



Producing nanofibres from carrots with a chemical-free process

Swambabu Varanasi, Leeav Henzel, Scot Sharman, Warren Batchelor, Gil Garnier*

Bioresource Processing Research Institute of Australia (BioPRIA), Department of Chemical Engineering, Monash University, VIC 3800, Australia



ARTICLE INFO

Keywords:

Nanocellulose
Nanofibres
Carrot
Energy consumption
Sustainable
Films

ABSTRACT

The production of nanofibres (NF) from fresh carrots residue was investigated with a mechanical process without using any pulping or bleaching chemicals. Refining with a PFI mill followed by mechanical fibrillation with a homogenizer was used to produce fine NF. Blanching with hot water was carried out to leach the extractives from carrot fibres prior to refining. The energy required to prepare carrot pulp is one order of magnitude lower than for wood pulp and the fibrillation of nanofibres from carrot residue is four times lower in energy than using wood pulp as feedstock. The average diameter and length of carrot NF are 18 nm and 5.1 μm , respectively. The chemical composition of the manufactured nanofibres, as measured by HPLC, was 53% glucose and 47% xylose. Translucent and strong flexible films were prepared from the carrot NF using a filtration based papermaking process. The strength and water vapor permeability of these carrot NF paper like composites are similar to those derived from wood-fibre of comparable dimensions.

1. Introduction

Vast amounts of organic waste from agricultural and food sources are currently lost to landfill. According to Gustavsson et al. (Gustavsson, Cederberg, & Sonesson, 2011), consumers in Europe and North America wasted 95–115 kg/year per capita compared to 6–11 kg/year in sub-Saharan Africa and South/Southeast Asia per capita. Overflowing landfills have required numerous legislations to reduce food waste. Organic waste in landfill produces methane – a potent greenhouse gas 30 times worse than carbon dioxide – which leaches into underground water affecting its quality (Themelis & Ulloa, 2007). One type of agricultural waste is carrot pomace. Worldwide, 37.22 million tons of carrots were produced in 2013, a 30% increase over the past decade (Crawford, 2012). Carrots may be processed into juice, concentrate, or dried food (Sharma, Karki, Thakur, & Attri, 2012). After juicing, approximately 30–50% of the initial mass remains as pulp (Bao & Chang, 1994), sometimes used for animal feedstock, but most often discarded as waste (Siqueira, Oksman, Tadokoro, & Mathew, 2016). This carrot pulp residue from food processing provides an inexpensive, sustainable and renewable source of cellulose fibres. For industrial applications, the waste product from commercial juice bars or food processing plants may also be used as source for carrot pulp at a smaller and local scale. Currently, some of this organic waste is used in bio-refineries to produce biofuels and bioenergy. Waste to product transformation, however, remains the best strategy. Here, we investigate the production of cellulosic nanofibres from carrot pulp waste; this study can then serve as protocol for other vegetables.

Cellulose nanofibres (CNF) are valuable natural polymeric fibres strong, stiff, opaque and of low thermal expansion (Eichhorn et al., 2009; Lavoine, Desloges, Dufresne, & Bras, 2012; Zhu, Fang, Preston, Li, & Hu, 2014). With such properties, CNF promises very versatile industrial applications such as paper packaging, membranes, flexible electronics, LCDs and biomedical. Deconstruction of the plant cell wall to nanofibres is a two-stage process. The first stage converts the plant cell wall to a pulp consisting of macrofibres using mechanical or chemical pulping methods. Depending on the pre-treatment conditions, hemicellulose molecules may be bonded to CNF (Amiralian et al., 2015). The second stage delaminates the macrofibres into nanofibres by mechanical fibrillation. In some cases, Mechanical fibrillation is the most common method to produce CNF. However, this process is highly energy intensive, requiring as much as 70,000 kWh/tonne, as stated by Lavoine et al. (Lavoine et al., 2012).

Various chemical pre-treatment methods, such as TEMPO-mediated oxidation, carboxymethylation, and enzymatic hydrolysis, were investigated to minimize the energy requirements. With the TEMPO-mediated oxidation method, cellulose fibres are oxidized with the addition of NaClO to produce an aqueous cellulose suspension in the presence of catalytic amounts of 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO) and NaBr at pH 10–11 and room temperature. Although this pretreatment drastically reduces the energy consumption of the process from 194400 kWh/tonne (for repeated cycles of homogenizer used) to 1944 kWh/tonne (Isogai, Saito, & Fukuzumi, 2011), it requires longer oxidation time. Further, TEMPO is a toxic (Lavoine et al., 2012), corrosive, expensive and difficult to recover catalyst. TEMPO also has an

* Corresponding author.

E-mail address: gil.garnier@monash.edu (G. Garnier).

unpleasant odour which makes it unacceptable for food application and packaging, for instance. The yield of TEMPO-oxidized Cellulose Nanofibres (TOCN) depends upon the oxidation time. When the oxidation time varies from 6 h to 10 days, yield increases from 20% to 90% (Saito, Kimura, Nishiyama, & Isogai, 2007). With the carboxymethylation pre-treatment method, cellulose fibres are reacted with aqueous NaOH in the presence of water and isopropyl alcohol, followed by reacting with monohaloacetic acid to produce carboxymethylated cellulose (Adden, Brackhagen, Müller, & Petermann, 2013). A key issue with this method is its low reaction efficiency. In the enzymatic pre-treatment method, the enzymatic hydrolysis is performed with endoglucanase (Pääkko et al., 2007). Although enzymatic treatment followed by mechanical disintegration is considered as eco-friendly, the energy consumption is still higher than that of TEMPO NF. None of the current technique produce quality fibres at low energy. Alternative approaches are required to develop a safe, sustainable and inexpensive process, especially for food and biomedical applications.

Never dried agricultural and food wastes have a cell wall structure more open, less dense and complex than wood which should require less energy for the pulping and fibrillation of nanofibres. This is the hypothesis behind this study. There are many reports describing the production of CNF from agricultural waste such as banana peels (Pelissari, Sobral, & Menegalli, 2014), palm fruit bunch (Ferrer, Filpponen, Rodríguez, Laine, & Rojas, 2012), T.pungens (Amiralian et al., 2015), sugar beet pulp (Agoda-Tandjawa et al., 2010) and carrot pomace (Berglund, Noël, Aitomäki, Öman, & Oksman, 2016; Siqueira et al., 2016). Previous investigations have relied on chemical pre-treatments and ignored the critical energy consumption analysis. Also, comparisons with the now commercial wood based CNF have consistently been omitted.

Carrot pomace is an important type of agricultural waste for the quantity produced and as model of food residue. This study reports a sustainable and industrially scalable method to produce Nanofibres (NF) from carrot residue. The fibres produced have similar or superior quality than those made from wood sources. A detailed mass and energy balance analysis of the whole process is performed. A thorough characterization of nanofibres and films made of those is provided. This study reveals that the energy required to manufacture NF from carrot is comparable to the energy consumed with the now classic TEMPO oxidation method using wood pulp.

2. Experimental method

2.1. Carrot nanofibres preparation

Fig. 1 presents the process flow diagram for the production of NF from carrots. Carrots were purchased from the local supermarket. 600 g of carrots were shredded using a 550W Kambrook food processor for approximately 5 min and then blended for 6 min using a 1.5HP Waring Commercial Blender Model 32BL80, USA to reduce the carrots to a pulp. Approximately 910 mL of water was added during blending. The pulp mixture was then blanched by heating to 80 °C for one hour, whilst being stirred continuously to maintain uniform heating. The pulp

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The pulp was refined according to Tappi standard method T 248 with a PFI mill to 10,000 revolutions. To reduce the cellulose fibres further to the nanoscale, the pulp was then homogenised with the GE A Niro Soavi (lab scale) homogeniser. After dilution to 2 wt%, the suspension underwent 5 passes at 1000 bar pressure. The homogenised suspension was then centrifuged for 10 min at 4500G (5000 rpm) to separate the beta-carotene isolated from the nanofibres during homogenization. Once separated, the supernatant was discarded and the fibres were diluted with deionised water, then collected. The resulting nanofibre suspension was 0.97 wt% solids and stored at 4 °C in a refrigerator.

2.2. NF suspension analysis with high performance liquid chromatography (HPLC)

This analysis was carried out as per the standard method NREL/TP-510-42618 (Laboratory Analytical Procedure for the Determination of structural carbohydrates and lignin in biomass developed by National Renewable Energy Laboratory, USA). Around 0.3 g of 10 wt% carrot NF suspension was hydrolysed with 3 mL of 72% H₂SO₄ at 30 °C for 60 min in a high-pressure tube in a water bath. Sample was mixed using a Teflon rod every 5 min. 84 mL of water was added to the hydrolysed mixture and the mild acid hydrolysis was carried out in an autoclave for 60 min at 120 °C then cooled until it reached room temperature. The mixture of pure components of D-Glucose, D-(+)-Cellobiose, D-Xylose, D-Mannose, D-Galactose, D-Arabinose was also hydrolysed under the same conditions of mild acid hydrolysis step and used as reference standard to determine complete hydrolysis products. Hydrolysed samples were filtered using syringe filter and analysed with HPLC (Agilent 1100 series) using RHM-Monosaccharide H+ (8%) column.

2.3. NF characterisation

The diameter distribution of carrot NF sample was measured from SEM images using a Magellan 400 FEGSEM at a voltage of 3kv and 50,000 × magnifications. A drop of suspension was casted on a metal plate, air dried and then platinum coated. From each image, the width of each nanofibre observable was measured using ImageJ. A total of 200 fibre diameters was measured from all images, sorted into bins of 5 nm size and then plotted as frequency percentage (%) versus bin range to illustrate the diameter distributions.

Nanofiber aspect ratio was estimated using the sedimentation method reported by Varanasi, 2013 (Varanasi, He, & Batchelor, 2013). To summarise, ten 250 mL measuring cylinders were filled with NF suspensions of decreasing concentration, ranging from 0.1 to 0.005 wt

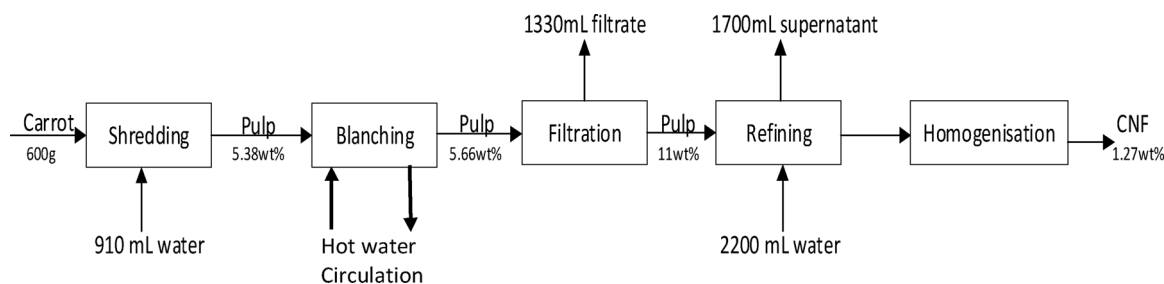


Fig. 1. Process flow diagram for the NF production from carrots.

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