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Corresponding Author: Dr. Kristiina Oksman, Dr

Corresponding Author's Institution: Luleå University of Technology

First Author: Gilberto Siqueira, Dr

Order of Authors: Gilberto Siqueira, Dr; Kristiina Oksman, Dr; Sandra K Tadokoro, Dr; Aji P Mathew, Dr

Abstract: The separation of nanofibers from carrot juice residue and their reinforcing potential is demonstrated. Morphological properties, X-ray diffraction (XRD), and specific surface area (SSA) measurements showed that carrot nanofibers (CNF) have maintained the crystalline structure of native cellulose, while presenting a SSA as high as 246 m².g⁻¹ and diameters between 3 to 36 nm. CNF could be redispersed in water after drying, giving nanofibers with SSA and diameter comparable to properties of initial never-dried CNF. Finally, we propose possible uses of CNF either as strong nanopaper with excellent mechanical properties, i.e. E' of 13.3 GPa and σ_y of 175 MPa, or as reinforcing phase in polymer matrices (CAB). Interesting properties of carrot nanofibers in terms of their possible transport and use from the dry state, as well as their remarkable mechanical properties afforded to nanopaper and nanocomposites may promote their use in environmentally benign constituent in industrial applications.

Suggested Reviewers: Monika Österberg PhD

Associate Professor, Department of Forest Products Technology, Aalto University

monika.osterberg@aalto.fi

She is working with nanocellulose fibers and their surface characteristics.

Gunnar Westman PhD

Professor, Chemical Engineering, Chalmers University of Technology

westman@chalmers.se

He is working with cellulose and its modification

Akira Isogai PhD

Professor, Graduate School of Agricultural & Life Science, The University of Tokyo

aisogai@mail.ecc.u-tokyo.ac.jp

He is expert on nanocellulose processing as well as its use in nanocomposites

Maria L Auad PhD

Associate Professor, Polymer and Fiber Engineering, Auburn University,
MLA0001@auburn.edu

She is working with cellulose and cellulose based nanocomposites

Hiroyuki Yano PhD

Professor, Lab. of Active Bio-based Materials, Kyoto University
yano@rishi.kyoto-u.ac.jp

He is expert on cellulose nanomaterials, their separation processing and nanocellulose use in composites.

Composite Centre Sweden
CCSWE

Luleå, 27th of April 2015

European Editor
Professor Schulte
Composites Science and Technology

Chair Professor Kristiina Oksman
Telephone no.: +46 920 49 33 71
E-mail:kristiina.oksman@ltu.se

Dear Professor Schulte

It is my pleasure to submit our study entitled "*Re-dispersible carrot nanofibers with high mechanical properties and reinforcing capacity for use in composite materials*". I would be very grateful if our manuscript can be considered for publication in Composites Science and Technology.

This manuscript presents an investigation about nanofibers separated from a carrot residue from juice process and they characteristics and use as reinforcement in nanocomposites. The results are very interesting and promising. The main findings are that carrot nanofibers shows very high mechanical properties, they are easily re-dispersed after oven drying, without the addition of surfactants or chemical modifications and this re-dispersion seems also be apparent in the nanocomposites. I think this is a unique property as cellulose usually can't be re-dispersed after drying and is also very difficult to disperse in polymers. It was also seen that the separation process of carrot nanofibers is more energy efficient compared to other raw materials sources when no pre-treatments are performed.

This work is original research and the manuscript is not submitted or published elsewhere.

We feel that Composites Science and Technology is an ideal forum to publish this work and these results would be useful for readers working with cellulose nanofibers and we look forward to receiving your feedback.

Yours sincerely,

Kristiina Oksman
Chair Professor
Director of Composite Centre Sweden

Division of Materials Science
Luleå University of Technology
SE-97187 Luleå, Sweden
+46 (0)920 49 3371
www.ltu.se

Re-dispersible carrot nanofibers with high mechanical properties and reinforcing capacity for use in composite materials

Gilberto Siqueira^{1,2}, Kristiina Oksman^{1,3*}, Sandra K. Tadokoro¹, Aji P. Mathew¹

¹Division of Materials Science, Composite Centre Sweden, Luleå University of Technology, Luleå, Sweden; ²Applied Wood Materials Laboratory, Empa, Swiss Federal Laboratories for Materials Science and Technology, Dübendorf, Switzerland,

³University of Oulu, Fibre and Particle Engineering, Finland

Keywords A: Nanocomposites; B: Mechanical properties, Interface; D: Atomic Force Microscopy; Scanning Electron Microscopy; Dynamic Mechanical Thermal Analysis

Abstract

The separation of nanofibers from carrot juice residue and their reinforcing potential is demonstrated. Morphological properties, X-ray diffraction (XRD), and specific surface area (SSA) measurements showed that carrot nanofibers (CNF) have maintained the crystalline structure of native cellulose, while presenting a SSA as high as $246 \text{ m}^2 \cdot \text{g}^{-1}$ and diameters between 3 to 36 nm. CNF could be redispersed in water after drying, giving nanofibers with SSA and diameter comparable to properties of initial never-dried CNF. Finally, we propose possible uses of CNF either as strong nanopaper with excellent mechanical properties, i.e. E' of 13.3 GPa and σ_y of 175 MPa, or as reinforcing phase in polymer matrices (CAB). Interesting properties of carrot nanofibers in terms of their possible transport and use from the dry state, as well as their remarkable mechanical properties afforded to nanopaper and nanocomposites may promote their use in environmentally benign constituent in industrial applications.

*Corresponding author: Kristiina Oksman, Division of Materials Science, Composite Centre Sweden, Luleå University of Technology, SE- 97187, Luleå, Sweden phone: 46-920-493371 E-mail: kristiina.oksman@ltu.se

1. Introduction

Carrot is an important root vegetable and it is often used for juice production [1]. After juicing, around 30 to 50% of carrot remained as a juice by-product [2]. Although thousands of tons of carrot residue produced in the process of juice extraction can be utilized for animal feed, this agricultural by-product is usually discarded as waste material. The high organic matter content present in that kind of waste generally causes serious environmental problems [1, 3]. Carrot residue (dry) has been reported to contain cellulose (81 %), hemicelluloses (9%), lignin (2.5%) and pectin (7.5%) [4, 5]. The residue also contains small amounts of proteins, lipids and carbohydrates, carotenes and minerals like Ca, P, Fe and Mg [3, 5]. The primary walls of plant cells usually consist of rigid cellulosic nanofibers embedded in a hemicellulose and pectin matrix [6, 7]. These nanofibers have diameters ranging of 3-20 nm and lengths of up to several micrometers depending on their origin [8, 9]. Each nanofiber can be considered as a string of cellulose crystals linked along the fiber axis by disordered amorphous domains [10], e.g. twists and kinks [11]. *In vivo*, cellulose nanofibers presents a predicted stiffness of 130 GPa and strength in the range of 7-8 GPa [12]. However the network of cellulose nanofibers has considerably lower elastic modulus. Abe and Yano [13] have compared the mechanical properties of cellulose nanofibers networks from three different sources, namely wood pulp, rice straw and potato tuber. The measured mechanical properties of the networks had similar properties; the E-modulus was about 11 GPa. Henriksson et al. [14] reported that networks made of cellulose nanofibers, pre-treated with enzymes prior to the mechanical separation, can have *E*-modulus of 14.7 GPa that is decreasing with increased porosity. Due to the high mechanical properties of the cellulose nanofibers, many researchers have utilized them as

reinforcing phase in polymer nanocomposites [15-19]. Different mechanical processes to separate cellulose nanofibers are reported in the literature. Usually, repeated mechanical treatments using high-pressure homogenizer, microfluidizer, grinders, cryo-crushing, or ultrasonic are required to individualize the nanofibers [18-27]. Nakagaito et al. [19] indirectly evaluated the degree of fibrillation of wood pulp, passing through a high pressure homogenizer from 2 to 30 times. It was observed that the disintegration was improved by increasing the number of passes to 30 times. It is not difficult to conclude that higher number of passes results in an increased energy necessary to isolation of nanofibers. Therefore, some pretreatments such as enzymatic acid hydrolysis, and TEMPO-mediated oxidation are being used in order to facilitate the disintegration of cellulose fibers to nanoscale and also to decrease the energy consumption of the process [14, 21, 27, 28]. In general, excessive treatments and passes through grinders and/or homogenizers required to isolate fine cellulose nanofibers can reduce the crystallinity and the degree of polymerization of cellulose chains, leading to degradation of mechanical properties of isolated nanofibers [13, 22]. Here, we report the isolation of carrot nanofibers from a residue of carrot juice process. Extensive characterization of the new cellulose nanofibers revealed that carrot nanofibers have unique properties and low energy consumption in the separation process, with potential application in high-performance polymer nanocomposites. Thus, the reinforcing capability of carrot nanofibers in cellulose acetate butyrate (CAB) nanocomposites is demonstrated.

2. Experimental section

2.1 Materials

Carrots, purchased from the local market, were used as raw material for the juicing. Carrot pulps obtained after juicing were washed with distilled water at 85°C for 2 h. Thereafter the pulp was treated with a 2 wt% NaOH solution at 80°C for 2 h under mechanical stirring, in order to purify cellulose by removing other constituents present in the pulp. The material was then filtered and washed with distilled water until the alkali was completely eliminated. A subsequent bleaching treatment was carried out to remove non-cellulosic components such as lignin, tannin and beta-carotene. The solution used in this treatment consisted of equal parts of acetate buffer, aqueous chlorite (1.7 wt% in water) and distilled water. The bleaching treatment was performed at 80°C for 2 h under mechanical stirring. Once again the material was filtered and washed with distilled water. Cellulose acetate butyrate (CAB-553-0.4), supplied by Eastman Chemical Company (Kingsport, USA) was used as polymer matrix for prepared nanocomposites. The used CAB has a glass transition temperature of 136°C and a melt temperature around 150°C, as reported by the supplier. Ethanol absolute (100 %) purchased from VWR, was used as solvent for the CAB and for the solvent exchange of carrot nanofibers.

Bleached carrot pulp was first disintegrated by a high-shear mixing (Silverson L4RT, Silverson Machine Ltd., England), using 2 wt% concentration. The pulp was ground using a supermass colloidier (MKCA6-2, Masuko Sangyo, Japan) and passed through the instrument for 20 minutes. The grinding was done in contact mode and the gap between the discs was adjusted to 10 µm. The ground fibers were homogenized using a two-chamber high-pressure homogenizer (APV-2000, Denmark) after being diluted with water to 0.1 % consistency and passed through the instrument up to 5 times. The pressure was kept at 600 bars in both chambers.

Fiber networks (nanopapers) of carrot nanofibers were prepared by vacuum filtration of a diluted suspension (0.5 wt%). The suspension was filtrated on a Büchner funnel using Whatman filter paper number 5. After filtration, the wet films were stacked between filter papers and placed between two metal plates and dried at 90°C for 2 h, at pressure of 20 bar. The dried nanopapers were cut in small pieces for the redispersion study. The pieces were swollen in water in order to make a 1% wt suspension and mixed with ultra-turrax for 5 min, at room temperature and 10,000 rpm.

For the nanocomposites preparation, carrot nanofibers (CNF) in aqueous medium, were solvent exchanged to ethanol by successive centrifuge procedures. Polymer matrix (CAB) was first dissolved in ethanol. The solution 15 wt% was magnetically stirred for 12 h at room temperature. Dissolved CAB suspensions were mixed with the appropriate amount of carrot nanofibers to achieve final nanocomposites concentration of 3, 6, 9 and, 12 wt%. The CAB/CNF suspensions were homogenized by magnetic stirring for 4 h, followed by sonication (UP 400S Hielscher Ultrasonics, Germany) for 3 min, to ensure good dispersion of nanofibers in CAB. The CAB/CNF suspensions were then cast in polystyrene Petri dishes and were left to evaporate for 24 h in a preheated vacuum oven at 60°C. In the first 3 h, the pressure of the vacuum oven was controlled to 200 mbar in order to avoid formation of air bubbles; thereafter, the pressure was increased to 850 mbar and maintained for 20 h. The neat CAB solution was exposed to the same procedure. The CAB and the nanocomposite films were removed from the Petri dishes using water and were placed into the vacuum oven, at 70°C and 0 mbar, for one week to remove the remaining solvent.

2.2 Characterization

Scanning Electron Microscopy (SEM) images were acquired with a FEI NovaNano SEM (FEI Company, USA), equipped with Schottky field emission gun. An acceleration voltage of 5.0 kV and working distance of 4.6-5.0 mm were used in the experiments. The samples were frozen in liquid nitrogen, fractured, mounted on a sample holder and coated with gold layer of 5 nm.

Atomic Force Microscopy (AFM) was performed on a Multimode Scanning Probe Microscope with a Nanoscope V (Veeco Instruments USA) using an etched silicon tip with a nominal spring constant of 5 N/m and a nominal frequency of 70 kHz. Height, phase and amplitude images were obtained in tapping mode at the resonance frequency of the cantilever with a scan rate of 0.5 line/s. Prior to AFM analysis a drop of a diluted suspension was deposited on a mica surface and allowed to dry at room temperature.

X-ray diffraction (XRD) analyses of prepared nanopapers were recorded on a Philips X'pert Multipurpose X-ray Diffraction System (MPD) at a speed rate of 0.05°/sec and a range from 5 to 45° (2 θ). Crystallinity index was calculated from the X-ray diffraction patterns according to the following equation [29]:

$$Cr I = \frac{I_{002} - I_{am}}{I_{002}} \quad (\text{eq.1})$$

Where I_{002} and I_{am} are the intensities of the peaks at 2- θ of about 22 and 18, respectively.

Brunauer-Emmett-Teller specific surface area (BET) of CNF was determined by nitrogen physisorption on a surface area and pore size analyzer (Coulter SA3100) by subjecting the CNF suspension beforehand to supercritical drying as reported elsewhere [30].

Mechanical properties of the nanopapers were measured using an Instron 3366 machine equipped with 1kN load cell. The tensile tests were conducted at constant speed of 2

mm/min, at relative humidity of 35% and room temperature (25°C). The samples were prepared by cutting strips of the nanopaper, 20 mm in length, 5-6 mm in width and 0.08-0.10 mm in thickness. A minimum of ten samples was used and the presented results correspond to the average values. Dynamic mechanical analysis (DMA) of the nanocomposite films was carried out using a Q800 (TA Instruments, USA) in tensile mode. The measurements were performed at a constant frequency of 1 Hz, strain amplitude of 0.05 %, in the temperature range from -100°C to 250°C, a heating rate of 5°C.min⁻¹ and sample gauge length 10 mm. The width of the samples varied from 3 to 5 mm, which were measured before each analysis. Two samples were characterized each nanocomposite.

3. Results and Discussion

3.1 Carrot nanofibers

Unbleached carrot pulp consists of thin cell walls associated together and individualization of these generally requires chemical treatments. Alkali treatment followed by bleaching was used for removal of lignin, tannins, and beta-carotene, the orange color of the carrot pulp. After this, the cell walls were well individualized, but the nanofibers were still associated within the cell wall. To individualize the nanofibers mechanical treatments were used and the effect of the treatments is shown in Fig. 1 (A) and (B). Very fine and homogeneous nanofibers were obtained from both, grinding and homogenizing processes. The diameters of nanofibers vary from about 3 to 36 nm for both types of nanofibers measured from the AFM height images. The BET surface area was high 230.5 m².g⁻¹ for ground carrot nanofibers (GCNF) and the homogenized carrot nanofibers (HCNF) presented surface area of 246.4 m².g⁻¹ and corroborate with AFM

measurements where the HCNF present slightly smaller diameters which is expected to lead to higher surface area than GCNF.

The bleaching and mechanical isolation resulted in a yield about 20%, based on the initial carrot weight.

The effect of chemical and mechanical treatments on the crystallinity of carrot fibers was studied using XRD. Fig. 1 (C) shows the diffraction patterns of carrot pulp, bleached carrot pulp and isolated nanofibers by grinding and homogenization process, respectively. Carrot fibers and nanofibers showed the characteristic peaks of cellulose at 2θ angles 16 and 22 from the reflections from 101 and 020 lattice planes, respectively. The crystallinity index values were 48, 73, 68, and 70% for unbleached carrot pulp, bleached carrot pulp, ground carrot nanofibers and homogenized carrot nanofibers, respectively. The slightly higher crystallinity index of bleached pulp and nanofibers compared to unbleached one is explained by partial elimination of amorphous regions from the fibers during alkali and bleaching treatments [23]. However, it is possible to observe that the crystallinity of native cellulose was maintained after disintegration of cellulose into nanofibers (GCNF and HCNF).

3.2 Redispersion of carrot nanofibers

Fig. 2 shows evidence that carrot nanofibers can be dried and then redispersed in water. This is a unique property and can be very important if nanofibers are going to be used in industrial production and in composites applications. It is well known that cellulose nanofibers undergo irreversible agglomeration once dried, known as hornification, which mean they cannot be redispersed [31]. Here, dried nanopaper before redispersion is shown in Fig. 2 (A), after redispersion the water suspension of carrot nanofibers has shown to be stable for more than a month, see Fig. 2 (B). Redispersed carrot nanofibers

presented high fibrillation degree as shown in representative AFM and SEM images of Fig. 2 (C) and Fig. 2 (D). Carrot fibers present around 80.9 wt% of cellulose, 9.1 wt% of hemicellulose, 7.5 wt% of pectin and, 2.5 wt% of lignin [4]. Dinand et al. [32] found residual amount of hemicelluloses and pectin (1 to 3 wt%) on the surface of sugar beet nanofibers. According to them, the small amount of pectin present at the nanofibers was considered as critical for the properties (non-flocculating and birefringence) of nanofiber suspensions. In this work, it was observed that carrot nanofibers were easier isolated, for instance, than bleached wood pulp either in terms of energy consumption (5.1 kWh/kg for carrot nanofibers and 9.6 kWh/kg for bleached wood pulp fibers) and initial dispersion of the fibers prior to fibrillation. It seems that the differences on the surface chemistry of carrot fibers play an important role on the redispersion of nanofibers after drying.

In order to verify the dispersibility of the carrot nanofibers, aerogels of ground nanofibers were prepared, shown in Fig. 3(A) in order to measure the surface area of initial ground nanofibers. Carrot nanopaper was also prepared and its surface morphology is shown in Fig. 3(B). This nanopaper was then cut into small pieces and redispersed in water. (The nanopaper production steps as well as the redispersion procedure are described in the experimental section.) After the redispersion, aerogel was prepared out of redispersed CNF suspension, and a representative image of its microstructure is presented in Fig. 3(C). The structure, corroborate with the surface area measurements obtained by BET. Initially, the ground carrot nanofibers showed high surface area of $231 \text{ m}^2.\text{g}^{-1}$ and high porosity as indicated in Fig. 3(A). No porosity was seen on the nanopaper shown Fig. 3(B) and its specific surface area was measured to be only $0.1 \text{ m}^2.\text{g}^{-1}$. It indicates that the nanofibers in the network were well assembled

by hydrogen bonds after hot-pressing. The redispersion property of the nanopaper is very interesting. The Fig. 3(C) shows highly individualized fibrils and this result is supported by the high surface area of $205 \text{ m}^2.\text{g}^{-1}$ measured for the redispersed carrot nanofibers. Compared to the initial ground carrot nanofibers, the slightly lower surface area measured for the redispersed carrot nanofibers. However, the surface area of redispersed carrot nanofibers is still in the same order of magnitude of ground carrot nanofibers before the redispersion. This can be viewed as an indication that it is possible to redisperse carrot nanofibers, after simple film drying at 90°C under pressure of 20 bars, without suppress their individualization.

3.3 Mechanical properties

Carrot nanopapers with good mechanical properties, can be interesting for many different uses for example in composites, packaging and electronic applications. The associated mechanical properties of the nanopapers prepared from carrot nanofibers isolated by grinding and followed by the homogenization are provided in the Table I, and their typical stress-strain graphs are shown in Fig. 4. The results of tensile tests, especially the yield and maximum strength are depending on phenomena such as the degree of fibrillation, capability to form network, as well as the dimensions and homogeneity of the nanofibers [33]. The properties are fairly linear up to 1.5 %, corresponding to the elastic region (Fig. 4). The yield strength, shown in Table I shows that the HCNF presented higher yield strength (175 MPa) than the nanopaper made of GCNF (129 MPa). Both homogenized and ground carrot nanofibers formed a network with very high E-modulus around 12 and 13 GPa, respectively. The E-modulus of carrot nanofibers network is about 130 times higher than that of cellulose paper [29]. These stiffness values are high when compared to nanopapers obtained from other sources

such as sugar beet (1.8 - 3.2 GPa) [25], wood pulp and rice straw (11 GPa) [13]. One important observation from Table I is that the maximum tensile strength is also very high, ranging 204 and 243 MPa, for homogenized and ground carrot nanofibers, respectively. For instance the tensile strength of prepared carrot nanopapers is higher than the ones obtained for pre-enzymatically treated wood pulp nanopapers (129 to 214 MPa) reported by Henriksson et al. [14] From the environmental and economic point of view, the production of carrot nanofibers could be viewed as an advantage compared to major sources reported in the literature, since they can be obtained from a residue (after juice extraction) and no additional pre-treatments are needed.

3.4 Nanocomposites

Fig. 5 show the mechanical behavior of the prepared nanocomposites analyzed with dynamical mechanical analysis (DMA) in tensile mode. Fig. 5(A) shows the storage modulus (E') of neat CAB as well as CAB/CNF composites as a function of temperature and nanofiber content. The neat CAB matrix displays the typical behavior of amorphous thermoplastic. At the temperature below the main relaxation, the storage modulus of the polymer show little temperature dependence and it slightly decreased with increasing temperature. At 30°C, taken as reference, E' is 2.1 GPa. Above the main relaxation (*e.g.* 160 °C), E' drops more than two orders of magnitude and reached a level of 31 MPa. This result is in agreement with previous studies, where CAB matrix was reinforced with wood cellulose nanofibers and nanocrystals [18, 34]. The E' graphs of carrot composites displayed, qualitatively, similar behavior than the neat CAB; however the storage modulus of the composites was significantly higher than the one of the matrix in the whole temperature range, even below the relaxation temperature. This can be observed in Fig. 5(A), as well as in the Table II. At the temperature of 30°C, E'

increased from ~ 2.1 GPa to ~ 2.6 and 3.5 GPa for the composites containing 6 and 12 wt% carrot nanofibers. A more important mechanical reinforcement could be observed above the transition temperature. For example, at 160°C , E' of the nanocomposites comprising 12 wt% carrot nanofibers presented a modulus of ~ 659 MPa, which corresponds to more than 21 fold enhancement compared to that of the CAB (31 MPa). The higher the carrot nanofiber content is, the higher the reinforcing capability. Fig. 5(B) presents the evolution of mechanical loss factor $\tan \delta$ of CAB and its nanocomposites as a function of temperature. The neat polymer exhibits a peak at 164°C , which can be correlated to the glass-rubber transition temperature of the polymer. The $\tan \delta$ peak remained nearly constant up to 6 wt% carrot nanofiber content in the matrix. For higher nanofibers content (9 and 12 wt%) the position of the peak shifted to slightly lower temperature of 160°C . The magnitude of the $\tan \delta$ peak was found to decrease when increasing the nanofibers content. This is related to a decrease in the number of mobile units participating to the relaxation phenomenon [18]. The results here are somewhat different compared to the DMA results we have reported earlier when nanocellulose crystals and fibers were used in the same matrix [18, 34]. In this study, no change of the transition temperature was observed, as seen in the case of cellulose nanocrystals [34] and cellulose nanofibers network impregnation [18]. Moreover the transition temperature is higher in the present work. The CAB/CNF composites shows a better thermal stability that for example shown in an earlier study by Nakagaito et al [35] where PLA was used as matrix and wood based cellulose nanofibers as reinforcement.

Fig. 6 shows fractured surfaces of the neat CAB and its nanocomposites with 3, 6, 9 and 12 wt% ground carrot nanofibers. The SEM micrographs clearly show the presence of

cellulose nanofibers in CAB matrix. They appear to be generally well dispersed and distributed in the matrix, and the concentration of white dots in the nanocomposites is shown to increase with increased concentration of cellulose nanofibers. The white dots present in the nanocomposites (indicated by white arrows in Fig. 6(E) and shown in higher magnification in Fig. 6(F) are considered as transversal sections of carrot nanofibers inserted in the matrix. The surfaces of the nanocomposites are rougher when compared to the neat matrix where no dots are observed. No presence of carrot nanofibers aggregates is seen and the good dispersion/distribution of nanofibers in the composites is helpful to explain the reinforcing capability of carrot nanofibers in CAB matrix as previously indicated by DMA tests.

4. Conclusions

Nanofibers were successfully isolated from juice process residue, by bleaching followed by mechanical processing, i.e., grinding and high-pressure homogenization. It was shown that the separation process of carrot nanofibers is more energy efficient when compared to other raw materials sources when no pre-treatments are performed. From the morphological point of view, the isolation was reached already during the grinding procedure.

In contrast to major cellulose nanofibers sources described in the literature, carrot nanofibers were easily redispersed after oven drying, without the addition of any surfactants or chemical surface modifications. Even after redispersion, the carrot nanofibers maintained their high surface area and high degree of fibrillation.

The mechanical properties of the produced nanopapers showed that carrot nanofibers created strong and stiff networks with E-modulus between 12.2 and 13.3 GPa and the

maximum strength between 243 and 204 MPa for ground and homogenized nanofibers, respectively.

Finally, the carrot nanofibers showed good dispersion and distribution ability within the polymer and the resulted nanocomposites presented high mechanical properties. In addition an increase in the reinforcing capability of carrot nanofibers at elevated temperatures was shown.

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Figure captions

Fig. 1. AFM micrographs (height and amplitude) of (A) ground carrot nanofibers (GCNF), (B) homogenized carrot nanofibers (HCNF) and their size measurements and (C) XRD patterns of (a) unbleached carrot pulp, (b) bleached carrot pulp, (c) ground carrot nanofibers, and (d) homogenized carrot nanofibers.

Fig. 2. Redispersion of dry carrot nanopaper to well-separated nanofibers (A) carrot nanopaper, (B) stable water suspension (1 wt%), (C) AFM and (D) SEM images of carrot nanofibers after redispersion.

Fig. 3. SEM micrographs of (A) GCNF aerogel with visible nanofibers, (B) GCNF nanopaper showing a dense structure and (C) redispersed carrot nanofibers (R-GCNF) aerogel with well-dispersed and visible nanofibers.

Fig. 4. Typical stress-strain behavior for carrot nanopapers made of ground (GCNF) and homogenized (HCNF) nanofibers.

Fig. 5. DMA of neat CAB and its nanocomposites. (A) Tensile storage modulus (E') and (B) loss factor ($\tan \delta$) of the nanocomposites as a function of temperature. The nanocomposites contain between 3 and 12 wt.% ground carrot nanofibers.

Fig. 6. SEM micrographs of the fractured surfaces of (A) neat CAB and CAB/CNF nanocomposites comprising (B) 3 wt%, (C) 6 wt%, (D) 9 wt% and (E) 12 wt% ground carrot nanofibers and (F) detailed view of CAB/CNF nanocomposites comprising 12 wt% CNF.

Table I. Average mechanical properties of the carrot nanopapers obtained from tensile tests at room temperature.

Sample code	E-modulus (GPa)	Tensile Strength (MPa)	Elongation to Break (%)	Yield Strength (MPa)
GCNF	12.2 ± 1.3	243 ± 28	8.7 ± 0.7	129 ± 5
HCNF	13.3 ± 0.8	204 ± 25	3.1 ± 0.5	175 ± 4

Table II. Tensile storage modulus (E') and loss factor ($\tan \delta$) peak of CAB and its nanocomposites.

Materials	Storage modulus (MPa)		Tan δ (°C)
	At 30 °C	At 160 °C	
CAB	2070	30.9	164 ± 0
CAB/CNF 3%	2111	161.3	163 ± 0
CAB/CNF 6%	2573	435.8	163 ± 1
CAB/CNF 9%	2971	562.5	160 ± 1
CAB/CNF 12%	3521	658.9	160 ± 1

Fig 1

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Fig 2

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