VAMAS

Nanoparticle Populations

Technical Work Area 34

Project 14

Crystallinity of Cellulose Nanomaterials by Powder X-ray Diffraction and Rietveld Modelling

Objectives

This interlaboratory comparison (ILC) will validate the performance of protocols for measuring the crystallinity of cellulose nanomaterials (CNM) using powder X-ray diffraction (PXRD) with Rietveld modelling. The ILC results will provide the pre-normative data for an ISO Technical Specification on crystallinity measurements.

Background and Standardisation needs

Cellulose nanomaterials are a family of emerging bioproducts with significant commercial potential. Realizing the potential of these materials requires methods to characterize a number of material properties, including crystallinity.

Crystallinity is an important indication of material quality, and is typically used to evaluate success of processing and potential material degradation, as processing can affect performance for various applications, particularly for nanocomposites. Crystallinity is also important for distinguishing between CNC grades and products and may provide information on the cellulose source and production method, as well as batch quality control and repeatability.

Powder X-ray diffraction (PXRD) is an easily accessible technique that can be run in most laboratories with the same level of proficiency and allows for routine and reproducible measurements for quality control purposes. This ILC will test precision and reproducibility of data collection and analysis methods for determining CNM crystallinity by PXRD using Rietveld analysis.

The results will be used to validate the proposed procedures for determining CNM crystallinity and to provide the pre-normative data necessary for development of a technical specification on CNM crystallinity measurements (ISO TC/229 TS 23361).

Work Programme

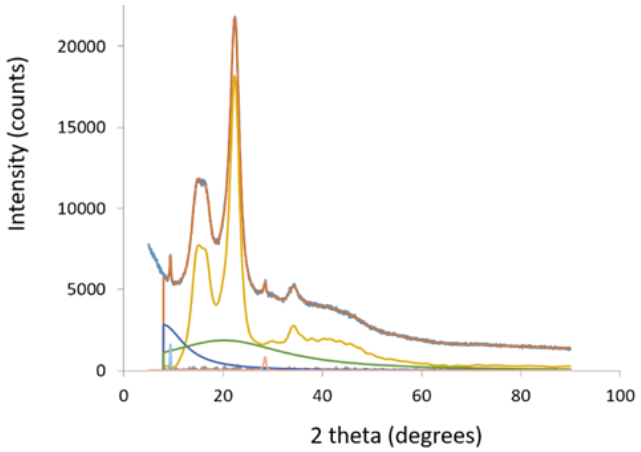
Phase 1 of the ILC will solicit participant input on the initial protocols for PXRD data collection and Rietveld modelling.

Phase 2 will test the modelling procedure by providing participants with several data sets for analysis, comparing the results obtained and optimizing the protocol as needed.

Phase 3 will consist of sending three CNM samples to each participant for PXRD data collection and Rietveld modelling. The three samples are cellulose nanocrystals, cellulose nanofibrils and individualized cellulose nanofibrils; each sample will be a dry powder ready for packing in a sample holder for PXRD.

Participants will return raw data and the results of Rietveld modelling to obtain the measured crystallinity.

CALL FOR PARTICIPATION



Diffraction pattern for cellulose nanofibrils and results of Rietveld modelling

Deliverables and Dissemination

The ILC will validate the protocols for measuring CNM crystallinity by PXRD with Rietveld modelling.

The ILC results will be used to determine uncertainty estimates and to provide the pre-normative validation data required for the ISO technical specification.

Results may also be published in a peer-reviewed journal and/or disseminated at relevant conferences.

Participation / Funding

Participants with expertise in PXRD will be recruited, aiming primarily at scientists with specific expertise in cellulose nanomaterials and those with prior experience with Rietveld modelling.

Participation is funded by in-kind contributions from the participants.

Status

The project will start in spring 2021. Samples will be sent to participants in summer 2021. Results should be reported within a month of receiving the samples and a final report should be completed by spring 2022.

For more information :

Dr. Wadood Hamad
Project Co-lead
FP Innovations, Canada
wadood.hamad@fpinnovations.ca

Dr. Linda Johnston
Project Co-lead
National Research Council Canada
linda.johnston@nrc-cnrc.gc.ca

Dr. Jeff Fagan
Chair, VAMAS TWA 34
NIST, USA
jeffrey.fagan@nist.gov

www.vamas.org

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