



Nanoparticle Populations

Technical Work Area 34

Project 12

Determination of Particle Size Distribution for Cellulose Nanocrystals (CNCs)

Objectives

This interlaboratory comparison (ILC) will validate the performance of image acquisition protocols and image analysis methods for determining CNC particle size distributions by atomic force microscopy (AFM) and transmission electron microscopy (TEM). These methods are complementary in that AFM provides height and length measurements while TEM provides length and width measurements; all 3 dimensions are needed to assess CNC morphology. The ILC results will provide the pre-normative data for a technical specification on CNC particle size measurements.

Background and Standardisation needs

CNCs are one member of a family of emerging cellulose nanomaterials (CNM) with significant commercial potential. Realizing the potential of these materials requires methods to characterize a number of material properties, including particle morphology and size distribution. They control the properties of individual particles and their assemblies in suspensions, dry films and after incorporation in matrices, which are key factors for developing applications. The particle dimensions are also important for distinguishing between CNC grades and may provide information on the cellulose source and production method.

A series of CNM standards are currently being developed at ISO TC 229 (Nanotechnologies) and ISO TC 6 (Paper, Board and Pulp). These include terminology and nomenclature, a general overview of CNC characterization, determination of sulfur and sulfate half ester content for CNCs and dry matter and ash content for CNM. CNC particle size has been identified as a key priority in a survey of industrial producers of CNM (developed by ISO TC 6).

Work Programme

Phase 1 of the ILC will test image analysis methods using a single set of AFM and TEM images that are circulated to participants. The results will be used to optimize the analysis routines. The 2nd and 3rd phases will test the full image acquisition and analysis protocols. The protocols for image acquisition will be provided in advance to allow time for participants to seek clarification of the procedures with the project leader to modify the protocol.

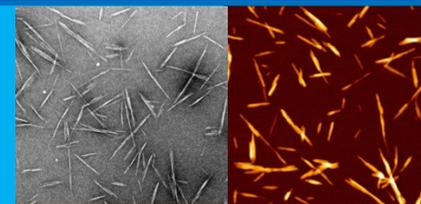
Pre-deposited samples of CNCs on mica or TEM grids will be supplied to participants with requests to image within a specified time frame. The CNC will be sourced from an NRC certified reference material, [CNCD-1](#), that will be dispersed using a standard sonication protocol to give a 2%

CALL FOR PARTICIPATION

Measurement Techniques

Atomic Force Microscopy (AFM)

Transmission Electron Microscopy (TEM)



by mass aqueous suspension that is diluted for preparation of microscopy samples. Duplicate sample slides (for each method) will be supplied to each participant.

Participants will be asked to image one of the two substrates (the second as a backup in case of any issues) and to obtain a sufficient number of images to measure the specified parameters (AFM, length and height; TEM, width and length) for the minimum number of particles specified in the protocol. Participants will be requested to provide details on the most recent microscope calibration. Excel templates to record all necessary data and calculations will be provided. Standard statistical methods will be used for further analysis of data sets returned by the participating laboratories. Participants must give permission for their data to be used in the final report and in publications or presentations arising from the ILC.

Deliverables and Dissemination

The ILC will validate the protocols for measuring particle size distributions for CNCs using AFM and TEM. The results of the study will be published in a peer reviewed journal and disseminated at relevant conferences. The ILC protocols and results will be used to determine uncertainty estimates, to develop ISO TS 23151¹ and to provide pre-normative validation data for this Technical Specification.

Participation / Funding

Participants with expertise in AFM and TEM will be recruited, aiming at a mix of participants with general expertise for imaging nanomaterials and with specific expertise in cellulose nanomaterials. Participation is funded by in-kind contributions from the participants.

Status

The project will start in January 2019. Samples will be provided in Feb/March 2019. Results should be reported within a month of receiving the samples.

Reference

1. ISO TS 23151 - Particle Size Distributions for Cellulose Nanocrystals .

For more information :

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December 2018