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Nanotechnologies — Characterization of cellulose nanocrystals

Nanotechnologies — Caractérisation des nanocristaux de cellulose



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Foreword

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The committee responsible for this document is ISO/TC 229, *Nanotechnologies*.

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Introduction

Cellulose nanomaterials, including cellulose nanocrystals (CNCs) and cellulose nanofibrils, are anticipated to have significant commercial impact. Cellulose nanocrystals are extracted from naturally occurring cellulose, primarily from wood and annual plants, by acid hydrolysis, or chemical or enzymatic oxidation.[1][2][3] Their production from cellulose sources, such as wood pulps makes them a candidate for use as a potentially non-toxic, biodegradable and sustainable nanomaterial. Furthermore, the recent demonstration of the feasibility of CNC production on a large scale 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.[4][5] Standards for characterization of CNCs are required for material certification to allow sustained commercial development and applications.

Cellulose nanocrystals are nanorods that have high aspect ratio, surface area and mechanical strength and assemble to give a chiral nematic phase with unique optical properties. They are smaller than cellulose nanofibrils and have a higher crystalline content. These properties, plus the ability to control CNC surface charge and chemistry for dispersion in a variety of matrices, lead to potential applications in many areas including nanocomposite materials, paints and adhesives, optical films and devices, rheology modifiers, catalysts and biomedical products. There are currently no International Standards for this emerging commercial nanomaterial, although an ISO/TC 229 project on terminology is in progress, a Canadian National Standard (CSA Z5100) was published in 2014 and two CNC reference materials were released in 2013. This Technical Report reviews information on sample preparation, data collection and data analysis/interpretation for the measurands that are predicted to be important for the development of commercial products containing CNCs. Information for the following CNC properties is included: composition (crystallinity, surface functional groups, degree of polymerization and contaminants), morphology as assessed by microscopy and light scattering methods, surface charge and specific surface area, viscosity and thermal stability. The Technical Report reviews various approaches that have been used for specific properties, but does not recommend standard methods or provide detailed information on the techniques. The coverage is restricted to CNCs as produced and does not extend to post-production modified CNCs or CNC-enhanced materials or products.

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Nanotechnologies — Characterization of cellulose nanocrystals

1 Scope

This Technical Report reviews commonly used methods for the characterization of cellulose nanocrystals (CNCs), including sample preparation, measurement methods and data analysis. Selected measurands for characterization of CNCs for commercial production and applications are covered. These include CNC composition, morphology and surface characteristics.

2 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

2.1
agglomerate
collection of weakly or medium strongly bound particles where the resulting external surface area is similar to the sum of the surface areas of the individual components

Note 1 to entry: The forces holding an agglomerate together are weak forces, for example, van der Waals forces or simple physical entanglement.

Note 2 to entry: Agglomerates are also termed secondary particles and the original source particles are termed primary particles.

[SOURCE: ISO/TS 80004-2:2015, 3.3]

2.2
aggregate
particle comprising strongly bonded or fused particles where the resulting external surface area is significantly smaller than the sum of surface areas of the individual components

Note 1 to entry: The forces holding an aggregate together are strong forces, for example, covalent bonds, or those resulting from sintering or complex physical entanglement, or otherwise combined former primary particles.

Note 2 to entry: Aggregates are also termed secondary particles and the original source particles are termed primary particles.

[SOURCE: ISO/TS 80004-2:2015, 3.4]

2.3
nanocrystal
nano-object with a crystalline structure

[SOURCE: ISO/TS 80004-2:2015, 4.15]

2.4
nanofibre
nano-object with two external dimensions in the nanoscale and the third dimension significantly larger

Note 1 to entry: The largest external dimension is not necessarily in the nanoscale.

Note 2 to entry: The terms nanofibril and nanofilament can also be used.

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2.5
nano-object
discrete piece of material with one, two or three external dimensions in the nanoscale

Note 1 to entry: The second and third external dimensions are orthogonal to the first dimension and to each other.

[SOURCE: ISO/TS 80004-2:2015, 2.2]

2.6
nanorod
solid nanofibre

[SOURCE: ISO/TS 80004-2:2015, 4.7]

2.7
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 might be implied by the absence of a lower limit.

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

3 Symbols and abbreviated terms

For the purposes of this document, the following symbols and abbreviated terms apply.

AEC	anion-exchange chromatography
AFM	atomic force microscopy
BET	Brunauer-Emmett-Teller (method for determination of specific surface area)
CrI	crystallinity index (also CI)
CNC(s)	cellulose nanocrystal(s)
CP-MAS	cross polarization magic angle spinning
d_h	hydrodynamic diameter
DP	degree of polymerization
D_t	translational diffusion coefficient
DSC	differential scanning calorimetry
DLS	dynamic light scattering
ε	dielectric constant
EM	electron microscopy
FE-SEM	field emission-scanning electron microscopy
FTIR	Fourier transform infrared spectroscopy

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