing dry CNCs for preparation of microscopy samples, provides image acquisition protocols for atomic force and transmission electron microscopy and summarizes image analysis procedures for determining particle size distributions. The methods are compatible with analysis of CNCs as produced by several processes and can be extended to surface modified CNCs with adjustment of dispersion and sample deposition methods. The two microscopy methods provide complementary information, and both have been widely used for size analysis of CNCs.

Nanotechnologies — Particle size distribution for cellulose nanocrystals

1 Scope

This document describes methods for the measurement of particle size distributions for cellulose nanocrystals using atomic force microscopy and transmission electron microscopy. The document provides a protocol for the reproducible dispersion of the material using ultrasonication, as assessed using dynamic light scattering. Sample preparation for microscopy, image acquisition and data analysis are included.

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*

ISO 21363:2020, *Nanotechnologies — Measurements of particle size and shape distributions by transmission electron microscopy*

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
cellulose nanocrystal
nanocrystal predominantly composed of cellulose with at least one *elementary fibril* (3.3), containing predominantly crystalline and paracrystalline regions, with an aspect ratio of usually less than 50 but usually greater than 5, not exhibiting longitudinal splits, inter-particle entanglement, or network-like structures

Note 1 to entry: The dimensions are typically 3 nm to 50 nm in cross-section and 100 nm to several µm in length depending on the source of the cellulose nanocrystal.

Note 2 to entry: The aspect ratio refers to the ratio of the longest to the shortest dimension.

Note 3 to entry: Historically cellulose nanocrystals have been called nanocrystalline cellulose (NCC), whiskers such as cellulose nanowhiskers (CNW), and microfibrils such as cellulose microfibrils; they have also been called spheres, needles or nanowires based on their shape, dimensions and morphology; other names have included cellulose micelles, cellulose crystallites and cellulose microcrystals.

[SOURCE: ISO/TS 20477:2017]

