Challenges and Opportunities in the Scale-up of Cellulose Nanofibril (CNF) Production

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ABSTRACT

Technologies to produce cellulose nanofibrils via either mechanical means (CNF) or chemical means (cellulose nanocrystals (CNC)) have improved dramatically over recent years and a limited number of companies and research labs can produce cellulose nanofibrils on the pilot-scale in significant quantities. The mass production of CNF, however, is not the only obstacle in research and development using nanoscale cellulose in material applications. A big challenge in the utilization of cellulose nanofibrils is agglomeration of the fibrils during processing that occurs because of hydrogen bonding among polymer chains when in close proximity to each other. The impact of agglomeration affects the size distribution of cellulose fibrils, resulting in loss of nanoscale material property effects. Cellulose nanofibrils are typically produced as aqueous suspensions varying from 0.1 to 10 weight percent nanofibrils in water. Obtaining sufficient amounts of dry cellulose nanofibrils in a non-agglomerated condition is challenging. The particular CNF manufacturing process can impact fibril morphology as well as the manufacturing process used to remove water from the CNF suspensions. The overall goal of current research is to develop robust, industrially relevant scalable drying process methodologies for the production of cellulose nanofibrils for application in nanocomposites and related materials.

Keywords: cellulose nanofibers, cellulose nanocrystals, nanofibrillated cellulose, pilot-scale production, drying

INTRODUCTION

Consumers, industry, and the government are increasingly demanding that more products be made from renewable and sustainable resources that are biodegradable, non-petroleum based, carbon neutral, and have low environmental, animal/human health and safety risks. Natural cellulose based materials such as wood, cotton, linen, jute, and hemp have been used by our society for thousands of years and their use continues today as verified by the enormity of the world-wide industries in building products, paper, and textiles. However, the properties, functionality, durability and uniformity that will be required for the next generation of renewable-based products and their engineering applications cannot be achieved with traditional renewable materials. Nanotechnology offers an opportunity for the development of the next generation of products from renewable materials, which will be led by the advances in production of renewable nanoparticles, material characterization, processing of renewable nanoparticles into composites, and the development of multi-length scale modeling capabilities. For example, there is a base fundamental reinforcement unit that is used to strengthen all subsequent structures within trees, plants, some marine creatures, and algae; cellulose nanofibers (CNF).

Recent advances in the production of nano-scale materials from renewable resources provide new opportunities to positively impact the nation's economy and overcome the shortcomings of other nano-scale materials that require imported rare earth metals, need high energy inputs, and are difficult to scale up. These renewable plant-based nanomaterials enable the development of new products and processes to sustainably meet the needs of several segments of the US economy such as packaging, construction, automotive, and water purification. Cellulose-based products are currently being used as fillers or additives in numerous applications including pharmaceuticals, films, paper, packaging materials, plastics as well as coatings. Because of their superior mechanical properties and relatively low cost compared to other nanofiller materials, cellulose nanofibers have been recognized for their potential in the reinforcement of composite materials, a fact reflected in the extensive research activity in the area of cellulose nanocomposites and related materials [1-6].

Advanced manufacturing has been identified as a key to promoting job creation and a strong economy [7]. Specifically identified is "materials by design" and "nanomanufacturing". In a report prepared by the National Nanotechnology Signature Initiative in July 2010, nanocellulose was identified as a future material for strengthening packaging and composites, windows, surfaces, and other structural materials. Cellulose nanomaterials are prominently cited in the recent National Science and Technology Commission (NSTC) subcommittee report that addresses *Sustainable*

Nanomanufacturing–Creating the Industries of the Future (NSTC committee on technology report, 2010). It is estimated that tens of millions of tons per year of wood-derived cellulosic nanomaterials would be used, if developed, and have the potential to add 800,000 direct jobs in rural America and $ 200 billion to the GDP of the U.S. by 2020 [8].

Technologies to produce cellulose nanomaterials from biological methods, mechanical action or chemical means have improved dramatically over the past decade. Companies and research laboratories are currently capable of producing considerable quantities of cellulose nanofibers on the pilot-scale. Mass production of CNF suspensions, however, is not the only obstacle in research and product development employing nanoscale cellulose.

Cellulose nanofibers are typically produced as aqueous suspensions varying from 0.1% to 20% weight percent nanofibers in water. Many applications of cellulose nanofibers require that it be utilized in a dry form, necessitating the removal of large quantities of water from CNF slurries while maintaining its nano morphology. In addition, having suitable quantities of CNF available for evaluation in various manufacturing processes requires substantial quantities beyond the technical and economic feasibility of what can be produced on the bench scale, thus the need for pilot-scale production capacity. CNF development and commercialization efforts have been hampered in the past by the lack of availability of CNF material in sufficient quantities to conduct meaningful technology demonstrations.

PILOT-SCALE CNF PRODUCTION CAPACITY IN THE U.S.

In August, 2012, the U.S. Forest Products Laboratory (FPL) in Madison, Wisconsin unveiled a $1.7 million pilot-scale production facility for renewable, forest-based nanomaterials. This facility is the first of its kind in the United States and one that positions the laboratory as the country's leading producer of nanocellulose materials. The facility is capable of manufacturing cellulose nanocrystals (CNCs) via acid hydrolysis, and TEMPO-based CNF, as well as freeze-drying capacity to produce dry CNFs. Both materials are currently being tested in 400 liter reactors reinforced with glass linings to contain the powerful acid and oxidation conditions necessary for the production of CNC and CNF, respectively. Typically, bleached wood pulp is the starting material for CNC and CNF production. The resulting nano-crystals are approximately 5 nm in diameter and 150 nm long, and the fibrils are about 20 nm in diameter and up to 2 μm long. After extraction, both materials undergo extensive dilution in a membrane filtration system. CNC is available as an aqueous suspension or freeze dried into a white powder. CNF can be cast into a clear film or freeze dried into an aerogel or white powder. The substances' unique structural properties enable them to strengthen a wide-range of materials such as films and fiber reinforced composites. The Forest Product Lab's new facility will aid in the commercialization of these materials by providing researchers and early adopters of the technology with working quantities of forest-based nanomaterials.

The University of Maine (UM) is involved in a research consortium led by the FPL and includes six other universities, and several non-profit research organizations. UM and the FPL entered into a joint venture agreement, with a common goal of researching scalable methods to convert wood components into novel nanomaterials, and developing new generations of high-performance wood-based materials. UM will be the supplier of CNF and CNC to all interested parties and especially researchers from other universities in the consortium, which includes the Georgia Institute of Technology, North Carolina State University, Oregon State University, Pennsylvania State University, Purdue University and the University of Tennessee. In April, 2013, the University of Maine held a ribbon cutting ceremony for a $1.5 million pilot-scale production facility for cellulose nanofibers (CNF) made with mechanical action, sometime also called nanofibrillated cellulose (NFC) or microfibrillated cellulose (MFC). The CNF can be produced via disk refining or using an ultrafine grinder. Suspensions of cellulose nanofibers, at approximately 3% solids, are currently being produced for research and application development purposes. The UM facility is capable of producing up to 1 ton per day of CNF in an aqueous suspension. UM is also installing a spray dryer from GEA-Niro. The drier will be operational by the mid-2013 and will be capable of producing 1 to 2 kilograms of dried nanocellulose per day. UM has developed a patented process [9] for drying cellulose nanofibers and this intellectual property is available for licensing. Spray-drying of NFC produces particulates of dried cellulose which range in size from nano to micron [10]. The resultant materials are viewed as potentially suitable for use as additives or fillers in nanocomposites and related materials. Further, particle morphology and sizes may be tailored by manipulating the spray-drying process parameters [11, 12].

CONCLUSIONS

The pilot-scale availability of novel renewable forest-based nanocellulose should enable the development of new products and processes to sustainably meet the needs of several segments of the US economy such as packaging, construction, and transportation. Imagine transparent nanocomposites produced from forest-based nanomaterials that result in carbon neutral, compostable, and combustible waste streams! Automobile components, fiber-based composites, insulation panels, and other building materials could be made from plant-based nanocomposites that are light weight, high strength, impact resistant, impossible to rust, and low cost. These products and related potentially disruptive technologies utilizing domestically produced renewable materials will positively impact our society and the environment, create new industries and domestic jobs, reduce our dependence on imported petroleum, and sequester carbon.

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