



## Direct mechanical production of wood nanofibers from raw wood microparticles with no chemical treatment

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### ABSTRACT

Wood nanofibers (WNFs) were directly isolated from raw wood micro-particles (WMP) of Paulownia using disk grinding with no chemical treatments. TEM analysis showed that the average diameter of the WNF is  $55 \pm 22$  nm, which is 4800 fold less than that of WMP ( $265 \pm 45$   $\mu$ m). The production mechanism of WNF was discussed. XRD results confirmed that the crystallinity and crystallite size of WNF were lower than those of WMP. Chemical composition measurements and FTIR results showed that the chemical compositions of WNF and those of WMP were almost the same. A TGA test also demonstrated that the thermal stability of WNF and WMP was the same (250–260 °C), and the char content of WNF was slightly lower than that of WMP. The direct production of WNF from WMPs with no chemical treatments is an environmentally valuable approach and WNF can be regarded as a promising sustainable nanomaterials with abundant potential applications.

### 1. Introduction

Wood is composed of several main components e.g. cellulose, hemicellulose, lignin, extractives and minerals. Cellulose, a semi crystalline unbranched homo-polymer derived from glucose, constitutes around 40% of wood. Next in abundance is hemicellulose and lignin which varies from 15 to 30 wt% depending on wood species. Hemicellulose, a branched hetero-polymer, is composed of several different carbohydrate monomers including xylose, mannose, galactose, rhamnose, and arabinose. Lignin is a branched hetero-polymer, which is relatively hydrophobic and aromatic in nature. Aside from the lignocelluloses, wood consists of a variety of low molecular weight organic compounds, called extractives which are mainly fatty acids, resin acids, waxes and terpenes. Wood also contains less than 1 wt% inorganic constituents mainly including calcium, potassium, sodium, magnesium, iron, manganese, and so on (Fengel and Wegener, 1989; Rowell, 2005).

Among wood components, cellulose is the main skeleton of wood. It is usually in the shape of micro-scale fiber with a length of 1–4 mm and a diameter of 5–50  $\mu$ m depending on the wood species (Fengel and Wegener, 1989; Lewin, 2006; Rowell, 2005). Cellulose fiber is composed of three main nano-structures including cellulose nanocrystal, elementary fibril and nanofibril, with the latter being the largest nano component of cellulose fiber with an average diameter around 20 nm and a length of around 5  $\mu$ m (Abdul Khalil et al., 2012; Afra et al., 2016;

Afra and Mousavi, 2015; Fengel and Wegener, 1989; Jonoobi et al., 2015; Lavoine et al., 2012; Lewin, 2006; Mashkour et al., 2014, 2015; Sehaqui et al., 2011; Yousefi et al., 2015). The cellulose nano-components are embedded in a matrix of lignin and hemicellulose. This matrix is placed in a nano-gap located along adjacent cellulose nano-components which are mainly located between laterally adjacent nanofibrils (Fengel and Wegener, 1989; Isogai et al., 2011; Lewin, 2006; Nishiyama et al., 2003; Yousefi et al., 2015, 2011b). Also, these nano-gaps are considered as the isolation points of cellulose nanostructures in different production methods. For example, the lateral gap and amorphous regions located between nanocrystals is the isolation point in the acid hydrolysis method which results in cellulose nanocrystal production, or the gap located between nanofibrils is the isolation point in mechanical production methods such as disk grinding, microfluidizer, homogenizer, etc. (Afra et al., 2013; Isogai et al., 2011; Yousefi et al., 2011b).

Fig. 1 shows three different conversion routes of raw wood micro-particles to nanostructures including previously reported methods and the new one introduced in the current study. To isolate pure cellulose nanostructures, wood first passes several chemical purification steps through pulping and bleaching processes (Adel et al., 2016; Kalita et al., 2015; Nechyporchuk et al., 2016; Park et al., 2017; Yousefi et al., 2013, 2011a). Also, lignocellulose nanofibers is produced from unbleached wood pulp and raw wood microparticles (Herzele et al., 2016; Iwamoto et al., 2014; Jahed et al., 2017; Park et al., 2017; Rangan et al., 2017; Rezayati Charani et al., 2013). Both pure cellulose nanofibers and

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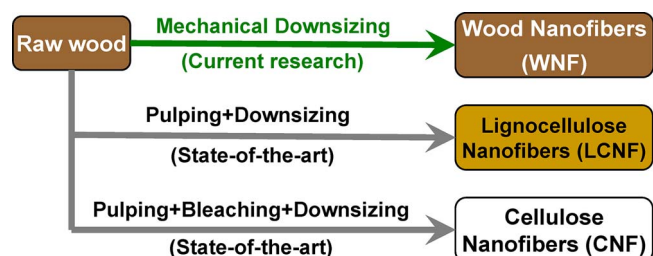


Fig. 1. The production routes of nanostructures from raw wood microparticles. The previously introduced routes contain chemical treatments while our method introduced in the current study does not use any chemicals.

lignocellulose nanofibers need pre-treated raw materials, produced through some chemical reactions including pulping and bleaching. The pulping and bleaching processes are usually time consuming and some of them produce environmentally unfriendly compounds. Many undesirable hazardous compounds are produced during pulping and bleaching processes which needs waste water purification process (Ghose and Chinga-Carrasco, 2013; Kamali and Khodaparast, 2015; Pokhrel and Viraraghavan, 2004). After pulping and bleaching, the purified cellulose fibers are downsized to nanoscale through several mechanical, chemical and chemi-mechanical top-down approaches including acid hydrolysis (Rånby et al., 1949), homogenizing (Turbak et al., 1983), disk grinding (Taniguchi and Okamura, 1998), enzymatic hydrolysis (Janardhan and Sain, 2006), TEMPO mediated oxidation (Saito et al., 2007), solvent-based isolation (Yousefi et al., 2015, 2011b), aqueous counter collision (Kondo et al., 2014) to name a few. Compared to the primary mass weight of wood raw material, the production yield of cellulose nanostructures through the mentioned methods falls between 20 wt% and 40 wt% depending on the cellulose content of the raw material, pulping and bleaching process types and downsizing methods; hence, less than 40 wt% of primary wood raw material mass can be converted to cellulose nanostructures (Abdul Khalil et al., 2014; Oliveira de. et al., 2016). This is regarded as one of the drawbacks of cellulose nanomaterial production from economic and environmental point of views. However, the production yield of lignocellulose nanostructures from less purified cellulose fibers such as mechanical and unbleached pulps can be higher than 40% (Abdul Khalil et al., 2014; Diop et al., 2017; Nechyporchuk et al., 2016).

Based on all the mentioned drawbacks of the previous production routes of nanostructures from raw wood (environmental problems, costly, time consuming, low production yield, etc.), this study was conducted to downsize raw wood microparticles (WMPs) of paulownia directly to nanoscale with no chemical utilization and yield reduction (Fig. 1). Paulownia is known as a hardwood fast growing tree with wood density around 0.35 g/cm<sup>3</sup>. Each paulownia tree could annually produce a cubic meter of wood at the age of 5–7 years (Rencoret et al., 2009). It can be used for several applications such as wood composites (Ayrilmis and Kaymakci, 2013; Candan et al., 2013) and pulp and paper (López et al., 2012), etc. We used a fast, simple and high-yield super disk grinding method to produce wood nanofibers (WNF) from WMP. The super disk grinder contains a pair of rotary and stator stone disks made from silicon carbide (SiC). As the result of centrifugation, a cellulose fiber suspension is forced through a gap between the disks and repeated cyclic pressure and shearing forces is turned it to nanoscale components.

## 2. Experimental

### 2.1. Materials

Paulownia Fortunei wood (cultivated in Gorgan, Iran) was used as the raw material in this study to produce WNF. Acetone, nitric acid and sulfuric acid (Merck Co., Germany) were used to obtain the chemical

compound contents of the raw wood microparticles and wood nanofibers.

### 2.2. Methods

Paulownia wood was first ground to obtain wood particles using a grinder (SK1, Retsch GmbH, Germany). The wood particles were then screened to produce particles with a 40-mesh screen which corresponds to 420 μm pore size. The wood micro particles with a mesh size of 40 are hereafter referred to as wood microparticles (WMP). The WMP were immersed in distilled water for 5 h and then ground again using a super disk grinder (MKCA6-2; Masuko Co., Japan) at 2 wt% concentration to prepare the WNF. The WMP was passed through the grinder 6 times, during which the gap between stator and rotator disks was stepwise decreased from +200 μm to –600 μm and the rotor speed was stepwise decreased from 2800 to 1800. The WNF gel was then concentrated from 2 to 4 wt% using a Sigma 3–18 KS centrifuge (Sigma Co., Germany) at 10,000 rpm for 5 min.

### 2.3. Measurements

A Canon SX 400 digital camera was used to take photos of dry WMPs and WNF gel (4 wt%). The suspension (1 wt%) stability of WMPs and WNFs was also checked after 72 h.

An optical stereo microscope (ZSM-100; Optic Isfahan Co., Iran) was used to observe WMPs before and after grinding with different gap sizes. The length and thickness of 50 WMPs were measured using a DigiMizer (MedCalc Software Co.).

A transmission electron microscope (TEM) (Zeiss EM 10C), at an accelerating voltage of 1.5–5 kV, was used to observe WNF specimens. The average diameter of 50 WNFs was also measured using a DigiMizer (MedCalc Software Co.). All specimens were dried in vacuum and coated with gold prior to observation.

The production yield obtained using the following equation:

$$Y = \left( \frac{W_n}{W_m} \right) \times 100 \quad (1)$$

where, Y is the production yield, W<sub>n</sub> is dry weight of WNF (after grinding) and W<sub>r</sub> is dry weight of WMP (before grinding).

An X-ray diffraction test was carried out using a Bruker D8 Advance (Bruker Co., USA). To prepare WNF for the XRD test, it was first freeze dried using a freeze drier (Beta 2–8 LD plus; Christ Co., Germany) at –40 °C for 8 h. The specimens were irradiated by CuKα radiation at 35 kV and 35 mA, in the range of 2θ = 5–50° at a scanning speed of 1.2°/min.

The Crystallinity index (CrI) was evaluated using the following equation (Segal et al., 1959).

$$CrI = \frac{100(I - I_a)}{I} \quad (2)$$

where I is the diffraction intensity assigned to the 200 reflection of cellulose fiber which is typically in the range of 21–23°, I<sub>a</sub> is the intensity measured at 2θ of 18–21°, where the maximum occurs in a diffractogram for lignin, hemicellulose and non-crystalline cellulose.

The crystallite size of cellulose was estimated by Scherrer's equation (Scherrer, 1918).

$$D = \lambda / \beta \cos \theta \quad (3)$$

where D is the crystallite size, λ is the X-ray wavelength (0.15418 nm), θ is the diffraction angle for the (200) plane, and β is the corrected integral width.

The chemical compound contents of WMP and WNF were measured according to TAPPI standards including T264-om 88 (cellulose), T222-om 97 (lignin), T207-om 97 (extractives) and T211-om 93 (ash). In case of WNF, the gel of WNF was first freeze dried using a freeze drier (Beta-2-8 LD plus; Christ Co. Germany) at –40 °C for 8 h prior to the measurement of its chemical compounds.

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