



Roadmap for the Development of International Standards for Nanocellulose

*A collaborative document prepared by an
international community of scientists and
professionals to chart the path forward in
developing international standards.*

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*Development of the Roadmap is administered
and coordinated through TAPPI's International
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1. EXECUTIVE SUMMARY

Nanocellulose is a class of renewable nanoscale materials from natural origins that has been the subject of increasing research and development (R&D) activities in many countries around the world. Current R&D in industry, academia and government laboratories suggests that it has a broad range of application potential in products and industries such as paper, building materials, polymer composites, medical, defense, electronics, automobile and aircrafts. By most accounts, nanocellulose-based products will be available in commercial quantities within five years.

On June 9, 2011, experts attending the Technical Association for the Pulp and Paper Industry (TAPPI) Workshop on International Standards for Nanocellulose in Arlington Virginia U.S.A. agreed that planning and coordination for nanocellulose international standards development should start immediately. In Terminology and Nomenclature, research groups around world have used their own terms to describe various types of nanocellulose and therefore there is a strong need to have a unified and universally accepted terminology followed by development of standard nomenclature. In Measurement and Characterization, experts at the Arlington workshop laid out a path forward by starting with identifying important nanocellulose properties and researching existing standards. The experts recommended organizing pre-normative tests with the most important properties and test methods to determine these properties before engaging and submitting standards-developing projects to standards-developing organizations. In Environment, Health and Safety (EHS), experts at the Arlington workshop examined potential exposure to three categories: workers, consumers and the environment. They recommended surveying existing industrial production and research laboratories on how these establishments address EHS issues as the first goal in nanocellulose EHS international standards development. Other important goals include assessing the state of development in toxicology testing, occupational safety evaluation, ecological assessment and exposure to substance in consumer products as a path to developing EHS international standards for nanocellulose. In Material Specifications, experts recommended the following path toward international standards development: first determine the properties that are required to describe nanocellulose for product specification and test methods to determine these properties; second, it is also important to differentiate between different types of nanocellulose and products; finally, assess the state of readiness to create international standards before submitting standards-development proposals to standards-developing organizations.

Coordination is of utmost importance in developing international standards for nanocellulose. For example, standards development in terminology and nomenclature should be coordinated with all other areas of standards development; standards development in Measurement and Characterization should be coordinated with EHS and Material Specifications. International standards development for nanocellulose should also be coordinated among international, regional, national and other standards developing organizations. However, due to its overarching nature, International Organization for Standardization (ISO) Technical Committee 229 (TC 229) Nanotechnologies should be the primary organization to submit standards-developing projects.

Key participants and contributors to this roadmap have formed the International Nanocellulose Standards Coordination Committee (INSCC). TAPPI in the U.S.A. will house the INSCC and provide the necessary support to execute this roadmap. Under the TAPPI organization, this roadmap will be updated periodically to reflect the advancement of nanocellulose science, R&D and product development.

2. INTRODUCTION

Nanotechnology holds enormous promise to revolutionize materials use in the 21st century and represents a major opportunity to generate new products among multiple industry sectors world-wide. The ability to determine and alter how materials are constructed at the nanoscale is providing the opportunity to develop new materials and products in unprecedented ways. As a result, nanotechnology has enormous promise to bring about fundamental changes and significant benefit to many industries around the world.

Nanocellulose, a cellulosic nanomaterial originating from flora and fauna, provide a key materials platform for the sustainable production of renewable, recyclable, and environmentally-preferable goods and products to meet the needs of people on a global scale. For example these materials will allow 1) the production of improved strength to weight performance materials and thus allow light-weight products to be produced without sacrificing end use performance 2) new generations of light-weight, hyper-performance nano-enabled structural and non-structural smart composites with unique multifunctional properties tailored to end-use requirements 3) unique optical, electrical and mechanical characteristics of nanocellulose will enable a new generation of high performance electro-optical materials and products.

As nanocellulose can be easily dispersed in a variety of media, the processing of such additives will be considerably more facile than other nanomaterials, particularly the spectrum of carbon nanotubes that tend to aggregate or agglomerate. The immediate challenge is to develop production facilities so that the various domains of possible application can be explored. Building on initial developmental research, the mid-term effort will most probably be focused on functionalized materials that will be tailored to the material media to be enhanced. The potential of nanocellulose in such applications is very large and will likely lead to large market potential. This will in turn emphasize the need for recognized international standards to facilitate the commercial transactions that will underpin the realization of that potential.

International standards have the potential to remove trade barriers, harmonize research and development activities, and contribute to development of regulations by national and international bodies. As government, academic and industrial research and development organizations endeavor to understand and develop nanocellulose-based products for multiple industrial sectors; the need for international standards in nanocellulose is necessary. Many international standards developing organizations have activities in nanotechnology, some for specific purposes, some for specific industrial sectors and others with an overarching scope such as International Organization for Standardization (ISO) Technical Committee 229 (TC 229) Nanotechnologies. This roadmap will lay out a path to develop a suite of international standards for nanocellulose and nanocellulose-based products in ISO TC 229 and in other standards developing organizations as appropriate.

For the purpose of this roadmap document, the term nanocellulose shall also include, besides cellulose nanocrystal (CNC) and cellulose nanofibril (CNF), cellulose microfibril (MFC) and cellulose microcrystal (CMC). Appropriateness of these terms and eventual terminology standards will be debated by experts in a formal standards-developing process.

This roadmap is a living document. The current version is written by a group of experts based on the best available science at the time the content of this document was debated. Since the science of nanocellulose is still in its early stages, this Roadmap for the Development of International Standards for

Nanocellulose will be updated as knowledge of nanocellulose advances in academia, industry and the policy arena.

2.1. Key Goals

The concept of a roadmap to chart the development of standards for cellulose nanomaterials was born at a one-day workshop held at the Sheraton Crystal City Hotel, Arlington, Virginia, U.S.A. on June 9, 2011. This workshop was hosted by the Technical Association of the Pulp and Paper Industry (TAPPI), and brought together 45 international experts to 1) determine priority areas for international standards development 2) discuss the state of science behind these priority areas and 3) discuss a path forward to effectively develop international standards for nanocellulose.

During the discussions and breakout sessions held at the workshop, it was very clear that increased engagement from a wide variety of communities, organizations, and industries is needed to successfully advance standards development. To engage individuals, garner support from organizations, and share progress with all interested parties, an overarching document was highly recommended to communicate effectively among the groups already involved, as well as to many others.

Consequently, several key goals of this roadmap document were developed:

- Highlight the key strategic and tactical pathways recommended to develop a suite of standards for nanocellulose.
- Generate awareness within industry, government and other sectors on the activities in standards development for nanocellulose materials.
- Encourage engagement from all professional fields to participate in nanocellulose standards development.
- Proactively address possible international trade barriers, regulation development and policy issues.

As described herein, there will need to be substantive communication among all groups working in nanocellulose materials, from those developing analytical laboratory methods to those designing new products incorporating nanocellulose materials.

2.2. The Importance of Standards for Commercial Development

Standards directly support the open, safe and responsible development and introduction of these nanomaterials into commerce and consumer products. To achieve the responsible and ethical commercialization of nanocellulose and nanocellulose-derived products in the global market place, it is critically important to develop internationally-accepted standards for the description and use of these products. Besides supporting commercialization, standards also serve to support market development, provide a basis for procurement materials' specifications and repeatability, and provide a basis for appropriate regulation.

Standards also allow sharing and better use of information for other reasons including consistency, interoperability, risk assessment, risk management and cost savings. Standards provide a broadly agreed upon framework for 1) naming, describing and specifying product materials; 2) measuring, testing and differentiating materials; and 3) creating standard reference materials that are certified as possessing specified characteristics traceable to a fundamental system of physical units of measurement and agreeing on specifications for commercial products. There are several types of standards. For

example, metrological standards deal with such things as length, mass, quantity, time, thermodynamic properties, conductivity, and luminosity. Other standards deal with such things as nomenclature, terminology, measuring methodologies and testing methodologies. Standards can be normative; defining what has been agreed upon nationally or internationally. Standards can also be informative, providing information only.

Standards that are formulated and validated broadly from a strong science base are most likely to be adopted by the global community. While individual nations have the right to develop their own national standards for nanocellulose nanomaterials, it is necessary to promote international collaboration for developing sets of widely-used and broadly supported international standards especially for industry sectors, such as forest products, where there is already a high degree of global marketplace integration.

2.3. Leveraging Existing Standards Committees for Nanomaterials

The International Organization for Standardization (ISO) already has a technical committee (TC 229, visit <http://www.iso.org/tc229> for more information) that is working to develop standards in the field of nanotechnologies that includes either or both of the following:

- Understanding and control of matter and processes at the nanoscale, typically, but not exclusively, below 100 nanometers in one or more dimensions where the onset of size-dependent phenomena usually enables novel applications.
- Utilizing the properties of nanoscale materials that differ from the properties of bulk matter, to create improved materials, devices and systems that exploit these new properties.

Specific ISO TC 229 tasks include developing standards for terminology and nomenclature; measurement and characterization, including specifications for reference materials; test methodologies; modeling and simulations; and science-based health, safety, and environmental practices.

Nanocellulose international standards development can take place in any one of the international standards organizations. However, since ISO TC 229 has an established overarching structure that can be overlaid onto many nanomaterials including nanocellulose, standards development activities for nanocellulose could align, when appropriate, with activities, structures, established standards and reports that already exist within the ISO TC 229 community. These documents will be extremely helpful in guiding work on standards for nanocellulose materials. Alignment with ISO TC 229 will also make it easier to submit standards development proposals to ISO TC 229 when appropriate.

3. CRITICAL SUCCESS FACTORS FOR STANDARDS DEVELOPMENT

The roadmap for development of standards for cellulose nanomaterials will be discussed via four main categories:

Terminology and Nomenclature
Measurement and Characterization
Environment, Health, and Safety (EHS)
Material Specifications

These categories align with the four Working Groups established within ISO TC 229. Significant activity within each of these working groups in ISO TC 229 is already underway for nanomaterials. Many of the activities highlighted within this roadmap leverage existing work by these communities.

3.1. Garnering Engagement from all Sectors

Inherent in each of the four standards development areas outlined in this document, is a critical need to reach out and engage interested groups from industry, academic, non-governmental and government institutions. For example, industry representation is essential for indispensable inputs on materials specification standards. Another example of this need is academic and government participation is essential for facilitating laboratory testing, methods development, and assessing reliability of emerging testing techniques. The TAPPI workshop held in June 2011 provided a good initial start in this process, but further development is needed, in particular more industry representation. The success of international standards development in nanocellulose will require a concerted effort from government, academia and industrial cooperators and participants.

3.2. Key Collaborative Intersections

As is evident in the discussions around each standard topic area, coordination and cooperation among the four identified areas are needed. There are several critical intersections of knowledge and activities that will be required: harmonizing of nomenclature to facilitate standards development in other areas; characterization method coordination among EHS, Material Specification and Measurement and Characterization groups.

Harmonized Nomenclature

Unified and universally accepted terminology and nomenclature are essential to successful communication among all groups working with nanocellulose materials. Unified and universally accepted terminology and nomenclature are also the essential step before developing international standards in other areas such as measurement and characterization, EHS and materials specifications. Many different terms are already being used widely in scientific and technical literature, and present a confusing picture to many. Some of these terms may be appropriate, but they need to be specifically defined for nanocellulose standards and accepted by the community as the correct terminology and nomenclature. Consequently, the establishment of terminology and nomenclature is an essential first step.

Nomenclature to differentiate the various nanocellulose materials with appropriate acronyms for cellulose nano-objects and cellulose nanostructured materials is needed. Terminology will also be needed to differentiate the branched fibers from individual particles, identify particle size, width (or diameter), length, aspect ratio, surface charge, degree of polymerization, and so on. The universally accepted vocabulary will be used by all groups developing standards.

Properties and Characterization Techniques for Nanocellulose

Another critical common element in standards development is the selection of properties and development of characterization methods for these properties. Particularly for nanomaterials, both EHS and material specifications may require development of characterization standards before commencing standards development. For example, toxicologists may need to understand the size, shape and aspect ratios of nanocellulose for toxicology tests. In a typical material specification, materials are described by relevant properties determined by acceptable test methods. Measurement and characterization, EHS

and material specification standards development experts will need to collaborate and coordinate their efforts in the standards development process.

4. STANDARDS FOR CELLULOSE NANOMATERIALS

4.1. Terminology and Nomenclature

Nanocellulose represents a new family of nanomaterials that appear to have very broad applications in a variety of materials related domains where physical characteristics such as strength, weight, rheology, optical properties and the like can be affected in a very positive manner. There is a strong need to have a unified and universally accepted terminology and nomenclature. While there is a particular urgency to develop terminology and nomenclature for plant-based nanocellulose, international standards for cellulose nanomaterials should also include nanocellulose from bacteria, algae, tunicates, and other sources.

The primary wood-derived nanocellulose materials are:

- Cellulose nano-objects: cellulose nanofibrils (CNFs) and cellulose nanocrystals (CNCs)
- Cellulose nanostructured materials: cellulose microfibrils (CMFs)

Scope

Terminology and nomenclature are needed to identify and differentiate the various types of nanocellulose materials. Acronyms are commonly used today by those active in the field, and also need to be standardized. For example: several commonly used today, some to describe the same object, terms include: cellulose nanomaterials (CN), cellulose nanocrystals (CNC), cellulose nanofibrils (CNF), nanocrystalline cellulose (NCC), and cellulose nanoparticles (CNP). Terminology will be also needed to identify and differentiate fibers versus particles, particle sizes, width (or diameter), length, aspect ratio, surface charge, degree of polymerization, crystallinity, sulfate groups, degree of sulfonation, and so on. Note that many already existing naming terms and descriptions in scientific and technical literature can be appropriate, but even those need to be specifically defined for international standards and the technical community.

Path Forward

A recommendation on the preliminary terminology framework for nanocellulose was presented at an initial workshop, held in Arlington Workshop, 9 June 2011. This framework was based upon existing ISO TC 229 nomenclature protocols, and included the following points (Figure 1):

- Use the terms cellulose nanocrystal (CNC), and cellulose nanofibril (CNF), cellulose microcrystal (CMC) and cellulose microfibril (CMF).
- CNC's and CNF's are cellulose nano-objects.
- Cellulose microfibrils and cellulose nanocomposites are cellulose nanostructured materials.

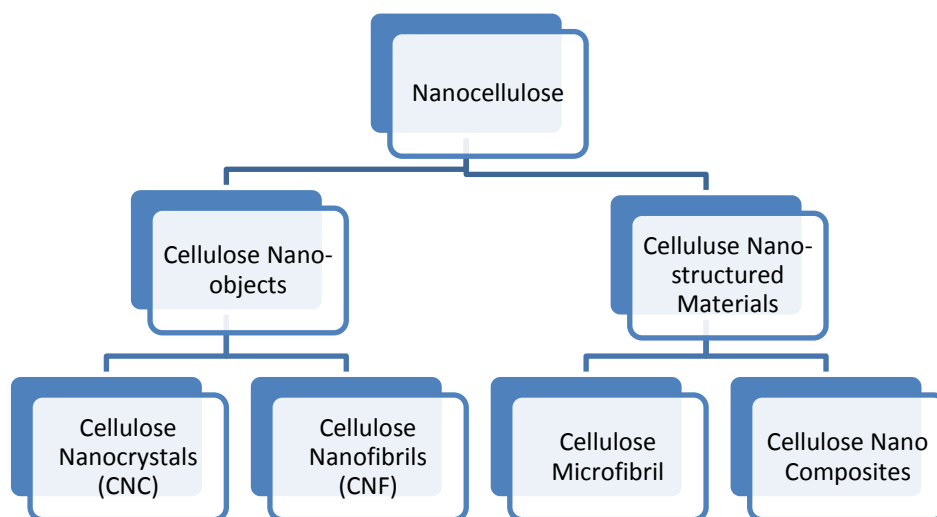


Figure 1. Naming Hierarchy for Nanocellulose

Goal 1: Expand Recommended Nanocellulose Hierarchy

Firstly, the preliminary terminology framework in Figure 1, developed at the Arlington Workshop, will be expanded to include definitions for the various terms identified, and deepened to include the next level down to involve terms for functionally substituted cellulose nanocrystals and for cellulose nanofibrils. In doing this, it will be clear how that next level down will be established for cellulose nanocomposites since these will, in general, be produced through functionalized cellulose nanocrystals and the matched medium such as polymers and ceramics. Also, to the extent that future development of microfibrils follows that of nanofibrils, the next level down would be a parallel set to that established for the nanofibrils. The remaining terminology domains related to coatings, films, ordered arrays, etc. will be developed later as core terms for these domains are developed within ISO TC 229 and as the applications of nanocellulose expands to include these products.

Goal 2: Develop Nomenclature Concurrently with Terminology

Secondly, careful consideration should be given to including nomenclature development for nanocellulose as part of the same work package as terminology. Functionalized nanocellulose will dominantly involve attaching well characterized chemical functional groups to the nanocellulose element – for example a urethane functional group for polyurethane matrices. Such functional groups already have well established nomenclatures and polymer matrices are already subject to fairly standard naming principles. This would allow the full nomenclature scenario to be built on a firm known base using a mixed terminological-chemical nomenclature approach.

Goal 3: Draft Standards and Submit to ISO TC 229

Based on the resulting discussions and decisions around terminology and nomenclature, a draft standard, as a seed document for further development, will be submitted to ISO TC 229.

4.2. Measurement and Characterization

Scope

Appropriate measurement and characterization standards are essential for efficient introduction of nanocellulose into the market place and for efficient commerce – the buying and selling - of nanocellulose. Standards must address industry needs (producers and users) as well as EHS regulations. Some of these basic characterization methods are expected to be done on a daily basis for characterization, testing and quality control purpose. Therefore, selected characterization methods need to be reliable, precise, validated and used in more than one laboratory.

Path Forward

The immediate goals are outlined below in proceeding to develop standards to characterize nanocellulose. For each of these goals, it will require close engagement of terminology with nomenclature standards to incorporate the agreed upon terminology and nomenclature as well as coordinating with the priorities of EHS and materials specification standards.

Goal 1: Identify and Rank Importance in Developing Measurement and Characterization Methods for Nanocellulose

Several parameters of cellulose nanocrystals and cellulose nanofibrils that need to be characterized have been identified and are listed in Tables 1 and 2, respectively. These are: particle size and distribution, shape, degree of branching, specific surface area, agglomeration, composition, surface charge, surface chemistry, crystallinity, gel point and viscosity, chirality, solubility and dispersibility, purity and contamination, and source of feedstock (i.e., wood, plant, bacterial, algae, tunicate, etc). Despite the extensive overlap, the characterization parameters for cellulose nanocrystals and cellulose nanofibrils (including microfibrils) are considered separately because significant differences in particle morphology, surface charge and surface chemistry will likely result in different measurement protocols. Additionally, there will likely be a different order of importance of parameter characterization to address industry needs. To move forward with standardization methods it would be beneficial to rank the order of importance of characterizing each property and the feasibility of the proposed characterization techniques.

Goal 2: Identify Existing Applicable Standards and Test Methods

There are various test methods for cellulose characterization internationally. There are other established test methods by, for example, American Society of Testing and Materials (ASTM) standard methods on characterization of polymers and suspensions. It will be necessary to understand which tests might be directly adopted or readily modified for use in characterizing nanocellulose. Additionally, for current characterization techniques that have been applied to nanocellulose, there are broad arrays of materials characterization issues, each of these will need to be assessed. Distribution of the written material on already existing and other metrology methods should be among the first steps.

Goal 3: ISO Technical Reports and Technical Specifications

There are broad arrays of materials characterization issues for nanocellulose, because of this it will be **extremely** challenging to develop standards for each. ISO Technical Reports (TR) and Technical Specifications (TS) provide an opportunity to publish characterization methods that do not pass the rigorous requirement for publication as an ISO standard. Both ISO TR and ISO TS could provide a useful mechanism for investigating and developing standardized measurement protocols for various testing methods of nanocellulose and the publication of the resulting protocols. A flexible process is

envisioned, so we can adapt our process and protocols for any subsequent testing and address the needs for our international community.

Task 1: Choice of Characterization Methods

To minimize work load, initially the most important or relevant characteristic(s) of the nanocellulose (cellulose nanocrystals, cellulose nanofibrils, cellulose microfibrils, etc) that address industry and/or EHS needs and regulations will be given higher priority for standard method development. These relevant characteristics will be based on industry input. Currently, these have NOT been identified but the rank in Tables 1 and 2 provide a general indication as to our current assessment of industry needs; additional input from industry is welcome at anytime and is highly encouraged.

Table 1. Cellulose nanocrystal properties and characterization techniques.

| Property | Method(s) | Existing Standards | Rank |
|---|---|---|------|
| Particle Morphology: <ul style="list-style-type: none"> • Size (Length, Width, Diameter, Aspect Ratio) • Distribution | <ul style="list-style-type: none"> • AFM, SEM, TEM (identify standard for – supporting substrate, loading & Staining approaches) • DLS, Laser scattering (can be used for relative distribution), www.nanosight.com • Light microscopy (with or without fractionation by centrifugation) • Morphological Analyzer (digital image analysis) | Identify useful standards from ASTM, TAPPI, ISO, PAPTAC, SCAN tests and others. | 1 |
| Surface Chemistry | <ul style="list-style-type: none"> • Functional Group – FT-IR, NMR, Raman spectroscopy • Inverse Gas Chromatography • Sulphur analysis by ICP • charge determination <ul style="list-style-type: none"> ○ Zeta potential (Coulter Delsa™) ○ Conductimetric titration | | 2 |
| Molecular Mass | <ul style="list-style-type: none"> • SEC-MALS • Viscosity • TGA | | 3 |
| Surface Area | <ul style="list-style-type: none"> • Congo red adsorption • BET surface area • N₂ gas adsorption (solvent exchange and freeze drying, super critical drying pretreatment) • Theoretical calculation based on particle morphology by electron microscopy | | 4 |
| Rheology: viscosity, gel point | <ul style="list-style-type: none"> • Low shear viscosity (modified Brookfield) • Drainage time | | 3 |
| Crystallinity <i>I-Alpha, I-Beta, II</i> | <ul style="list-style-type: none"> • WAXS • SAXS • Raman • IR • Solid state NMR • TGA | | 2 |
| Density | • Methods to be proposed in the future | | 4 |
| C, N, O, H | • LECO analyzer | | 2 |
| Explosion Potential (powder) | • Deflagration Index (K _{st}) | ASTM E1226-1 | 3 |

Table 2. Important properties of cellulose micro- and nanofibrils and suggested methods for their characterization.

| Property | Method(s) | Existing Standards | Rank |
|--|--|---|------|
| Particle Morphology: • Size (Length, Width, Diameter, Aspect Ratio) • Distribution • Degree of branching | <ul style="list-style-type: none"> • Transmittance spectroscopy (UV/VIS) • AFM/SEM/TEM • Light microscopy (with/without fractionation by centrifugation) • Viscosity (for aspect ratio) | Identify useful standards from ASTM, TAPPI, ISO, PAPTAC, SCAN tests and others. | 1 |
| Surface Chemistry | <ul style="list-style-type: none"> • Functional Group – FT-IR, NMR, Raman spectroscopy • Inverse Gas Chromatography • Charge Determination <ul style="list-style-type: none"> ○ Zeta potential (Coulter Delsa™) ○ Conductimetric titration | | 2 |
| Amount of Nanomaterials | <ul style="list-style-type: none"> • Fractionation by centrifugation • FFF | | 3 |
| Rheology : viscosity, gel point, | <ul style="list-style-type: none"> • Low shear viscosity (modified Brookfield) • Drainage time | | 3 |
| Dissolved (colloidal) Substance (amount and quality) | <ul style="list-style-type: none"> • CE • SEC • HPLC • AFM/SEM/TEM | | 3 |
| Crystallinity I-Alpha, I-Beta, II | <ul style="list-style-type: none"> • WAXS • SAXS • Raman • IR • Solid state NMR • TGA | | 2 |
| Specific Surface Area | <ul style="list-style-type: none"> • SAXS | | 4 |
| Explosion Potential (powder) | <ul style="list-style-type: none"> • Deflagration Index (K_{st}) | ASTM E1226-1 | 3 |

Task 2: Draft Measurement Protocols

For each relevant characterization method, a draft of the measurement protocols will be written up. This working document will be based on existing standards and test methods (from goal 2) and by input from experienced scientists and technicians (from industry, academia and government laboratories). The resulting set of measurement protocols will then be used as a draft standard to be used for all subsequent testing. Since this is a working document, it can be adapted as we learn more about the systematic testing of these materials. Also, it might be necessary to consider a system of characterization techniques to be used in combination to give greater insight as to a particular parameter and property. For example, particle morphology, it might be advantageous to use more than just combined microscope techniques (light microscopy, TEM, SEM, AFM), but also include information from dynamic light scattering, or viscosity measurements. The trick will be to

sort out the combination of techniques that results in the most efficient use of time and cost while giving the greatest fidelity in property measurement. Lastly, protocols on the use of fractionation (by centrifugation or other method) may also give additional clarity in property measurement.

Task 3: Conduct Feasibility Studies

For each relevant characterization method identified in Task 1, a feasibility study will be initiated using the draft standard protocols developed in Task 2. A “learning approach” will be used to ensure that proposed methods can be evaluated and modified through an iterative process, and then compared by a number of different laboratories to ensure validation. The term feasibility study is used here in relation to the overall goal of the development of an ISO and other standard(s). At the very least the information gained in these studies will be used in the development of measurement protocols that will be written up as ISO TR and ISO TS publications and will be accessible for industry, academia and governmental laboratories. A sample draft timeline for a given proposed feasibility study is given below. Note that it is likely that several feasibility studies will be running concurrently. The times given are based on an arbitrary start date.

- **1st Month:** identification of a lead laboratory to organize the working team
- **1st Month:** identify suppliers of the CNC and CNF samples suitable for feasibility testing
- **2nd Month:** identification of laboratories able and willing to participate in the evaluation
- **3rd Month:** identify characterization methods (see Task 1), and finalize 1st draft of specific measurement protocol to be followed by each laboratory of candidate test procedures (see Task2)
- **3rd Month:** identify the quantity of CNC and CNF material needed to be sent to each of the participating laboratories
- **3rd Month:** drafting and agreeing on a comparison timetable or schedule and a template for reporting measurement details (including particulars of instrumentation) and laboratory results (including statistical information where possible)
- **4th Month:** obtaining CNC and CNF starting materials, “confirming” particle size via TEM, SEM, AFM techniques, producing testing samples and circulating them to the labs
- **5th Month:** conducting the measurements, gathering the results from the individual participant laboratories
- **7th Month:** analyzing the collected measurement comparison data to understand the degree of equivalence among the participants (e.g., using standard deviation of results as a measure of agreement)
- **10th Month:** writing the comparison draft report and sharing with all participants
- **12th Month:** finalize the report (e.g., for presentation or publication at a TAPPI conference or workshop)

Task 4: Assess Readiness to Create ISO TR or ISO TS, or Start Pre-Normative Study

The finalized report for each feasibility study will be considered for the development of either an ISO TR or ISO TS. If the outcomes are sufficiently promising (high consistency in reported results from different labs, narrow spread in measured properties, etc), the specific characterization technique would then be considered for pre-normative study (see Goal 4).

Goal 4: Conduct Pre-Normative Study

Pre-normative studies will be initiated for specific characterization technique(s) demonstrated to have high consistency in the feasibility studies (from Task 4). Pre-normative studies will be much more rigorous as *there is a strong need for scientific evidence that the proposed methods have been validated and used in more than one laboratory*. The pre-normative study(s) needs to pass the 10 item check list

developed by the ISO TC 229 JWG2 Study Group on Metrology, first developed for Carbon Nanotubes (Table 3). Pre-normative work, such as inter-laboratory round-robin comparisons, must be conducted and completed prior to launching the normative process. A round-robin comparison study could be completed under the auspices of the Versailles Project on Advanced Materials and Standards (VAMAS) with assistance from scientists already connected with that community. This model approach is seen to be open to any and all interested participants, and would be conducted in a collaborative, pre-market and pre-competitive spirit. A simple timeline for the completion of each step of the proposed pre-normative could follow the feasibility time line given in Goal 3, Task 3.

Table 3. Outline of the Metrological Check-List Prepared by the Study Group on Metrology

1. Has the system / body / substance that will be subjected to the measurement procedure, been clearly described, including its state?
2. Is the definition of the system / body/ substance not unnecessarily restrictive?
3. Is the measurand clearly described?
4. Has it been clearly indicated whether the measurand is operationally or method-defined, or whether the measurand is an intrinsic, structurally defined property?
5. Is the measurement unit defined? Are the tools required to obtain metrological traceability available?
6. Has the method already been validated in one or more laboratories?
7. Are any quality-control tools available to enable the demonstration of a laboratory's proficiency with the test method?
8. Have the results of measurements using the proposed method already been published in peer-reviewed journals by several laboratories?
9. Is the instrumentation required to perform the test widely available?
10. Does the document propose a measurement uncertainty budget?

Source: Ichimura, "Current Activities of ISO TC 229/WG2 on Purity Evaluation and Quality Assurance Standards for Carbon Nanotubes," Anal. Bioanal. Chem., 396:963-971:2010.

Goal 5: Assess State of Readiness to Create TC 229 Work Item

The results of the pre-normative study will be assessed with the ISO TC 229 Metrology Check List (Table 3) to determine the characterization method's readiness for launching a standard development project. If the a characterization method is deemed to be ready for standards development, a proposal based on the method will be prepared and submitted to an international standards developing organization such as ISO TC 229 for international standards development.

4.3. Environment, Health and Safety

Scope

It is critical to the commercial success of nanocellulose to have methods to assess and manage the potential for exposure and risks to workers, consumers and the environment. There are a variety of standards for assessing substances used in industrial and consumer products, but they have not yet been validated for nanomaterials. Further, because of the potential for unique attributes of materials at the nanoscale, there is a need to characterize the physical and chemical properties of nanocellulose at various life cycle stages to inform toxicity studies.

Path Forward

There are several steps toward a strategy for developing EHS standards for nanocellulose. Three main components are needed: occupational, environmental, and consumer exposure test methods. Below, a process is proposed to identify the necessary test components, the options for measuring these components, a selection process for including components in the standard, and a protocol for testing how well the standards measure key endpoints, their reliability, and ease of implementation by the nanocellulose community (e.g. the type of equipment needed).

Goal 1: Outline the Components of the Standard

The purpose of establishing an EHS standard is to create a baseline of studies for evaluating specific materials. Currently, the suite of testing varies by market and application. As a starting point for developing the scope of the EHS standard, there is a need to assess how companies that are already producing commercial microcellulose products have been addressing EHS issues. In addition, it should also be assessed as to what has been achieved regarding EHS issues by the few research institutes that are already producing pilot-scale quantities of CNC and CNF. Additional components of nanosafety standard evaluations will also be included as possible components

Goal 2: Assess the State of Development of Material Characterization for Toxicity Testing

There are several proposed approaches for measuring key properties (e.g. ISO; ASTM; MINChar; OECD). These need to be considered in determining which properties are important for the nanocellulose EHS standard, and whether to simply adopt an existing standard, or if nanocellulose requires additional or subsets of proposed characterization. Further, analytical methods are needed for the properties. This effort must be consistent with the work of the characterization group, and will require collaboration.

Goal 3: Determine the Availability of Methods for Occupational Safety Evaluation and Their Applicability to Nanocellulose

The first step is to identify existing methods for assessing occupational exposures during manufacturing. Next, is to compile methods developed specifically for nanoscale materials, and assess their applicability to nanocellulose, as a carbonaceous material. The available methods will be compared with methods needed for assessing occupational exposure for nanomaterials generally, and specifically for nanocellulose.

Goal 4: Compile Existing Standards for Ecological Assessment of Substances, and Assess Their Potential Utility for Nanocellulose (OECD, EPA, others)

The first step is to identify existing methods for assessing ecological exposures during manufacturing and use. Next, is to compile methods developed specifically for nanoscale materials, and assess their applicability to nanocellulose, as a carbonaceous material. The available methods will be compared with methods needed for assessing environmental exposure for nanomaterials generally, and specifically for nanocellulose.

Goal 5: Develop a Compendium of Available Methods for Assessing Potential Exposure to Substances in Consumer Products as a Baseline to Assess Their Applicability to Nanocellulose

The first step is to identify existing methods for assessing consumer exposures to products. Next, is to compile methods developed specifically for nanoscale materials in products, and assess their applicability to nanocellulose. The available methods will be compared with methods needed for assessing consumer exposure to nanomaterials in products generally, and specifically for nanocellulose.

Goal 6: Determine Scope of Standard(s)

In this step, the group will assess the state of methods development in comparison with the baseline of testing needed to ensure the safety of materials, and establish the scope for initial EHS standards for nanocellulose. Additional testing needs and methods development may be required for a comprehensive standard, and it will be necessary to establish a realistic scope for standards development. In particular, as with other proposed nanocellulose methods, there is a need to assess the state of readiness of EHS methods to create an ISO TC 229 work item.

Goal 7: Draft Standards

Goal 8: Work to Include Nanocellulose in the OECD Round Robin Process

As a longer term goal, effort should be made to get nanocellulose in the OECD Round Robin Process. This international testing program is developing a body of data for several nanoscale materials on a breadth of endpoints, and includes validation across laboratories. It would be beneficial for nanocellulose to become part of the testing program.

4.4. Material Specifications

Scope

Material Specification Standards will be used for commercial purposes and to support future trade. In order to produce such standards, close collaboration and communication with the community working on Measurement and Characterization standards will be essential. Some of these methods used for classifying are expected to be done on a daily bases for quality control purposes. Selected characterization methods need to be reliable, precise, validated and used in more than one laboratory. Since application of nanocellulose is still in the R&D stage mostly by developers and future sellers, standard grades of nanocellulose has yet to emerge. Consequently, material specification standards at this stage may not be able to include any definitions on standardized grades of nanocellulose.

There is currently no commercial production of cellulose nano-objects or cellulose nano-structured materials; however, manufacturing facilities are planned. The current knowledge about these novel materials is limited, based on small or medium scale laboratory or pilot studies. In moving forward with material specifications, input from both producer and user industries will be needed for assessing current and future needs to characterize nanocellulose. Additional commitment from Industrial partners is needed in order to focus the work properly.

This includes selecting what parameter(s) needs to be measured in order to classify the material to different groups.

Path Forward

Goal 1: Determine the Sets of Measurement Methods Needed

A set of measurement methods will be needed to provide basic information required in the manufacturing and marketing of cellulose nanomaterials. These methods would provide material specifications for the customer, as well as serve as internal quality control standards. Standardized methods are needed especially in specifying material properties for the customer, because it should be possible to compare different manufacturers. It is also emphasized that in internal quality control such methods could be used and may not be reported to the customer. The measurement methods needed are different for different types of nanocellulose materials (CNC's of CNF's). Besides that CNC's and CNF's require somewhat different methods; within the group of CNF's chemically modified grades (i.e.

TEMPO-oxidized) and unmodified grades require, if not fully different methods, at least modified versions of the same methods.

Goal 2: Provide Methods to Distinguish Between Different Types of Nanocellulose Materials

Nanocellulose materials can be defined by their manufacturing methods. It is unclear what additional information the definition of the material by its characteristics could bring about. This may be avoided if proper terminology and nomenclature are used.

Material specification standards need to address current as well as future materials that industry will produce in large scale, and specifications should account for the characterization methods that may be applicable for measuring large quantities of the materials. These materials should be classified, for example, based on size, dimensions, branching, charge and mass (Figure 3). Effective commerce will depend on developing a classification scheme that is aligned with potential applications.

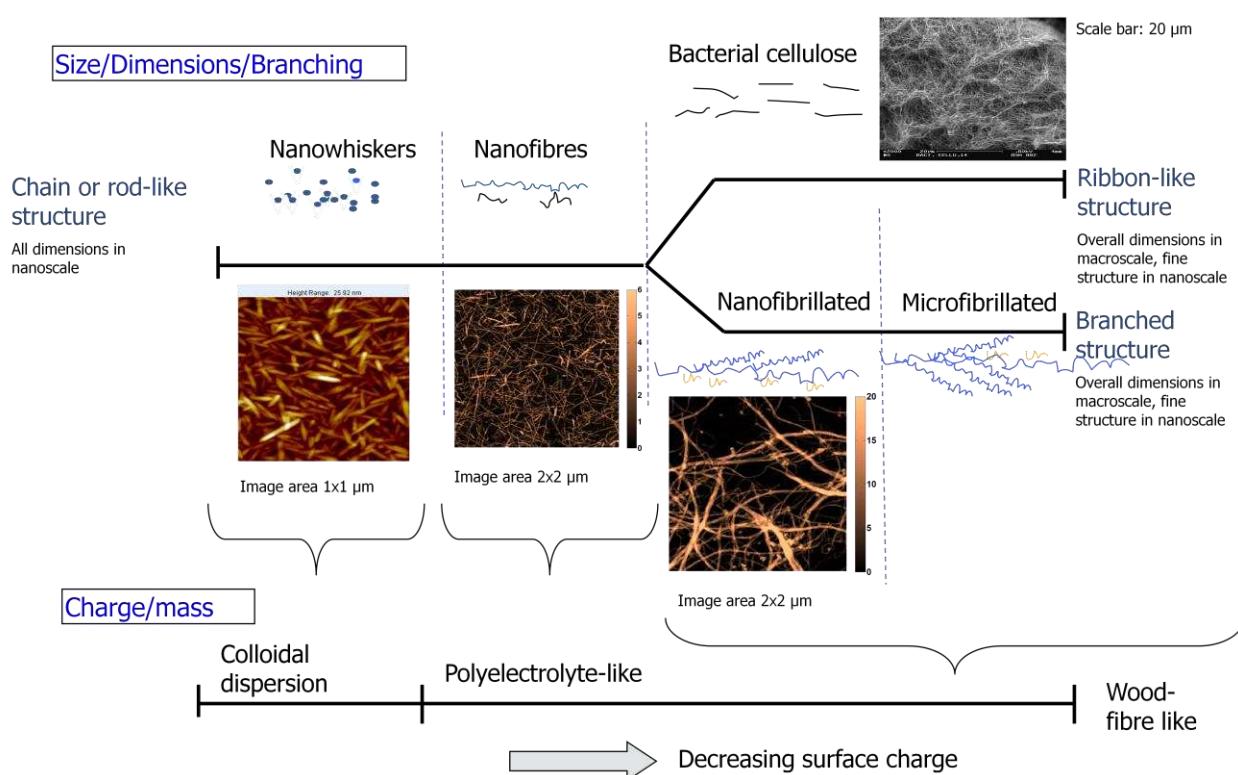


Figure 3. Classification proposed by A. Sneek et al., 2011.

Goal 3: Identify Methods for Early Product Differentiation

Material specification could include in the beginning the “fast” methods, mainly those for differentiating various products, that could also be used for quality assurance at production or application sites in order to support near-term commercialization. The possible properties for classification and quality control purposes for cellulose micro- and nanofibrils (CMF and CNF) are in Table 4. The most suitable properties for cellulose micro- and nanocrystals (CMC and CNC) are size and aspect ratio, degree of crystallinity, surface chemistry including nature of ions, surface charge and concentration of ions and/or functional groups, pH, source of feedstock (wood vs. bacterium for example), and chirality and nematic properties. Additional specific application-related characterization is not a priority at this stage. More methods in material specification should be discussed later and reconsidered when the final applications are in commercial use.

Table 4. Important properties of cellulose micro- and nanofibrils and suggested methods for material specification.

| Property | Characterization method | Apparatus |
|---------------------------------|---|-----------------------------------|
| Dry matter content | According to standard | Scales, oven |
| pH | pH measurement | pH electrode |
| Brightness | According to standard (sample preparation not standardized) | Elrepho |
| Size range | Light scattering | Nanosight, Beckman Coulter N5 etc |
| | Fiber analyser | FiberLab |
| Rheology | Low shear viscosity, yield stress | Brookfield (modified) |
| Turbidity | Transmittance | UV-VIS spectroscopy |
| | Transmittance with settling/sedimentation rate | Turbiscan Labexpert |
| Surface charge | Conductimetric titration | Metrohm Conductimeter |
| | Zeta-potential | Coulter Delsa |
| Chemical composition and purity | Chemical analysis | GC-MS AAS/AES IR UV-VIS |

Goal 4: Assess State of Readiness to Create ISO TC 229 Work Item

The ISO TC 229 Metrology Check List (Table 3) should be used as a guide to assessing the state of readiness for launching standard development projects. Based on the results of the pre-normative study conducted by the Measurement and Characterization Group, an assessment should be made to determine if it is appropriate to begin the very time consuming process of standard development. The Material Specification Group can also aim for a technical report only to define the classification.

5. KEY MILESTONES

| Activity | Milestone | Date |
|--|--|---|
| Terminology and Nomenclature | | |
| Expand recommended nanocellulose hierarchy | Draft copy of the expanded nanocellulose hierarchy | December 31, 2012 |
| Develop nomenclature concurrently with terminology | Draft nomenclature system | December 31, 2014 |
| Draft standards and submit to TC 229 | Submit new work item proposal to ISO TC 229 | June 30, 2012 |
| Measurement and Characterization | | |
| Identify and rank importance in developing measurement and characterization methods for nanocellulose materials | Create ranked list of measurement and characterization methods | November 30, 2011 |
| Identify existing applicable standards and test methods | Complete survey on existing standards and test methods | March 31, 2012 |
| Conduct feasibility study and submit proposal for ISO TR or ISO TS | Submit new TR or TS project proposal to ISO TC 229 | May 31, 2013 |
| Conduct pre-normative study | Complete pre-normative study | May 31, 2014 |
| Assess state of readiness for international standards development | Submit standards development project proposal to ISO TC 229 or other standards development organizations | June 30, 2014 |
| Environment, Health and Safety | | |
| Outline the components of the standard | Develop outline for EHS standards | March 31, 2012 |
| Assess the state of development of material characterization for toxicity testing | Complete assessment for toxicity testing | Three months after identification of test methods |
| Determine the availability of methods for occupational safety evaluation and their applicability to nanocellulose | Complete availability and assessment study for occupational safety evaluation and their applicability to nanocellulose | June 31, 2012 |
| Compile existing standards for ecological assessment of substances, and assess their potential utility for nanocellulose (OECD, EPA, others) | Create list of existing standards for ecological assessment and determine their utility for nanocellulose | September 30, 2012 |

| | | |
|--|--|--------------------|
| Development a compendium of available methods for assessing potential exposure to substances in consumer products as a baseline to assess their applicability to nanocellulose | Create list of available methods for assessing exposure to substance in consumer products and determine their applicability to nanocellulose | November 30, 2012 |
| Determine scope of standards(s) | Written scope statement | December 31, 2012 |
| Draft standards | Submit standards development proposal to ISO TC 229 or other standards development organizations | January 31, 2013 |
| Work to include nanocellulose in the OECD round robin process | Establish collaborative working relationship with OECD to include nanocellulose in OECD round robin tests | March 31, 2013 |
| Material Specifications | | |
| Determine the sets of measurement methods needed | Table of most relevant methods to be used by industrial partners for quality control of future nano objects (CNC, CNF) and nano structured materials (CMF) based on industrial experience. | September 30, 2012 |
| Provide methods to distinguish between different types of nanocellulose materials | Choose the most relevant method to differentiate different grades for general characterization use. | December 31, 2012 |
| Identify methods for early product differentiation | Choose relevant application related methods for different end-uses based on the industrial experience about the real end-uses. | December 31, 2013 |
| Assess state of readiness to create ISO TC 229 work item | Propose work item to prepare TR or TS to define the classification | December 31, 2013 |

6. COORDINATION IN NANOCELLULOSE INTERNATIONAL STANDARDS DEVELOPMENT

6.1. Coordination Areas and Needs

An essential part of the planned standards activity is coordination with the standards community. However, this coordination effort must not lose sight of the fact that standards are intended to facilitate commerce by improving the quality and accuracy of communications between seller and buyer. A second principle is to avoid duplication and conflict between the various kinds of standards organizations.

The International Organization for Standardization (ISO) is one of the international organizations comprised of national member bodies active in developing international standards. The key organizational features in ISO that debates and develops consensus based on science with regards to developing international standards are the technical committees (TC). ISO TC's are organized around specific topics and the technical committee for nanotechnologies is TC 229 which was organized in 2005. From the beginning, it has been the position of ISO TC 229 Chair, Dr. Peter Hatto, that ISO TC 229 could not alone handle all the possible standardization activities in nanotechnology. In particular, there has been a focused effort to establish liaisons with other ISO technical committees and international groups such as the Organization for Economic Co-Operation and Development (OECD), The European Committee for Standardization (CEN) and others.

There are also organizations with interest in developing national or regional standards. In Scandinavia for example, the Nordic Standardization Programme (NSP) is an initiative for the harmonization and standardization mainly of test methods and terminology for the pulp and paper industry in Finland, Norway and Sweden. NSP also looks after the pulp and paper industry's interests in the standardization activities within ISO TC 6 (Paper, Board and Pulp), CEN TC 172 (Pulp, Paper and Board) and the Scandinavian Pulp, Paper and Board Testing Committee SCAN-test. In Northern America, TAPPI in the U.S.A. and the Pulp and Paper Technical Association of Canada (PAPTAC) in Canada develop standard test methods for the pulp and paper industries in their respective country.

Examples of Standards Development Bodies for Nanocellulose (not an exhaustive list)

ISO TC 229 Nanotechnologies – ISO TC 229 is the ISO technical committee that focuses on developing ISO standards for nanotechnology. It features four working groups including terminology, measurement, EHS, and material specifications. The committee has in general focused on generic over-reaching subjects, although a number of standards have focused on more specific topics such as quantification of carbon nanotubes.

ISO TC 6 Paper, Board and Pulp – The Secretariat is held by Canada. The committee focuses on products.

ISO TC's other than TC 229 and TC 6 - ISO has 230 TC's and 8 other bodies developing standards and guides in various areas. Nanotechnology is also within the scope of many of these ISO standards making bodies.

OECD WPMN - To evaluate regulatory challenges with regards to safety in using nanomaterials, the OECD established a Working Party on Manufactured Nanomaterials (WPMN). This group focuses on testing and assessment methods in implications on the safety of human health and the environment associated with the use of nanomaterials.

CEN - CEN established CEN TC 352 'Nanotechnologies' at the end of 2005 to develop a set of standards addressing the following aspects of nanotechnologies: a) classification, terminology and nomenclature; b) metrology and instrumentation, including specifications for reference materials; c) test methodologies; d) modeling and simulation; e) science-based health, safety and environmental practices; and f) nanotechnology products and processes.

Examples of Areas of Coordination among Standards Developing Bodies

It follows that certain forms of standardization might be best accomplished in various organizations that have appropriate expertise in their committees. This may not preclude parallel activities but the efforts must be coordinated and harmonized, however, the nanocellulose community can best benefit from a coordinated effort in international standards development.

Examples of coordination under each of the four main standards areas include, but are certainly not limited to:

- **Terminology and Nomenclature**
 - Terminology needs to be closely coordinated with TC 229 JWG 1. The terms appropriate for nanocellulose need to fit into the TC 229 JWG1 hierarchies and nomenclature which is also coordinated with the International Union of Pure and Applied Chemistry (IUPAC) on chemical naming. Coordination with ISO TC 229 JWG 1 will ensure standard terminology for nanocellulose is harmonized with core terms in nanotechnology and terminology for other nanomaterials.
- **Measurement and Characterization**
 - Nanocellulose standards development in EHS and Materials Specification needs to be coordinated with Measurement and Characterization. For example, the EHS group may need to coordinate with the Measurement and Characterization group to develop measurement methods to characterize nanomaterials for toxicity studies. Similarly, the Material Specification group may need to coordinate with the Measurement and Characterization group to standardize measurement methods to describe material properties for setting standards in material specification.
 - Several organizations including ISO TC 229 JWG 2, OECD, Scandinavian Pulp, Paper and Board Testing Committee (SCAN), TAPPI, PAPTAC, the National Institute of Standards and Technology (NIST) in the U.S. and FPInnovations in Canada have experience with measurement of nanomaterials or measurement of cellulosic fibers. Collaboration among organizations with expertise in measurement and characterization of nanomaterials and cellulosic fibers can bring past experience in nanomaterials and cellulosic fiber together, eliminate duplication and shorten standards development time. A pre-normative round-robin comparison study could be completed under the

auspices of VAMAS with assistance from scientists already connected with that community.

- Environment, Health and Safety
 - The EHS group needs to collaborate with the Measurement and Characterization group on measurement methods.
 - The EHS questions related to nanocellulose are believed to be benign because the source is from plants and microorganisms. However, internationally accepted protocols need to be developed related to EHS to validate this assertion. Standardization may take two approaches: standalone documents focusing on nanocellulose or general nanosafety documents that include guidance for nanocellulose. International collaboration in EHS nanocellulose standards may include IC TC 229 WG 3, OECD, NIST, FPInnovations, government laboratories, occupational safety laboratories etc.
- Material Specification
 - The material specification group needs to collaborate with the Measurement and Characterization group on measurement methods for material specification.
 - Since material specification requires identifying and measuring nanocellulose properties for production control and description of a material for transaction between sellers and buyers, it is important that standards development bodies such as ISO TC 229 coordinate with nanocellulose producers, nanocellulose users, other ISO TC's (e.g. TC 6), industry technical standards organizations (TAPPI, PATAC, SCAN) and government standards and measurement laboratories (NIST).

Coordination with other international standards groups and groups with interest and expertise in nanocellulose should be considered as these groups and expertise are identified.

6.2. Management and Execution of Roadmap Activities

TAPPI, a technical professional organization, has worked with the technical community of experts in nanotechnology in the pulp and paper industry over the last two decades. On behalf of this community, TAPPI has hosted conferences, created a new division, and supported other activities to foster communication and development of nanotechnology.

TAPPI's Nanotechnology Division focuses on nanotechnology for renewable materials, including the advancement of research and development for cellulose nanomaterials. The International Nanotechnology Division will concentrate on developments for traditional forest-based products like pulp, paper and building materials, and also on applications such as coatings, plastics, digital displays, military body armor, and medical implants, among others. Comprised of producers, academia, government, consulting companies, suppliers and others in the industry, it serves as the leading global forum for knowledge sharing in this high growth sector. This forum is an excellent opportunity for those interested to get involved.

TAPPI's Nanotechnology Division has created the ***International Nanotechnology Standards Coordination Committee*** to provide guidance and oversight of this Roadmap. The committee will work to:

- Coordinate and manage completion of the final draft and future revisions of this Roadmap
- Provide a forum that transcends country boundaries to coordinate standards development around the world
- Provide basic resources for the activities of this committee (collection and distribution of minutes, committee website, support for conference calls, etc.)
- Provide meeting planning support services to host future workshops needed to continue work on the Roadmap

Path Forward

The nanocellulose research community, industry stakeholders and the standards development community need to continue to work together to coordinate international standards development in nanocellulose. The formation of the International Nanotechnology Standards Coordination Committee will be communicated as widely as possible, with invitations for those to become engaged and/or to be kept informed on the committee's activities and progress in completing the Roadmap.

This committee will work to:

1. Disseminate this roadmap to ISO as well as other standards developing organizations; disseminate this roadmap to government, industry, academia and all audiences as appropriate.
2. Guide and coordinate execution of this Roadmap.
3. Define and prioritize goals and tasks (if not clearly identified in this roadmap document), assign responsibilities, measure progress against milestones in this roadmap document and liaise with other standards development bodies.
4. Identify and recruit experts.
Foster working organizational units with ISO, other standards bodies, subject matter experts, industry, academia and appropriate government representatives to provide input, review, and develop the standards.

An example is a potential project group to develop voluntary guidance on product labeling. Knowledge of that activity would be important to nudge activities in appropriate directions. A diagram showing potential coordination is in Figure 4. Specific actions include:

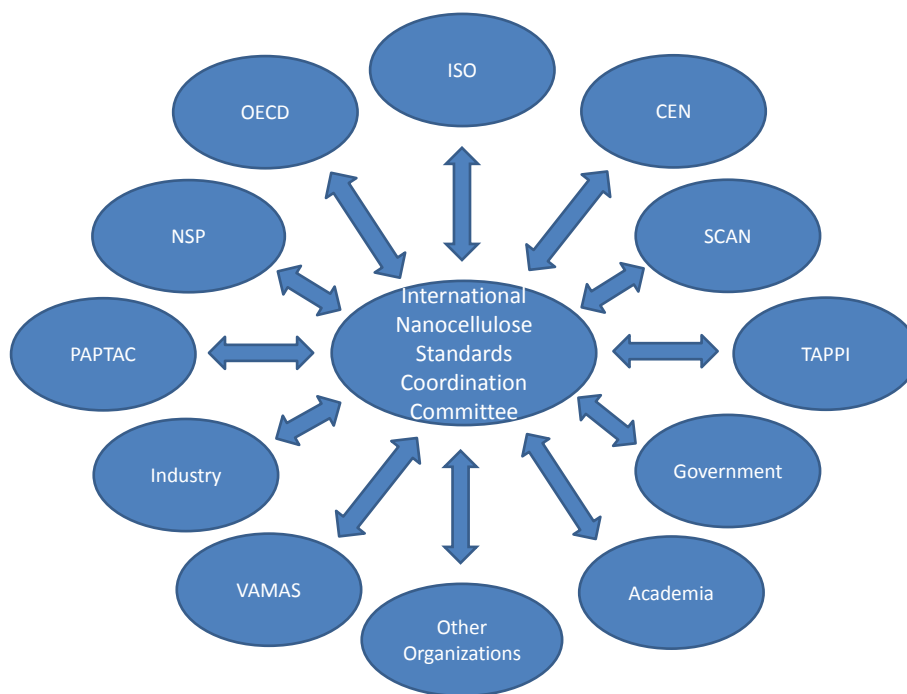


Figure 4. Diagram of potential coordination among standard developing organizations.

7. STRATEGIC COORDINATION WITH GOVERNMENT, INDUSTRY AND ACADEMIA

It is recognized that there is a need to develop international standards for nanocellulose in terminology and nomenclature, measurement and characterization, EHS and material specifications. Governments internationally often have the expertise in nanocellulose research and development, measurement and standards development, regulatory issues in areas of food safety, environmental protection, consumer protection, occupational health and policy issues. Although academic institutions have diverse departmental boundaries, many academic institutions have formed cross-disciplinary centers to target nanotechnology research. As diverse as academic researchers' backgrounds are, they often have the liberty to understand the fundamental principals in nanocellulose research, whether it is material properties, modification chemistry, manufacturing or research related to products development. As nanocellulose has the potential to be commercialized in products across multiple industries, industrial expertise in nanocellulose can cross sectors and vertically cross internal departments. In their pursuit to commercialize nanocellulose products, companies will need to acquire expertise from R&D, marketing, production, sales, legal and regulatory departments. Non-governmental organization participation is also critical, to bring the perspectives of those representing the diverse public interests.

To effectively develop international standards in nanocellulose, government agencies, academia, nongovernmental organizations, and private sectors will have to collaborate and coordinate their efforts to stake out a common target and develop a strategy to achieve the target. Experts from these organizations will bring a diverse knowledge base when they coordinate their efforts to develop international standards for nanocellulose as representatives of their organizations.

The Arlington workshop was an excellent example of government, academia and industry collaboration in international standards development. Workshop participants included experts in nanocellulose, products development, measurement and testing, environmental protection, occupational health, consumer protection and international standards from universities, government and the private sector. When this group of experts met face-to-face at the workshop, they collaborated in discussing the development of international standards in nanocellulose and mapped out a path forward towards the goal.

8. FUNDING OPPORTUNITIES

As evident from the scope of work outlined in this roadmap, significant resources will be required to achieve the stated goals for developing standards for nanocellulose. Resources will be needed to support pre-normative testing, face-to-face meetings, document preparation and other significant tasks in this effort. The Nanocellulose International Standards Coordination Group will be tasked to develop funding, whenever possible, to support nanocellulose international standards development.

9. CONCLUSIONS

The international workshop on nanocellulose international standards held in Arlington VA, U.S.A. provided an opportunity for academic researchers of nanocellulose, representatives from government research institutes, nanomaterial experts, national measurement institutions, industry experts and ISO experts to discuss international standards development for nanocellulose. Experts from several countries presented to workshop attendees the state of nanocellulose development in their countries, the prospect of commercialization in different industrial sectors and the need for international standards based on the state of nanocellulose development. Workshop participants discussed nanocellulose international standard development in four areas: Terminology and Nomenclature, Measurement and Characterization, EHS and Material Specification. Key conclusions from the workshop are:

- Capture workshop results as a roadmap for nanocellulose international standards development.
- Leverage existing standards in nanocellulose international standards development.
- Engage in multiple sector stakeholders from academia, government and industry in nanocellulose international standards development.
- Take a coordinated approach in nanocellulose international standards development: terminology and nomenclature standards may need to be first established before developing international standards in measurement and characterization, EHS and material specification; standards development in measurement and characterization may need to consider measurement needs in EHS and material specification.
- Nanocellulose international standards development needs to be coordinated among standards developing bodies such as ISO, CEN, OECD, NSP, SCA, PAPTAC and TAPPI in different topical areas. The nanocellulose community will establish an International Nanocellulose Standards

Coordination Committee to coordinate nanocellulose international standards development in various standards developing bodies.

- Nanocellulose international standards development in terminology and nomenclature should use the ISO TC 229 nanomaterials naming hierarchy and develop nomenclature concurrent with terminology.
- Development of nanocellulose international standards in Measurement and Characterization will take a “learned approach”. There is a need to conduct pre-normative tests on methods selected from a prioritized list with round robin tests conducted under testing frameworks such as VAMAS.
- EHS nanocellulose international standards need to include toxicology testing, environmental assessment and potential exposure in consumer products. Efforts should be made to get nanocellulose in the OECD round robin process.
- To develop nanocellulose international standards in Material Specification, the nanocellulose community will have to determine the properties for material specification and the test methods to describe these properties. Material specifications methods will need to distinguish between different types of nanocellulose and provide early product differentiation.

Although nanocellulose is still in its early stages of development, commercial scale products will be available in a few years. In order to facilitate commercialization of nanocellulose-based products and remove trade barriers, it is evident that the international nanocellulose community needs to start developing international standards before any nanocellulose-based products are sold in the market place. Terminology and nomenclature of nanocellulose will be the first area ready for international standards development in the near future for two reasons: 1) agreed upon nanocellulose terminology needs to be in place before standards development in other areas; and 2) there is sufficient information to commence international standards development in nanocellulose terminology. The areas of measurement and characterization, EHS and material specification still require pre-normative work and may have to publish normative or informative reports before assessing their readiness for drafting formal new standards development proposals.

TAPPI in the U.S.A. will house the INSCC and TAPPI and its volunteers will provide the necessary support to execute this roadmap including the coordination of drafting new standards proposals and to which standards developing organizations these proposals will be submitted. This roadmap was developed based on the best available knowledge when its content was debated. It will be updated as the science and development of nanocellulose products advances.

10. ABBREVIATIONS

| | |
|-----------------|--|
| AAS/AES | Atomic Absorption/Emission Spectroscopy |
| AFM | Atomic Force Microscope |
| ASTM | American Society for Testing and Materials |
| BET | Brunauer-Emmitt-Teller |
| CE | Capillary electrophoresis |
| CEN | European Committee for Standardization |
| CMC | cellulose microcrystal |
| CMF | cellulose microfibril |
| CNC | cellulose nanocrystal |
| CNF | cellulose nanofibril |
| CNP | cellulose nanoparticle |
| DLS | Dynamic Light Scattering |
| EHS | Environment, Health and Safety |
| EPA | Environment Protection Agency |
| FFF | Field Flow Fractionation |
| FS | Forest Service, United States Department of Agriculture |
| FT-IR | Fourier Transform-Infrared Spectroscopy |
| GC-MS | Gas Chromatography-Mass Spectrometer |
| GPC | Gel Permeation Chromatography |
| HPLC | High Performance Liquid Chromatography |
| ICP | Inductively Charged Plasma |
| INSCC | International Nanocellulose Standards Coordination Committee |
| IR | infrared Spectroscopy |
| ISO | International Organization for Standardization |
| IUPAC | International Union for Pure and Applied Chemistry |
| JWG | Joint Working Group, ISO |
| K _{st} | deflagration index, in testing to assess explosion characteristics of powder |
| MINChar | Minimum Information on Nanoparticle Characterization |

| | |
|----------|--|
| NCC | Nanocrystalline cellulose |
| NIST | National Institute of Standards and Technology |
| NMR | Nuclear Magnetic Resonance |
| NSP | Nordic Standardization Programme |
| OECD | Organization for Economic Co-Operation and Development |
| PAPTAC | Pulp and Paper Technical Association of Canada |
| R&D | Research and development |
| SAXS | Small Angle X-Ray Scattering |
| SCAN | Scandinavia Pulp and Paper Board Testing Committee |
| SEC | Size Exclusion Chromatography |
| SEC-MALS | Size Exclusion Chromatography – Multi-Angle Laser Light Scattering |
| SEM | Scanning Electron Microscope |
| TAPPI | Technical Association for the Pulp and Paper Industry |
| TC | Technical Committee, ISO and CEN |
| TEM | Transmission Electron Microscope |
| TEMPO | 2,2,6,6-tetramethylpiperidine-1-oxyl |
| TGA | Thermal Gravimetric Analysis |
| TR | Technical Report, ISO |
| TS | Technical Specification, ISO |
| VAMAS | Versailles Project on Advanced Materials and Standards |
| WAXS | Wide Angle X-Ray Scattering |
| WG | Working Group, ISO |
| WPMN | Working Party on Manufactured Nanomaterials, OECD |

11. LEAD AUTHORS

Yaman Boluk, Ph.D.

Professor, Nanofibre Chair In Forest Products
Dept. of Civil & Env Eng
University of Alberta
3-142 Markin/CNRL NREF Building
Edmonton, AB T6G 2W2
Phone: +1 780-492-6295
Email: yaman.boluk@ualberta.ca

David Ensor, Ph.D.

RTI Distinguished Fellow
RTI International
3040 Cornwallis Rd
Research Triangle Park, NC 27709
Phone: +1 919-541-6735
Email: dse@rti.org

Ulla Forsström, Ph.D.

Development Manager, Energy and Pulp & Paper
VTT
P.O. Box 1000
02044 VTT, FINLAND
Phone: + 358-40-8202191
Email ulla.forsstrom@vtt.fi

Douglas J. Gardner

Professor
University of Maine
School of Forest Resources and
AEWC Advanced Structures and Composites
Center
231 AEWB Building
Orono, Maine 04469
Phone: 207-581-2846
Email: douglasg@maine.edu

Brian Haydon, P.Eng.

Project Manager
CSA Standards (Canadian Standards Association)
5060 Spectrum Way, Suite 100
Mississauga, ON Canada
Phone: +1 416-747-4006
Email: brian.haydon@csa.ca

Robert Moon, Ph.D.

Materials Research Engineer - US Forest Service
Purdue University
Adjunct Assistant Professor in the School of
Materials Engineering
Birck Nanotechnology Center
1205 W. State Street
West Lafayette, IN 47907-2057
Phone: +1 765-496-3397
Email: robertmoon@fs.fed.us

World L.-S. Nieh, Ph.D.

Coordinating Lead Author

National Program Leader, Forest Products and
Wood Utilization
USDA Forest Service, R&D, Resource Use Science
1601 N. Kent Street, RPC-4
Arlington, VA 22209
Phone: +1 703 605-4197
Email: wnieh@fs.fed.us

Jo Anne Shatkin, Ph.D.

CEO
CLF Ventures Inc
62 Summer St
Boston, MA 02110-1008
Phone: +1 617-850-1715
Email: jashatkin@clf.org

Clayton Teague, Ph.D.

Independent Technology Consultant
8621 Plum Creek Drive
Gaithersburg, MD
Phone: +1 301-330-2018
Email: nanophile@verizon.net

Colleen Walker, Ph.D.

Coordinating Lead Author

Project Manager & Technical Lead
TAPPI
15 Technology Parkway South
Norcross, GA 30092
Phone: +1 770-209-7349
Email: cwalker@tappi.org

Clive Willis, Ph.D.

President
CWIC Inc.
5 Ruelle Kinkora
Pointe-Claire, QC Canada
Phone: +1 514-693-5062
Email: cwillis@videotron.ca

Theodore H. Wegner, PhD

Acting Director
USDA Forest Service
Forest Products Laboratory
One Gifford Pinchot Drive
Madison, WI 53726-2398
Phone: + 1 608-231-9434
Email: twegner@fs.fed.us

12. CONTRIBUTORS

The following individuals participated in the 9 June 2011 Workshop on International Standards for Nanocellulose held in Arlington, Virginia. Four groups were formed to discuss technical needs in the four main areas of standards development. The contributions of all are very much appreciated.

12.1. Terminology and Nomenclature

Ulla Forsström, Ph.D.

Development Manager, Energy and Pulp &
Paper
VTT
P.O. Box 1000
02044 VTT, FINLAND
Phone: + 358-40-8202191
Email ulla.forsstrom@vtt.fi

Douglas J. Gardner

Professor
University of Maine
School of Forest Resources and AEWCA Advanced
Structures and Composites Center
231 AEWCA Building
Orono, Maine 04469
Phone: 207-581-2846
Email: douglasg@maine.edu

Rene Goguen

Vice-President Manufacturing
CelluForce inc
625 Rene-Levesque, suite 1501
Montreal, QC, H3A 1K2
Phone: + 819-845-2771 ext : 58276
Email: rene.goguen@celluforce.com

Brian Haydon, P.Eng.

Project Manager
CSA Standards (Canadian Standards Association)
5060 Spectrum Way, Suite 100
Mississauga, ON Canada
Phone: +1 416-747-4006
Email: brian.haydon@csa.ca

Nathalie Lavoine

Student
LGP2 - INP.Pagora
461 Rue De La Papeterie
Saint Martin D'heres, France
Phone: + 33476826974
Email: nathalie.lavoine@pagora.grenoble-inp.fr

Martha Marrapese, Esq.

Keller and Heckman LLP
1001 G Street N.W.
Suite 500 West
Washington, D.C. 20001
Phone: +1 202-434-4123
Email: marrapese@khlaw.com

Nicole A. Poirier

President
ArboraNano
246 Beaconsfield Boulevard
Beaconsfield, QC, H9W 4A4 Canada
Phone: + (514) 941-0252
Email: nichole.poirier@arbornano.ca

Clayton Teague, Ph.D.

Independent Technology Consultant
8621 Plum Creek Drive
Gaithersburg, MD
Phone: +1 301-330-2018
Email: nanophile@verizon.net

Theodore H. Wegner, PhD

Acting Director
USDA Forest Service
Forest Products Laboratory
One Gifford Pinchot Drive
Madison, WI 53726-2398
Phone: + 1 608-231-9434
Email: twegner@fs.fed.us

Clive Willis, Ph.D.

President
CWIC Inc.
5 Ruelle Kinkora
Pointe-Claire, QC Canada
Phone: +1 514-693-5062
Email: cwillis@videotron.ca

Masato Yamaguchi

Nippon Paper Industries
5-21-1, Oji, Kita-ku, Japan
Phone: +81-3-3911-3408
Email: masato.yamaguchi@np-ja.com

12.2. Measurement and Characterization

Yaman Boluk, Ph.D.

Professor, Nanofibre Chair In Forest Products
Dept. of Civil & Env Eng
University of Alberta
3-142 Markin/CNRL NREF Building
Edmonton, AB T6G 2W2
Phone: +1 780-492-6295
Email: yaman.boluk@ualberta.ca

Jean Bouchard

Principal Scientist
FP Innovations
570 Blvd. Saint-Jean
Pointe-Claire, H9R 3J9 Canada
Phone: +1 514-782-4495
Email: jean.bouchard@fpinnovations.ca

John Cowie

Vice President and Director of Technology
Agenda 2020 USA
1111 19th St NW
Washington, D.C. 20036
Phone: + (202) 463-2449 x2749
Email: john_cowie@agenda2020.org

Christophe Danumah, Ph.D.

Research Council Officer
National Institute for Nanotechnology/NRC
11421 Saskatchewan Drive
Edmonton, Alberta, T6G 2M9, Canada
Phone: (780) 641 1757
Email: christophe.danumah@nrc-cnrc.gc.ca

Dr. Alain Dufresne

Grenoble INP
Pagora - Grenoble INP, BP 65
38402 Saint Martin d'Hères cedex, France
Phone +33 4 76 82 69 95
Email Alain. Dufresne@pagora.grenoble-inp.fr

Angela R. Hight Walker

Senior Scientist
National Institute of Standards and Technology
(NIST)
100 Bureau Drive, Mailstop 8443
Gaithersburg, MD 20899 USA
Phone: +1 301-975-2155
Email: angela.hightwalker@nist.gov

Thi Thu Thao Ho

PhD student
Empa Wood Laboratory, Swiss Federal
Laboratories for Materials Science and
Technology
Ueberlandstrasse 129
8600 Duebendorf, Switzerland
Phone: +41 58 765 4543
Email: thuthao.ho@empa.ch

John Husband

Lead Research Scientist
Imerys Minerals UK
Paper Technology Group, Par Moor Centre
Par Moor Road, Par
Cornwall, UK PL24 2SQ
Phone: + 44-1726818161
Email: john.husband@imerys.com

Robert Moon, Ph.D.

Materials Research Engineer
US Forest Service
Purdue University
Adjunct Assistant Professor in the School of
Materials Engineering
Birck Nanotechnology Center
1205 W. State Street
West Lafayette, IN 47907-2057
Phone: +1 765-496-3397
Email: robertmoon@fs.fed.us

Fernando Pescatori

Senior Technology Specialist
Fibria Celulose S.A.
Centro de Tecnologia Fibria - Rod. Gal. Euryale
de Jesus Zerbine km 84
Jacarei, SP, ZIP 12340-010, Brazil
Phone: +55-12-2128-1317
Mobile +55-12-9717-4123
Email: fernando.pescatori@fibria.com.br

Elaine Cristina Ramires

Universidade de São Paulo, Instituto de Química
de São Carlos
Avenida Trabalhador São-carlense, 400, CP 780
São Carlos, São Paulo, 13560-970, Brazil
Phone: +55 16 81156444
Email: ecramires@iqsc.usp.br

Galina Rodionova

Student
Norwegian Univ. of Science & Tech (NTNU)
Hogskoleringen 6B
7491 Trondheim, Norway
Phone: + (474) 101-8485
Email: galina@radionova.no

John Simonsen

Professor
Oregon State University
120 Richardson Hall
Corvallis, OR 97331 USA
Phone: +1 541 737 4217
Email: john.simonsen@oregonstate.edu

Asko Sneck

VTT Finland
Biologinkuja 7
02044 Espoo, Finland
Email: asko.sneck@vtt.fi

Andras E. Vladar Ph.D.

SEM Project Leader
National Institute of Standards and Technology
Nanometer-Scale Metrology Group
Bldg. 220 A117 Ms 8212
100 Bureau Dr.
Gaithersburg, MD 20899
Phone: +1 301-975-2399
Email: andras.vladar@nist.gov

Hiroyuki Yano, Ph.D.

Professor
Research Institute for Sustainable
Humanosphere, Kyoto University
Gokashou
Uji, Kyoto 611-0011, Japan
Phone: +81-774-38-3669
Email: yano@rsh.kyoto-u.ac.jp

Liyan Zhao

Researcher
Alberta Innovates - Technology Futures
250 Karl Clark Road
Edmonton, Alberta, T6N 1E4, Canada
Phone: 780-450-5226
Email: Liyan.zhao@albertainnovates.ca

12.3. Environment, Health and Safety

Lars Axrup

Research Manager
Stora Enso
Karlstad Research Centre
P.O. Box 9090
SE-650 09 Karlstad, Sweden
Phone: + 46 10 4653122
Email: lars.axrup@storaenso.com

Marie A. Dumontier

Deputy Director, Pulp and Paper Green
Transformation Program
Natural Resources Canada
615 Booth St.
Ottawa K1A 0E9, Canada
Phone: + (613) 947-9039
Email: marie.dumontier@nrcan.gc.ca

Martha Marrapese, Esq.

Keller and Heckman LLP
1001 G Street N.W.
Suite 500 West
Washington, D.C. 20001
Phone: +1 202-434-4123
Email: marrapese@khlaw.com

Scott McNeil

Director
National Cancer Institute of Frederick
PO Box B
Frederick, MD 21702
Phone: + (301) 846-6939
Email: mcneils@ncifcrf.gov

Antonio Norio Nakagaito

The University of Tokushima
Minami Sanjojima-cho 2-1
Tokushima 770-8506 Japan
Phone: + 81-88-656-7364
Email: norio@me.tokushima-u.ac.jp

Brian O'Connor

Program Manager-Environment
FP Innovations
570 St Johns Blvd
Pointe Claire QC H9R 3J9 Canada
Phone: + (514) 630-4121
Email: brian.oconnor@fpinnovations.ca

Hans Henrik Øvrebo

CTO
Borregaard
P. Box 162
1701 Sarpsborg
Phone: +47 913 74 715
Email: hans.henrik.ovrebo@borregaard.com

Juulia Rouhiainen

Consultant
Pöyry Management Consulting Oy
P.O. Box 4 (Jaakonkatu 3)
01621 Vantaa Finland
Phone: +358 10 33 22714
Email: juulia.rouhiainen@poyry.com

Jo Anne Shatkin, Ph.D.

CEO
CLF Ventures Inc
62 Summer St
Boston, MA 02110-1008
Phone: +1 617-850-1715
Email: jashatkin@clf.org

David Skuse

Project Manager
Imerys
Par Moor Road
St. Austell PL242SQ UK
Phone: + 44 7909890907
Email: dskuse@imerys.com

Aleksandr B. Stefaniak

Research Industrial Hygienist
National Institute for Occupational Safety and Health
1095 Willowdale Road
Morgantown, WV 26505
Phone: +1 304-285-6302
Email: ASTefaniak@cdc.gov

12.4. Material Specifications

Ron Crotogino

ArboroNano
246 Beaconsfield Boulevard
Beaconsfield, QC, H9W 4A4 Canada
Phone: +1 514-630-4111
Email: ron.crotogino@arboronano.ca

Gilles Dorris

Program Manager, Biomaterials
FPInnovations
570 boul. Saint-Jean
Pointe Claire, Quebec, H9R 3J9
Phone: +1 514 630 4130
Email: gilles.dorris@fpinnovations.ca

Dan Djukich

Alberta Innovates - Technology Futures
Phone: +1 780-450-5563
Email: dan.djukich@albertainnovates.ca

David Ensor, Ph.D.

RTI Distinguished Fellow
RTI International
3040 Cornwallis Rd
Research Triangle Park, NC 27709
Phone: +1 919-541-6735
Email: dse@rti.org

Anne Savage Franey

President
BioVision Technology
35 Peter St
New Minas, Nova Scotia Canada
Phone: + 1-902-847-1367
Email: afraney@biovisiontech.ca

Dr. Isko Kajanto

UPM

Tekniikantie 2C

Espoo, 02200, Finland

Phone: +358 40 507 3314

Email: isko.kajanto@upm.com

Seung-Hwan Lee

National Inst. of Advanced Industrial Science
&Tech

Email: seunghwan-lee@aist.go.jp

Steven Masia

Research Scientist

SAPPI Fine Paper N.A.

Westbrook Mill

89 Cumberland St

Westbrook, ME 04092

Phone: +1 207-856-3579

Email: steve.masia@sappi.com

Steve Price

Executive Director

Alberta Innovates - Bio Solutions

18th Fl Phipps-McKinnon Building

Edmonton, AB T5J 3G2 Canada

Phone: +1 780-427-2567

Email: steve.price@albertainnovates.ca

World L.-S. Nieh, Ph.D.

National Program Leader, Forest Products and
Wood Utilization

USDA Forest Service, R&D, Resource Use
Science

1601 N. Kent Street, RPC-4

Arlington, VA 22209

Phone: +1 703 605-4197

Email: wnieh@fs.fed.us