Research pathways and outreach to drive cellulosic nanomaterials development

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ABSTRACT: There are many opportunities for development and use of cellulosic nanomaterials, sustainably made from renewable raw materials. Significant challenges in manufacturing and application are being overcome. The Agenda 2020 Technology Alliance has issued a technology roadmap identifying key research challenges in a number of priority areas. The purpose of the roadmap is to seek the engagement of researchers and the support of partners to execute the identified research needs to accelerate commercialization. New research funding opportunities are identified. In addition to the research challenges, there are communications needs that must also be addressed, and public-private partnerships that must be executed.

Technologies are rapidly emerging to unravel the structure and properties of cellulosic nanomaterials, enable their efficient large-scale production, and demonstrate their marketplace potential. Enterprises are increasingly exploring ways to access this wide-ranging potential. In recent months, commercial ventures throughout the world have been announced for application of cellulosic nanomaterials in absorbent personal hygiene products, cosmetics, oil and gas production, lightweight paper-based packaging for liquid food, and biomedical applications such as hydrogels for cell culturing. With so much potential, the question arises what technical challenges remain that, if overcome, could accelerate the pace of development even further.

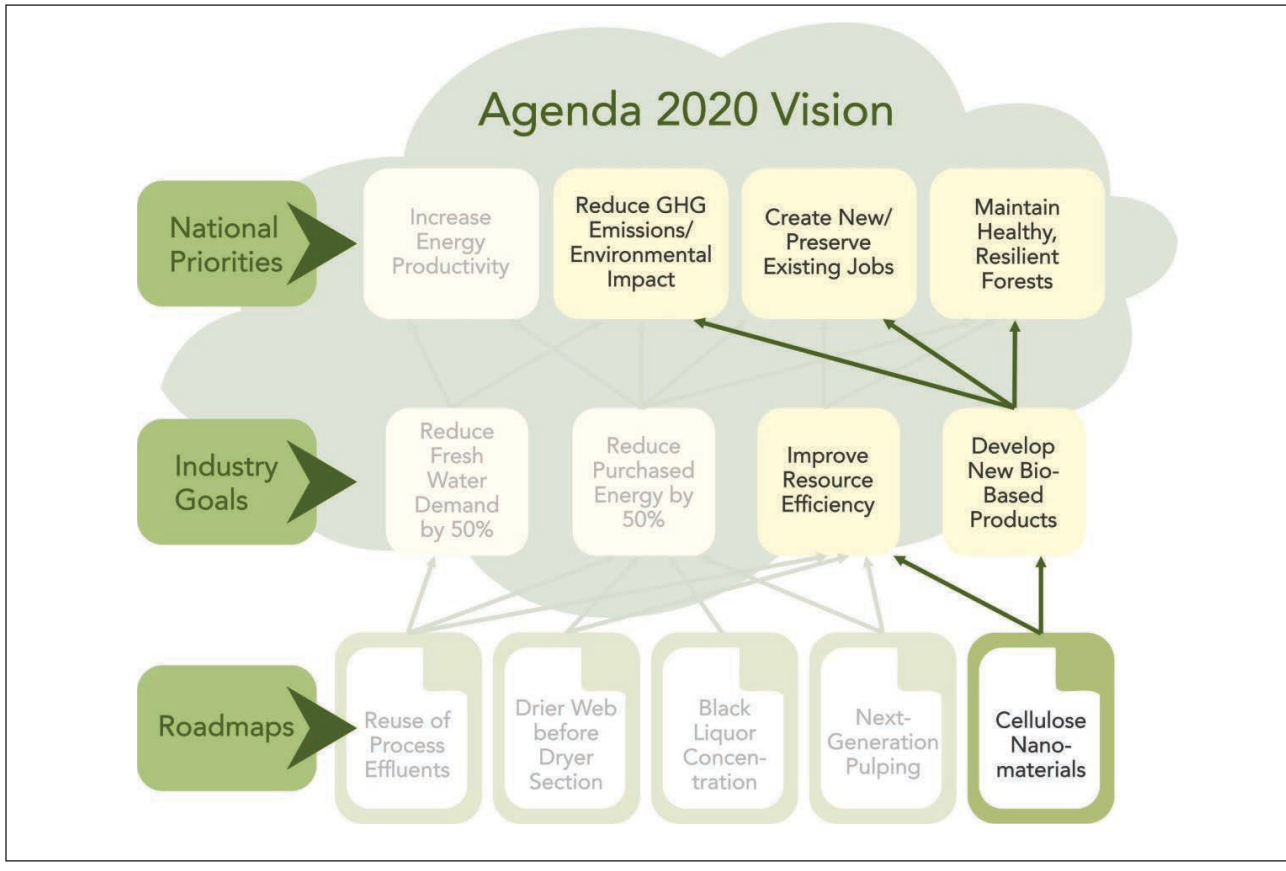
After significant investments, there is now a sufficient volume and diversity of supply of cellulosic nanomaterials available to support a range of product development opportunities. Arriving at this point has required persistentence and ingenuity on the part of researchers and manufacturers to overcome challenges including energy intensity of production, process chemicals recovery, and product variability. Yet market penetration to date is only a tiny fraction of the 35 million metric tons of global market potential forecast by a USDA Forest Service-commissioned study. More research and development (R&D), particularly in the areas of drying, redispersion, and controlling the natural water-holding capacity of cellulosic nanomaterials, would drive further progress.

Cellulose nanocrystal pilot production equipment at the USDA Forest Products Laboratory in Madison, WI.

Vaulting the barriers: Agenda 2020 roadmap priorities

Product developers generally want to move at the speed of the marketplace and not at the speed of research. Realizing the potential of cellulosic nanomaterials is among the forest products industry's most important R&D priorities identified by the Agenda 2020 Technology Alliance. The Alliance exists to identify high-priority, pre-competitive technology challenges for the pulp and paper industry and to promote scientific research and development projects to address them. The goals of the Alliance are reducing water consumption and enhancing water re-use; reducing energy use and greenhouse gas emissions; increasing manufacturing process efficiency; improving raw material yield; and developing new biobased products. Cellulosic nanomaterials fall under new biobased products. These priority goals are well aligned with the sustainability goals of its member companies and with national priorities.

In 2013, the Alliance established work-teams in five areas to identify the technology barriers to achieving its priority goals and develop research pathways to overcome them. In 2014, the Alliance received an Advanced Manufacturing Technology (AMTech) grant from the National Institute of Standards and Technology (NIST) to develop in-depth technology roadmaps to achieve these goals. With this grant, Agenda 2020 conducted a number of workshops and has just published a



1. Agenda 2020 strategic approach.

roadmap on cellulosic nanomaterial research and on each of the other technology areas shown in **Fig. 1**. These roadmaps are available for download at no charge from the Agenda 2020 website (www.Agenda2020.org).

Strategy

The Agenda 2020 cellulosic nanomaterials work team's strategy was to mount a two-pronged pre-competitive research attack on remaining barriers to near-term, large-scale production and application: (1) manufacture and (2) development of technologies to drive commercial application. For manufacture, the team focused on development of product characterization standards and on understanding and removing obstacles to cost-effective dewatering and drying processes that allow for cellulosic nanomaterial redispersion into a slurry for even and economical application in the end-use system.

Today, characterization of a given sample of cellulosic nanomaterial is a laboratory effort. The material is variable due to many factors, such as wood species, age, and manufacturing method, to name just a few. Commonly understood definitions do not yet exist, complicating comparison and replication of experiments and product trials. Broadly accepted nomenclature, coupled with robust methods of characterizing and analyzing cellulosic nanomaterials — methods suited to manufacturing process management and control, as well as to the marketplace — are needed. Markets are difficult to develop in the face of uncertainty about the raw material supply chain.

For enabling applications, the Agenda 2020 team's focus was on identifying surface functionalization methods for improved cellulosic nanomaterial dispersion and compatibility with end-use composite materials. The work team also decided to lay groundwork for addressing product safety and stewardship questions that arise when products are brought to market.

The projects

A set of six priority research pathways emerged:

1. Develop viable dewatering methods.
2. Develop viable drying methods for cellulosic nanomaterials that preserve cellulosic nanomaterials characteristics, prove cost-effective, and meet environment, safety and health, and sustainability goals.
3. Facilitate commercialization of high-volume cellulosic nanomaterial composites by addressing technical challenges associated with scalability, dispersion, and compatibility.
4. Develop standard characterization methods for structure and mechanical properties of cellulosic nanomaterials and cellulosic nanomaterial composites.
5. Prepare an application for U.S. Food & Drug Administration designation of Generally Recognized as Safe (GRAS), to demonstrate product safety and to facilitate use in packaging and food-contact products.

6. Conduct a life-cycle analysis of cellulosic nanomaterials production and application pathways.

Executing the roadmaps

The cellulosic nanomaterials and other Agenda 2020 roadmaps are providing the platforms to move the Alliance from developing "roadmaps" to taking "road trips" (more on procuring the gas money follows). Agenda 2020 plans to seek proposals in 2016 to identify and fund projects to address these high-priority challenges, and to be prepared for rapid response to Federal, State and non-profit funding solicitations for proposals as they emerge.

Agenda 2020 does not suggest that the projects listed in this and its other roadmaps represent exhaustive lists of all of the challenges to be addressed for accelerated commercialization of cellulosic nanomaterials. Agenda 2020 welcomes additional thoughts as to impactful research approaches.

Research funding sources

Where will the pre-competitive funding (i.e., gas money) be found? There are several sources of funding available, and more would be welcome.

Agenda 2020 manufacturing and supplier members have committed to provide company funding for well-grounded research projects supported by solid business cases. Some Agenda 2020 work teams have already begun to develop Requests for Proposals (RFPs). These are being presented to manufacturing company investors to identify interested parties. The RFPs will be issued when there is sufficient expression of member company support for the business cases.

The USDA Forest Service Forest Products Laboratory has provided three rounds of supplemental cellulosic nanomaterials-related research and development funding in recent years, motivated by a desire to create commercial demand for forest resources. The Forest Service views creating demand for forest biomass as a strategy for reducing forest fires; commercial use of forest biomass prompts removal of fuel from the forest while helping to pay for forest restoration operations. Since 2011, this supplemental funding has amounted to approximately $9 million from its annual appropriations. Overall since 2011, USDA Forest Service has invested about $28.1 million total in internal and external cellulosic nanomaterials research (as reported in the NNI annual supplements to the President's budget). This has funded prototype cellulosic nanomaterials production facilities at the Forest Products Laboratory and the University of Maine, university research projects, and an engineering study on commercial production. This past May, the Forest Products Laboratory announced a fourth round of spending, for $1.55 million, to support the P³Nano public-private partnership as described in the following paragraph.

The U.S. Endowment for Forestry and Communities instituted the P³Nano Project to support the development of cellulosic nanomaterials as a means of creating and retaining jobs in forest-reliant communities. The P³Nano project seeks to take cellulosic nanotechnology from the laboratory to commercialization. The U.S. Endowment has invested approximately $5 million in cellulosic nanomaterials research since 2013, with another $2.5 million anticipated this year.

The Renewable Bioproducts Institute (formerly, the Institute of Paper Science and Technology) at Georgia Tech manages an endowment devoted to graduate research and leadership development. The endowment grants about ten new fellowships each year. For the last several years, one of the strategic priorities for the annual RFP has been research in cellulosic nanomaterials. Since academic year 2010-11, the RBI endowment has provided approximately $2.7 million for graduate research related to cellulosic nanomaterials (including three fellowships announced April 20, 2016 — RBI Fellowships - Fall 2016). This amount does not include the additional sums being invested in other areas of Georgia Tech for research by 15 or more additional faculty.

Additional Federal funding

There have been other Federal funding initiatives in addition to those at the Forest Service. Development of ultra-strong, lightweight materials to reduce transportation costs and save energy is in the national interest — even better, if manufactured from renewable forest materials. Accordingly, national laboratories such as Oak Ridge, National Renewable Energy Laboratory, and others, as well as the Bioenergy Technology Office at the U.S. Department of Energy, and other agencies have research programs under way.

The U.S. National Nanotechnology Initiative (NNI) has formed a signature initiative to advance the commercialization of cellulosic nanomaterials (see Nanotechnology Signature Initiative). With the National Institute of Standards and Technology, the NNI has sponsored a number of technical conferences and symposia, notably on metrology and characterization.

USDA National Institute of Food and Agriculture (NIFA), through its various competitive and formula funding programs, supports some nanoscale

science and nanotechnology related to food security through improved productivity, quality, and biodiversity; improved nutritional value of feeds and more effective therapies that significantly impact animal health and wellness; enhanced food safety and biosecurity; and increased protection for natural resources, the environment, and agricultural ecosystems. For 2015, NIFA reported funding its nanoscale science and nanotechnology programs at a level of $13.5 million.

There may also be interest in developing a manufacturing innovation institute devoted to cellulosic nanomaterials. Such a Federal center would provide a coordinating forum and help to reduce commercialization risks, accelerate technology transfer to the marketplace, and facilitate innovation across supply chains.

Ahead in this TAPPI JOURNAL

The researchers presenting their concepts in this *TAPPI JOURNAL* (*TJ*) issue are making important contributions to address objectives that align well with the Agenda 2020 priorities mentioned previously.

1. Beck and Bouchard of FP Innovations explore the relationship between ionic strength and viscosity, critical to many applications.
2. Chauve et al. investigate the effect of surface chemistry on thermal stability and steps to preserve other material properties.
3. Hellström and coauthors compare pretreatment options for microfibrillated cellulose (MFC) production in a high-pressure homogenizer and identify a low-energy option.
4. Johnson and co-authors at the University of Maine and GL&V document the impacts of CNF addition at various levels on paper strength, surface properties, and runnability.
5. Shatkin, Wegner, and Goergen (from Vireo Advisors, the Forest Products Lab, US Forest Service, and the P^{3}Nano project, respectively) describe assessment of information needs for developing Material Safety Data Sheets (MSDS) for cellulosic nanomaterials.
6. Schenker and colleagues address linkages and influences among the rheological properties of carboxymethyl cellulose and microfibrillated cellulose raw materials to the responses of various composites and the potential to generate functional interactions.
7. Wang and Zhu report a correlation between optical transparency of cellulose nanofibril (CNF) suspension and the degree of fibrillation. They found increased tensile strength and Young's modulus of CNF films produced with reduced grinding times.

Agenda 2020 will be reviewing this work with enthusiasm, and hopes readers of *TJ* will, as well.

Moving forward

To accelerate commercialization of cellulosic nanomaterials, key research remaining to be addressed involves optimization of manufacturing processes and development of technologies that leverage the unique properties and features of cellulosic nanomaterials in advanced applications. Agenda 2020 has identified priorities, sources of funding, and outstanding experts at world-class institutions. Agenda 2020 and the TAPPI International Nanotechnology Division urge readers to engage in this intriguing research challenge.

There is also the need to connect developers and manufacturers with demand for advanced materials in current and emerging markets so as to create additional market pull. Nurturing of existing public-private partnerships and the formation of new ones would provide forums to spread awareness, and to anchor critical research in the real world of manufacturing and the marketplace. Who will advocate for cellulosic nanomaterials to those with needs for advanced, versatile, sustainable, renewable, and recyclable raw materials? The industry's marketing and communications arms can help. Commercial success will need a broad, purposeful campaign that traverses the distance from exciting promise to the marketplaces of today and tomorrow. **TJ**

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2. NIST NNMI Funding Opportunity Announcement, 19 February 2016.

ABOUT THE AUTHOR

Kathleen Bennett served as vice president for Bowater, Georgia-Pacific and Fort James. She served as Assistant Administrator of the U.S. Environmental Protection Agency during the Reagan Administration. She is a former president and chair of TAPPI. Bennett currently supports the efforts of Agenda 2020 and the Renewable Bioproducts Institute, Georgia Institute of Technology, in a long-standing consulting role.

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