

Production of nanocellulose fibers from pinecone biomass: Evaluation and optimization of chemical and mechanical treatment conditions on mechanical properties of nanocellulose films



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ABSTRACT

Nanocellulose fibers were produced from pinecone (Jack pine: *Pinus banksiana* Lamb) using chemical and mechanical treatments. The effects of pretreatment and mechanical grinding on the tensile strength and modulus of the cellulose fiber sheets were studied to optimize the treatment conditions and grinding process used for the generation of high strength nanocellulose fibers. Pinecone and cellulose fibers were characterized for their chemical composition, morphology, crystallinity, and thermal properties using HPLC, FTIR, SEM, ESEM, XRD, and TGA to provide insight into the mechanism involved in the reactions. Cellulose suspensions that had different pretreatments were processed for different time periods in a Supermasscolloider to produce cellulose nanofibers. The cellulose fibers produced at the optimum chemical concentrations of 4 wt% NaOH and 5 wt% of acidified sodium chlorite solution contained approximately 89% of cellulose, 4% hemicellulose, and 6% lignin. About 67% of the prepared nanocellulose fibers showed a diameter range between 5 and 25 nm. The tensile strength and modulus of the nano fiber films prepared at the optimized grinding condition were 273 MPa and 17 GPa, respectively. The high crystalline index (70%) and improved thermal stability of the nanofibers indicate their suitability for manufacturing bionanocomposites.

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1. Introduction

Cellulose is the most important biopolymer used in various fields worldwide due to its availability, biocompatibility, biological degradability, and sustainability (Alemdar and Sain, 2008a). Cellulose fibers exhibit a unique structural hierarchy derived from their biological origins. The production of cellulose nanofiber from biomass is gaining attention because of its exceptional mechanical and thermal properties, as well as environmental friendliness and cost-effectiveness. Cellulose fiber from lignocellulosic materials is used in various applications such as building materials, particle board, insulation, human food, animal feed, cosmetics,

and medicine (Fukuzumi et al., 2009). Cellulose nanofibers can be produced from various forest and agricultural resources such as wood fibers (Chen et al., 2011), rice straw and potato tuber (Abe et al., 2009), soybean (Wang and Sain, 2007), wheat straw and soy hull (Alemdar and Sain, 2008a), banana rachis (Cordeiro et al., 2012), sugar beet (Liu et al., 2012), and renewable feed stocks (Bhatnagar and Sain, 2004). Since most agricultural biomass is used as animal feed, the sustainable utilization of forest resources is recommended over agricultural resources for the production of nanocellulose fibers, films, and composites.

Pinecone is one of the most common lignocellulosic forest residues in North America and vast quantities are produced throughout the world, especially in pine plantations, for pulp and paper industries. After seed collection, pinecones are generally discarded or burned and there is no additional cost for collecting and drying them. The conversion of this inexpensive and widely available resource to various types of value-added materials is quite promising. Recent studies have shown the effective utilization of pinecone in manufacturing medium density fiberboard (MDF)

Abbreviations: RPC, raw pinecone; ASC, acidified sodium chlorite; BL, bleached (NaOH and acidified sodium chlorite treated) pinecone; C, cellulose; H, hemicellulose; L, lignin; DI, deionized water.

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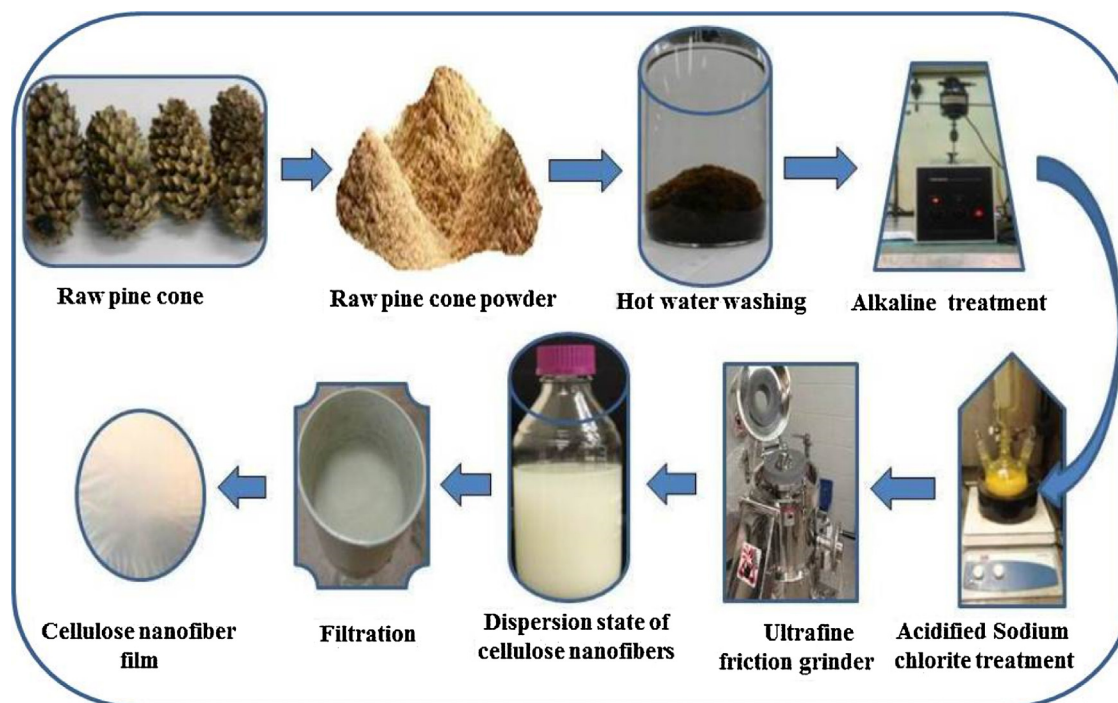


Fig. 1. Schematic view of production of nanocellulose from pinecone biomass using chemical and mechanical treatments.

using a combination of red pinecone, barks, and red pine wood particles in various proportions as raw material (Ayrilmis et al., 2009). Dunder et al. (2010) prepared wood and polypropylene (PP) composites using different mixtures of cone and wood flour. Büyüksarı et al. (2010) studied the effect of pinecone ratio on the wettability and surface roughness of particle board using various combinations of pine and beech wood particles. An extensive literature search indicates that no research in the direction of production of nanofibers from pinecone has been reported to date. This is the first study on the utilization of pinecone as a resource for producing nanocellulose fiber using a chemi-mechanical process. The study focuses on the optimization of pretreatment conditions and the effect of grinding on various mechanical properties of pinecone nanocellulose fibers.

2. Experimental design and procedure

2.1. Materials

Jack pinecones were collected from the University of Saskatchewan campus. All ACS reagent grade chemicals such as sodium hydroxide, sodium chlorite, acetic acid, hydrochloric acid, and ammonium hydroxide were obtained from VWR Chemicals, Canada.

Table 1
Chemical composition of pinecone: (Jack pine: *Pinus banksiana* Lamb).

Chemicals	Content (wt%)
Extractives	5.2 ± 0.5
Ash	0.4 ± 0.1
Cellulose	43.8 ± 2.0
Hemicellulose	27.2 ± 0.1
Kalsol lignin	21.5 ± 1.2
Acid soluble lignin	0.6 ± 0.1

*Sugars and lignin presented as extractives free pinecone.

2.2. Composition analysis of pinecone

To determine the chemical composition of pinecone before and after chemical treatment, the Technical Association of Pulp and Paper Industry (TAPPI) standards TAPPI T222 om-02 for lignin and TAPPI T203 cm-99 for hemicelluloses and cellulose were used. The detailed procedures are discussed in later sections.

2.2.1. Moisture content

The moisture content was determined according to ASTM D4442-07 Standard Test Methods for Direct Moisture Content Measurement of Wood and Wood-Base Materials.

2.2.2. Determination of extractives

The pinecone material was dewaxed in a Soxhlet apparatus with a 2:1 (v/v) mixture of Toluene/ethanol for 6 h at 90 °C, according to the procedure described in NREL/TP-510-42619. After extraction, the solvent was removed using a rotary evaporator equipped with a water bath set to 40 ± 5 °C and inlet pressure to 15–18 psi. After removing all visible solvent, the flask was kept in a 40 °C vacuum oven for 24 h and the amount of extractives in the sample were calculated using the following Eq. (1):

$$\% \text{Extractives} = \frac{\text{Weight}_{\text{flask plus extractives}} - \text{Weight}_{\text{flask}}}{\text{ODW}_{\text{sample}}} \times 100 \quad (1)$$

2.2.3. Determination of lignin, cellulose, and hemicellulose

Holocellulose of the fibers was determined according to the method described by Zobel and McElvee (1996) by dissolving the lignin in acidified sodium chlorite solution. In brief, the procedure involved heating the representative sample with sodium chlorite and acetic acid for 3 h at 70 °C. After heating, the solution was cooled to below 10 °C and filtered through a previously weighed crucible, then washed and dried at 105 °C.

The α-cellulose content was determined according to the Technical Association of Pulp and Paper Industry (TAPPI) procedure T203 om-93 after dissolving the hemicellulose from the

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