Aloe Vera Rind Nanofibers: Effect of Isolation Process on the Tensile Properties of Nanofibre Films

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The effect of different physico-chemical treatments used in the isolation process of aloe vera (AV) rind nanofibers on the tensile properties of the nanofiber films were studied to understand the root of the low strength values of these films. In the first stage of the investigation, ground AV rind was subjected to different chemical treatments before mechanical defibrillation. In the second stage, cuticle present in the AV rind was removed before subjecting them to chemical treatment. Nanofiber films were prepared using these defibrillated AV rind pulp fibers, and tensile properties were measured to examine the effect of chemical treatments and the raw material characteristics on the nanofibrillation process. The results showed that tensile values were not affected significantly with the chemical treatments; however, the thick cuticle coated on AV rind had a significant effect on the tensile strength and Young's modulus of the nanofibre films. The maximum tensile strength and Young's modulus (234.5 MPa and 12.6 GPa) of AV rind nanofibre films without cuticle were very high compared to the nanofibre films with the cuticle (tensile strength of about 110 MPa and Young's modulus of 10 GPa). The study also found that mild chemical treatments were sufficient to isolate nanofibres from AV rind. The morphological analyses illustrated that the isolated AV rind nanofibers had a diameter size smaller than 20 nm and a high aspect ratio of approximately 1000 to 1500 or larger.

Keywords: Aloe vera rind; Nanofibers; Nanofibrous films; Cuticle; Tensile strength; Young's modulus

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INTRODUCTION

Aloe vera (*Aloe barbadensis* Miller), a succulent plant prevalent in hot and dry regions of Mexico, India, South and Central America, Africa, Australia, Caribbean, and Iran, belongs to the Liliaceae family (Moghaddasi and Verma 2011). Its leaves consist of three layers: a hard green outer rind (around 15 cells thick) covered with very thick cuticle, inner jelly-like parenchyma known as "aloe vera gel", and a thin layer of latex beneath the rind (Klein *et al.* 1988). Aloe vera (AV) is widely cultivated throughout the world for its gel and latex that has been employed for many centuries in cosmetics products, beverages, wound healing, and in the pharmaceutical industry (Klein *et al.* 1988; Choi *et al.* 2001; Eshun and He 2004; Ahmed and Hussain 2013). However, there is little information on chemical composition analyses and utilization of AV rind portion that is currently treated as a waste or fertilizer. The rind layer, which contains vascular bundles running the length of the leaf, is important to the plant due to its physical protection function. Moreover, it is responsible for synthesis of all the naturally occurring nutrients (currently over 200 have

been identified) found in AV (Barcroft and Myskja 2003; Surjushe *et al.* 2008). The rind is covered with a thick cuticle.

A previous investigation by the authors on the analysis and utilization of AV rind as a resource for nanofibers revealed that AV rind has a high amount of α-cellulose (58%) (Cheng *et al*. 2014), and the isolated nanofibers were found to have a diameter under 20 nm. However, the films prepared using the isolated nanofibers in the preliminary studies exhibited a lower tensile strength (102 MPa) and Young's modulus (5.3 GPa) compared to the nanofiber films using the nanofibers isolated from other resources such as wood, rice straw, potato tubes, *etc*. (Abe and Yano 2009; Panthapulakkal and Sain 2012), but using a similar isolation techniques. The low strength characteristics of the AV rind nanofibre (AVRNF) films could be either due to the harsher chemical-physical processing conditions such as pulping, bleaching, and mechanical grinding, that we used for the isolation of nanofibers, or it could be due to the inherent characteristic of the raw material itself. Our aim of this study was to investigate the root of the low strength characteristics of the AV rind nanofibre films. The effect of chemical pre-treatments and the effect of the cuticle present on the AV rind on the mechanical defibrillation of the AV rind pulp were studied to demonstrate the effect of processing conditions and the nature of AV rind on the isolation of nanofibers from AV rind. Tensile properties of nanofibrous films prepared using nanofibers isolated by different conditions were used to achieve the objective. We used tensile properties of the nanofibre films as an indicator of the defibrillation of the fibre bundles in the pulp fibres, as many studies have reported the increase in the strength of the films with increase in the extent of defibrillation (Panthapulakkal and Sain 2012;2013; Iwamoto *et al.* 2008).

EXPERIMENTAL

Materials

Fresh AV leaves (Southern Fields Aloe Inc., South Texas, USA) with length between 40 and 50 cm were used as the raw material in all experiments. Reagent grade chemicals, hydrochloric acid (36 to 38%; Caledon Laboratories Ltd.; Ontario, Canada), glacial acetic acid (Caledon Laboratories Ltd.), sodium hydroxide (Caledon Laboratories Ltd.), ammonium hydroxide (29.7%; J. T. Baker; Ontario, Canada), sodium chlorite (nominally 80%; Alfa Aesar, Ward Hill, MA), 72% sulfuric acid (Ricca Chemical Company; USA), and 1-butanol (99.6%, Caledon Laboratories Ltd., Ontario, Canada) were used as received without further purification.

AV Rind Nanofibers Isolation Process

In the first part of the study, two different chemical methods were used to prepare AV rind pulp fibers before subjecting them to mechanical defibrillation. In another process, cuticle of the AV rind was removed before the chemical treatment and the mechanical defibrillation. The experimental procedures used in these processes are described below.

Process I

The detailed aloe vera rind nanofiber (AVRNF) isolation process used in this process is shown in Fig. 1. The AV leaves were washed to remove dirt from the surface. Gel inside the leaves was then removed using a knife. The obtained outer rind was dried at 60 °C and ground to powder < 2 mm. The dry powder was used to isolate pulp fibers

according to a four-step chemical treatment in Fig. 1. The method used was similar to the previous study by the authors (Cheng *et al.* 2014) except for the bleaching procedure. In the previous study, an acidified sodium chlorite bleaching method at 70 to 80 °C for 4 h was employed twice to remove the lignin. It was anticipated that the harsh bleaching process might have degraded the cellulose chain and could be the reason for a low tensile strength (102 MPa) and Young's modulus (5.3 GPa) of the AV rind nanofibrous films. Therefore, in this study, the bleaching was carried out with a shorter bleaching time of 1.5 h at 70 to 80 °C, aiming to avoid the degradation of the cellulose chain and hence to improve the mechanical properties of the reinforced nanofibrous films. In a typical run, for 100 g of AV rind, 8.6 mL of glacial acetic acid, and 9 g of sodium chlorite in 800 mL of distilled water was used as the bleaching media and the bleaching was carried out at 70 to 80 °C for 1.5 h.

The obtained pulp fibers were then dispersed in water and defibrillated using a commercial grinder (Masuko Corp., Japan, 1500 rpm) to generate a 2% nanofiber suspension at the University of Toronto. The AVRNF after different passes (2, 4, 6, 8, 10, 12, and 15) through the grinder were collected for further characterization and for preparing films.

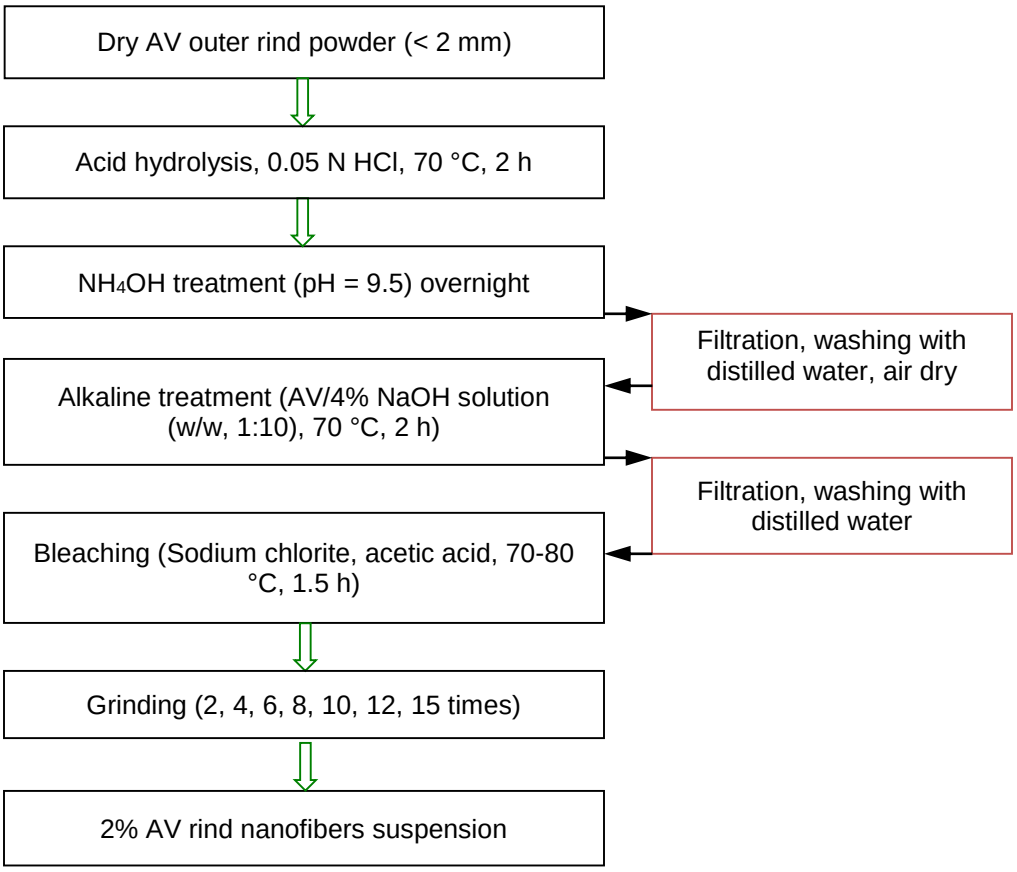

Fig. 1. Isolation process of AV rind nanofibers used in Method I

Process II

The Aloe vera rind pulp fibers were prepared using a modification of the procedure employed in Process I. In this method, NaOH treatment was omitted and a milder bleaching process conditions were used compared to the procedure in Process I. The

procedure used is described in Fig. 2. Bleaching of the AV rind was carried out using a 2% acidified sodium chlorite solution (solid/liquid, 1:20, v/v) with ratio of glacial acetic acid/sodium chlorite 1:1 (v/w) at 50 °C for 4.5 h. The obtained AV pulp fibers were then defibrillated using a similar procedure as in Process I to generate a 2% nanofibers suspension. The AVRNF with different passes (5, 8, 10, 12, and 15) through the grinder were collected for preparing films.

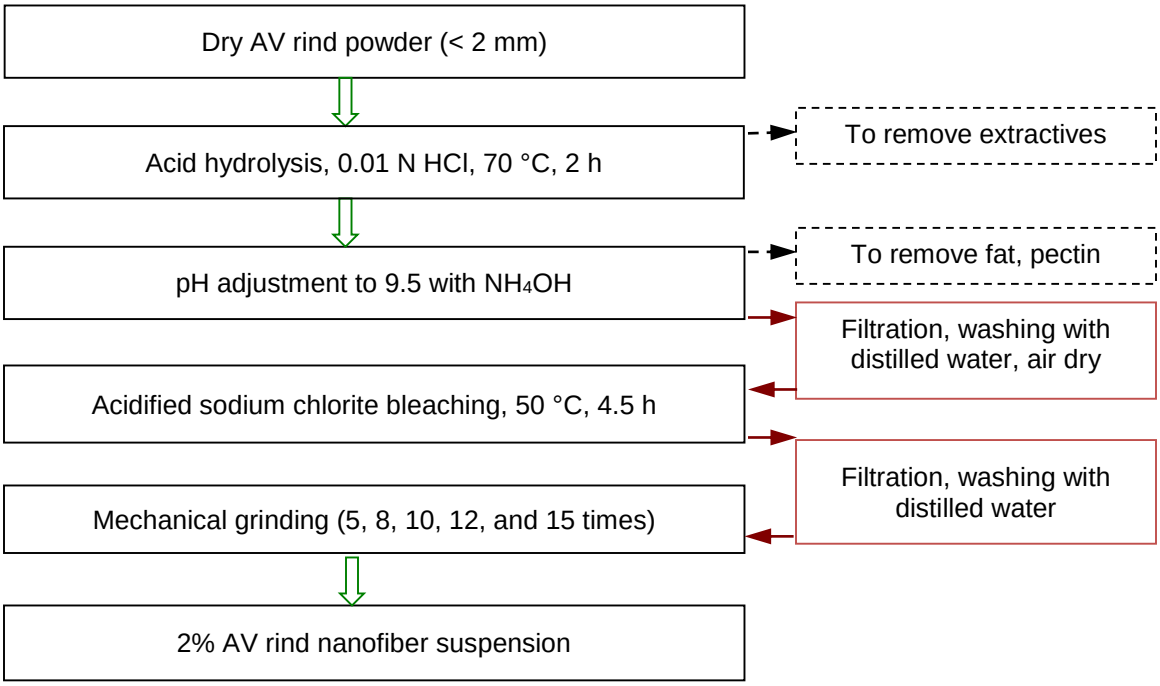

Fig. 2. Isolation process of AV rind nanofibers used in Method II

Process III

A thick translucent cuticle layer is present on top of an AV rind (shown in Fig. 3). It was found that this cuticle could be easily peeled off after boiling for 2 to 3 h.

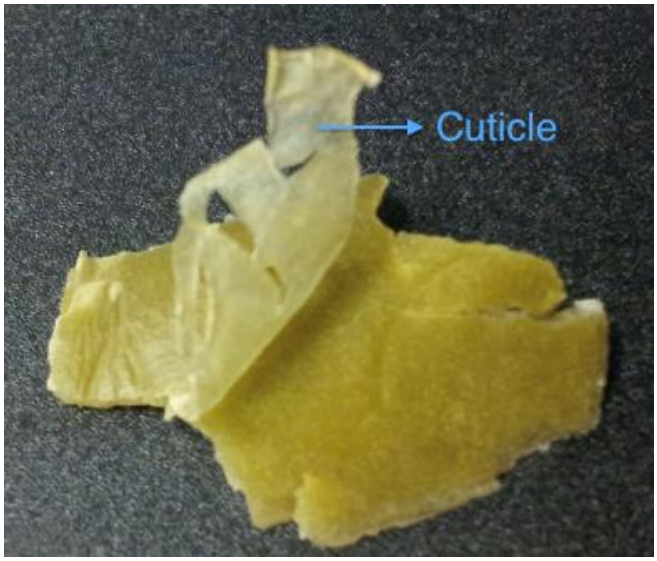

Fig. 3. Thick cuticle coated on top of a piece of boiled AV rind

During Process III, the AV rind, after removal of the cuticle, was used as starting material to prepare pulp fibers using a similar isolation method as in Process II to evaluate the effect of the starting material on the nanofibers preparation. The AVRNF after different passes (5, 8, 10, 12, 15, 20, 25, and 30) through the grinder were collected for preparing nanofibrous films.

Chemical Composition Characterization of AV Rind without Cuticle and AVRNF without Cuticle

Extractives in the cuticle and AV rind (powder, 40 mesh) without cuticle were detected by ASTM standard method D1105-96 (2013) with the three-step process of toluene/95% ethanol (2:1, v/v) extraction, 95% ethanol extraction, and distilled water extraction. The chemical compositions (ash, α-cellulose, hemicellulose, soluble lignin, and Klason lignin) of extractive-free AV rind powder without cuticle and AVRNF powder without cuticle were determined. The detailed procedures were described in previous work by the authors (Cheng *et al.* 2014). In short, holocellulose was measured by treating with sodium chlorite and glacial acetic acid for 3 h at 70 °C. The α-cellulose was subsequently determined by a treatment with 17.5% sodium hydroxide to remove hemicellulose. Klason lignin was analyzed based on an acid hydrolysis method (adapted from TAPPI Standard T222 2002). Soluble lignin was determined according to TAPPI UM 250 standard (1991). Total lignin was calculated by sum of the Klason lignin and soluble lignin.

Preparation of Nanofibre Films and Evaluation of Tensile Properties

Preparation of AV rind nanofibre films and evaluation of mechanical properties in terms of tensile strength and Young's modulus were performed according to the same procedures as detailed in the previous work (Cheng *et al.* 2014). The AVRNF suspension was diluted with distilled water and mixed in a blender to obtain a uniform suspension. Then the diluted suspension was vacuum filtrated using a membrane filter (0.1 μm pores, 9 cm, Pall Corporation; Ontario, Canada) to generate a film with a thickness of around 40 μm. The film was then pressed in between two membranes at ambient conditions under a pressure of 47 psi for 21 min. The films were finally dried at 55 °C overnight before cutting mechanical test specimens, which were then oven-dried at 105 °C for 2 more hours to ensure complete drying.

Evaluation of Density and Tensile Properties of Films

The density of each dried film was calculated according to Eq. 1,

$$\text{Density} = \frac{Weight}{Thickness \times Area} \quad (1)$$

where the area of each specimen was 4.71 cm^2. Tensile properties of all AV rind nanofibrous films prepared were determined according to ASTM D638-10 Type V standard (2010) with the samples cut from the sheet using a standard die ASTM D-638-5-IMP-2. The mechanical properties, tensile strength, and Young's modulus, of the film specimens were then tested using a 3367 Universal testing machine (Instron Corp., USA). The tests were performed at a crosshead speed of 2.5 mm/min with a gauge length of 25 mm and load cell capacity of 2 kN. At least 5 specimens were tested for each sample.

Morphological Analyses of AV Pulp Fibers without Cuticle, AVRNF without Cuticle, and the Nanofibrous Films

The morphology of AV pulp fibers and nanofibres without cuticle was observed using a scanning electron microscope (SEM) and a transmission electron microscope (TEM), respectively. For preparing samples for SEM analysis, a drop of diluted AV pulp fibers in water was deposited on a carbon-coated tape on an aluminum stub, dried at ambient conditions, and sputter coated with 2 to 3 nm of gold. The samples were analyzed on a Hitachi S-3400N Variable Pressure SEM (Japan) operating at an accelerating voltage of 15 kV. To prepare the TEM samples, 10 µL of diluted AV pulp fiber solution was deposited onto glow-discharged carbon-coated TEM grids (400 mesh copper), with the excess water removed by blotting with a piece of filter paper after 2 min. The specimens were stained with 2% uranyl acetate solution for 2 min, then blotted with filter paper to remove the excess stain. The specimens were dried at atmosphere for 5 min before observing (Jiang *et al.* 2013). The AVRNF samples were also investigated with the same method. A Hitachi H-7000 TEM (Hitachi Ltd., Japan) with an accelerating voltage of 75 kV was used for acquiring TEM images. The diameter distribution of AVRNF obtained from TEM was calculated based on two hundred individual measurements using an image processing program of ImageJ (National Institutes of Health, USA). In order to observe the nanofiber distribution in the AV rind nanofibrous films, one film sample prepared without press (47 psi for 21 min) was visualized using SEM. The obtained wet nanofibrous film was soaked in 1-butanol to remove the water, and then the film was freeze-dried (Virtis Frezeemobile 35XL-70, USA). A small piece of the film was then put on carbon-coated tape on an aluminum stub and coated with 3 to 4 nm of gold (Iwamoto *et al.* 2008).

RESULTS AND DISCUSSION

Effect of Process of isolation of Aloe Vera Nanofibers on Tensile Properties

Table 1 shows the density and tensile properties of the AV rind fibre films prepared using defibrillated fibers obtained in Process I, where a shorter bleaching time of 1.5 h at a temperature of 70 to 80 °C was used compared to our preliminary studies (4 h bleaching at 70 and 80 °C, twice) (Cheng *et al.* 2014). The table also shows the effect of mechanical grinding (number of passes through the grinder: 2 to 15) on the density and tensile properties of the films.

Density of the films was slightly lower initially (up to 4 passes) and the value remains more or less constant after 6 numbers of passes, indicating that density did not change significantly with the increase in the extent of defibrillation. Initially, the fibre bundles were not defibrillated, and these fibers forms may lack a close network in the films and exhibits a low density. Once the fibres are defibrillated, these fibers can form a close network in the film leading to an increased density. The values of tensile strength and Young's modulus increased with the number of passes increasing from 2 to 12 times. Similar results of increasing the tensile strength with the increase in the mechanical grinding were reported earlier (Panthapulakkal and Sain 2012; 2013, Cheng *et al.* 2014). This could be attributed to the isolation of the cellulose fibrils from the pulp fibre bundles. These fibrils can form a close network due to the hydrogen bonds and impart higher strength to the films compared to the lower strength pulp fibres in the film (Janardhanan and Sain 2011). However, the highest values of tensile strength (110 MPa) and Young's modulus (8.52 GPa) were still low and similar with the values obtained from the previous

investigation (Cheng *et al*. 2014) as shown in Table 1. These results show that modification of bleaching process did not remarkably improve the mechanical properties of the nanofibrous films, indicating that bleaching is not the only process, but the whole chemical treatment may have to be considered to examine the cause of the lower strength of the AVRNF films.

It is also worth mentioning that both values leveled off for the 15 passes nanofibrous film. The decrease at 15 passes revealed that the degradation of the defibrillated fibers occurred due to the severe mechanical grinding involved in repeated passes through the grinder. Therefore, the number of passes through the grinder needs to be optimized as well.

Table 1. Mechanical Properties of Aloe Vera Rind Nanofibrous Films Prepared using Fibers Obtained in Process I

Number of passes	Density (g/cm^3)	Tensile strength (MPa)	Young's modulus (GPa)
2	0.97 (± 0.04)	40.71 (± 5.22)	6.15 (± 0.28)
4	1.06 (± 0.05)	63.13 (± 7.10)	7.70 (± 0.48)
6	1.14 (± 0.08)	71.83 (± 2.77)	7.80 (± 0.33)
8	1.12 (± 0.03)	83.17 (± 5.11)	8.16 (± 0.25)
10	1.18 (± 0.03)	100.33 (± 8.83)	8.67 (± 0.81)
12	1.11 (± 0.06)	110.22 (± 6.16)	8.52 (± 0.23)
15	1.17 (± 0.09)	86.47 (± 8.95)	8.09 (± 0.51)
Previous investigation (15 passes)[1]	1.28 (± 0.06)	101.12 (± 10.22)	5.29 (± 1.63)

[1] Refer to reference Cheng *et al.* 2014
Data expressed as mean data ± standard deviation

Cellulose microfibrils in agricultural fibers are less tightly wound in the primary cell than in the secondary wall in wood (Siro and Plackett 2010). The NaOH employed above is a pre-swelling agent that could increase the accessibility of the core material to further hydrolysis action to dissolve non-lignin material. Iwamoto *et al.* (2008) reported that alkali treatment on isolation pulp fibers would decrease the tensile strength and Young's modulus of fibrillated pulp sheet. The authors suggested that hemicellulose could contribute to the adhesion between nanofibers and to the stiffness and strength of fibrillated pulp fibers sheet. Fengel (1970) reported that some of the hemicelluloses were incorporated within the cellulose aggregates. The AV rind has a low amount (16.39%) of hemicellulose according to our previous investigation (Cheng *et al.* 2014). This suggests that the 4% NaOH treatment used in the process could be omitted. Moreover, the low lignin content (13.73%) determined in AV rind suggests the use of a mild bleaching method for generating strong and a high amount of cellulose fibers. The milder isolation method should also help reduce the pulp fiber's production costs by reducing the chemical and energy requirements. Therefore, the new pulping process (Process II) was employed to prepare the AVRNF.

Table 2 shows the density, tensile strength, and Young's modulus of the AV rind fibre films with the defibrillated fibres isolated using the process II, as shown in Fig. 2. All the parameters increased with the increasing of number of passes through the grinder. The results show a similar trend as that of the Process I and can be explained in terms of the isolation of the cellulose fibrils from the fibre bundles. However, the highest value of tensile strength and Young's modulus of nanofibrous films after 15 passes were still low and similar with previous results. Nanofibers with more than 15 passes through the grinder were not collected. These results indicate that a mild chemical method for the preparation of AV rind pulp fibers also could not improve the mechanical properties of the reinforced AV rind nanofibrous films. This leads to the conclusion that the low strength properties of the nanofilms could be related to the characteristic of the AV rind and the investigation on the AV rind had been performed to verify this.

Table 2. Density and Mechanical Properties of AV Rind Nanofibrous Films Reinforced with Nanofibers Collected from Different Passes through the Grinder

Number of passes	Density (g/cm^3)	Tensile strength (MPa)	Young's modulus (GPa)
5	1.06 (± 0.06)	70.66 (± 4.40)	7.18 (± 0.53)
8	1.27 (± 0.04)	73.96 (± 6.73)	8.13 (± 0.47)
12	1.42 (± 0.03)	91.72 (± 4.80)	8.74 (± 0.60)
15	1.54 (± 0.04)	110.98 (± 8.12)	10.38 (± 0.52)

Data expressed as mean data ± standard deviation

A thick layer of cuticle on the AV rind was observed, and it was found that this layer comprises about 25% of the total dry weight of the rind. The cuticle has a rough interface with the outer wall of the epidermal cells (Reynolds 2004). It can be easily peeled off after boiling for 2 to 3 h in water. It has a high amount (19.7%) of extractives. It was also noticed that this cuticle covering could not be completely removed by the HCl hydrolysis, or the treatment in 3% H_2SO_4 solution at 121 °C for 1 h, or even when using a bleaching method. It is also harder to grind than AV rind pulp fibers. The presence of this cuticle might be the reason for the lower mechanical properties of the isolated AVRNF films. Therefore, it was peeled off before the chemical-mechanical treatment to isolate AV rind nanofibers. In order to understand the effect of cuticle on the defibrillation (the tensile properties of the nanofibrous films), the AV rind without cuticle were subjected to chemical pulping described in the Process II (Fig. 2). The defibrillated fibres obtained after a different extent of defibrillation (number of passes through the grinder: 5, 8, 12, 15, 20, 25, and 30) were collected to investigate the effect of mechanical defibrillation on the strength of the nanofibre films.

Figure 4 shows the tensile strength and Young's modulus of the AV rind films prepared using defibrillated AV rind fibres without cuticle after different passes through the grinder (number of passes from 5 to 30). The cuticle was also ground through the grinder for 20 passes. The produced suspension was used to prepare films whose density, tensile strength, and Young's modulus were evaluated and illustrated in Fig. 4.

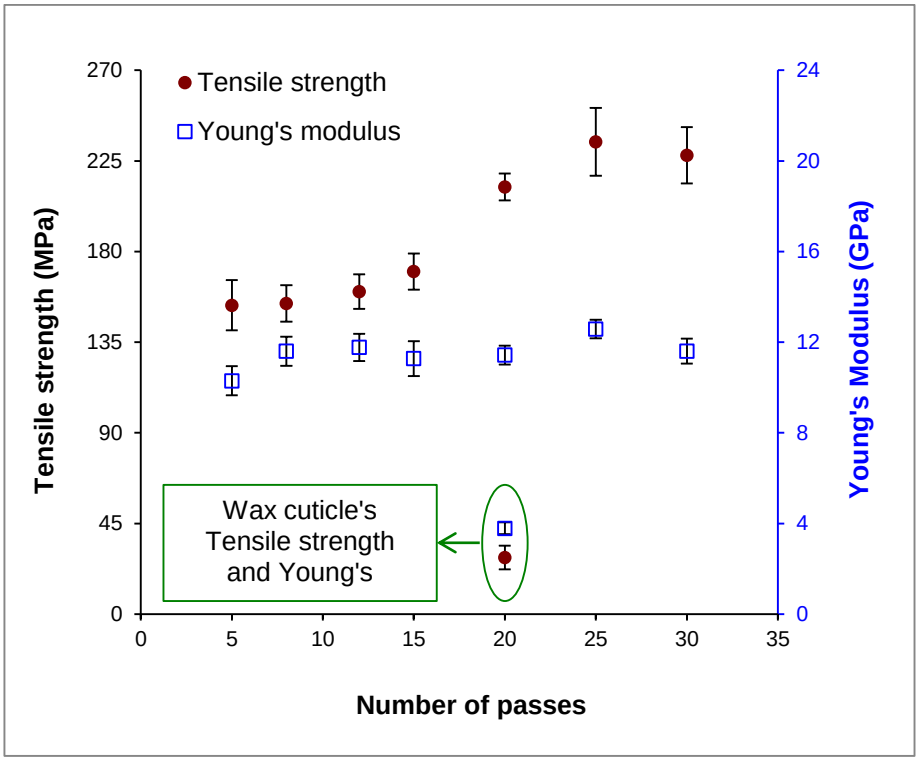

Fig. 4. Tensile strength and Young's modulus of cuticle films and AV rind fibre films prepared with defibrillated AV rind fibres without cuticle

As expected, the cuticle films were found to be very brittle. Both the tensile strength (28.08 MPa) and Young's modulus (3.79 GPa) of the films were very low. They also had a low density of 0.93 g/cm^3 due to the larger thickness of 63 μm than that of the AV rind nanofibrous films ranging from 35 to 40 μm. All the AV rind nanofibrous films without cuticle achieved a similar density of around 1.5 g/cm^3. The tensile strength and Young's modulus of the AV rind films prepared from pulp fibres without cuticle were significantly higher than the films obtained from the fibres with cuticle. The maximum strength and modulus values for the films prepared with AVRNF with cuticle obtained in Process I and II were respectively 110 MPa and 8.5-10 GPa, and the respective number of passes through the grinder were 12 and 15 respectively. The tensile strength and modulus values of the films prepared with AVRNF without cuticle was 170 MPa and 11 GPa after 15 passes, and the maximum value of the strength and modulus were 234 MPa and 12 GPa, respectively. The increase in strength was contributed similarly by the increase of the cellulose fibrils isolation from the fibre bundles with the mechanical grinding. These fibrils can form a strong fibre network in the film due to hydrogen bonds and impart high strength to the films. This indicates that the presence of cuticle in the AV rind had a significant effect on the defibrillation of nanofibres. The tensile strength of the films was leveled off after 25 passes. Further grinding may not increase the strength, as degradation of cellulose-chain may occur due to severe mechanical defibrillation. The results indicate that the optimum number of passes through the grinder was around 25 passes. These results suggest that it is necessary to peel the cuticle off before the isolation of AVRNF fibers. It is worthy of note that milder chemical treatments were sufficient for the isolation of cellulose fibrils from AV.

Chemical Composition Characterization

Table 3 shows the chemical composition of AV rind without cuticle and the prepared AV pulp fibers from the rind without cuticle. Both AV rind and AV pulp fibers had a high amount of ash that was slightly lower than ash content (8.89 %) in AV rind with cuticle (Cheng *et al.* 2014) but much higher than 0.4% in wood (Cheng *et al.* 2010). The AV rind without cuticle presented a high amount of α–cellulose of 50.05 % that was even higher than 40.2% in wood (Cheng *et al.* 2010). The α–cellulose identified in AV pulp fibers was remarkably increased to 77.22%. This finding reveals that AV rind is a good potential resource for isolation of natural nanofibers. Both hemicellulose and lignin contents decreased. The chemical composition and high tensile properties of AV rind nanofibrous films in Fig. 4 further demonstrated that the NaOH treatment was not necessary for isolation of AV rind pulp fibers. In other words, mild chemical treatments are sufficient for the isolation of cellulose fibrils from the AV rind.

Table 3. Chemical Composition of AV Rind and AV Pulp Fibers without Cuticle

Chemical compositions	Ash[1] (%)	α - cellulose[2] (%)	Hemicelluloses[2] (%)	Klason lignin[2] (%)	Soluble lignin[2] (%)
AV rind without cuticle	7.23 (± 0.23)	50.05 (± 0.70)	14.41 (± 0.71)	5.41 (± 0.73)	2.24 (± 0.19)
AV pulp without cuticle	6.65 (± 0.11)	77.22 (± 4.31)	7.17 (± 4.36)	2.25 (± 0.27)	3.28 (± 0.02)
Eastern White pine sawdust[3]	0.4	40.2	21.9	28.4[4]	

[1] On dry and extractive-free basis; [2] On dry, extractive-free, and ash-free basis
[3] Refer to reference Cheng *et al.* 2010; [4] Total lignin (Klason lignin and soluble lignin)
Data expressed as mean data ± standard deviation

Morphological Properties

Figure 5a shows an SEM image of AV rind pulp fibers without cuticle. It is clearly shown that the isolated AV rind pulp fibers consisted essentially of round/ellipsoid-flattened cells commonly called "cell-ghosts" with different diameter size generally smaller than 100 μm. The cells were possibly the chlorenchyma cell wall isolated from the rind. The microfibrillar composition in the "cell-ghosts" wall could be determined by TEM as shown in Fig. 5b. The observed microfibrils were organized in a random interwoven network consisting of individual elements in the "cell-ghosts" wall.

Figure 5c shows a TEM image of AVRNF without cuticle after 25 passes through the grinder. Most of the observed nanofibers were individual and long fibers. The nanofibers were very uniform with diameter size less than 20 nm as measured by ImageJ software. Cellulose microfibrils of 3 to 4 nm in diameter were aggregated with each other into large features called cellulose fibrils or cellulose aggregates ranging from 16 to 25 nm in diameter in processed wood (Fahlen and Salmen 2003). This means that the mechanical treatment by the grinder seems to defibrillate the AV rind pulp fibers into microfibril aggregates. Figure 5d shows that the nanofibers without cuticle after 25 passes through the grinder were *ca.* 20 to 30 μm in length. This means that the aspect ratio (length/diameter) of the nanofibers is around 1000 to 1500 or even larger. The ratio was about 10 to 15 times or even higher than wood fibers (generally 100) (Eder and Burgert 2010). The aspect ratio plays an important role in its performance as a fiber material. The high aspect ratio of AV rind nanofibers could also explain the high tensile strength of the nanofibrous films.

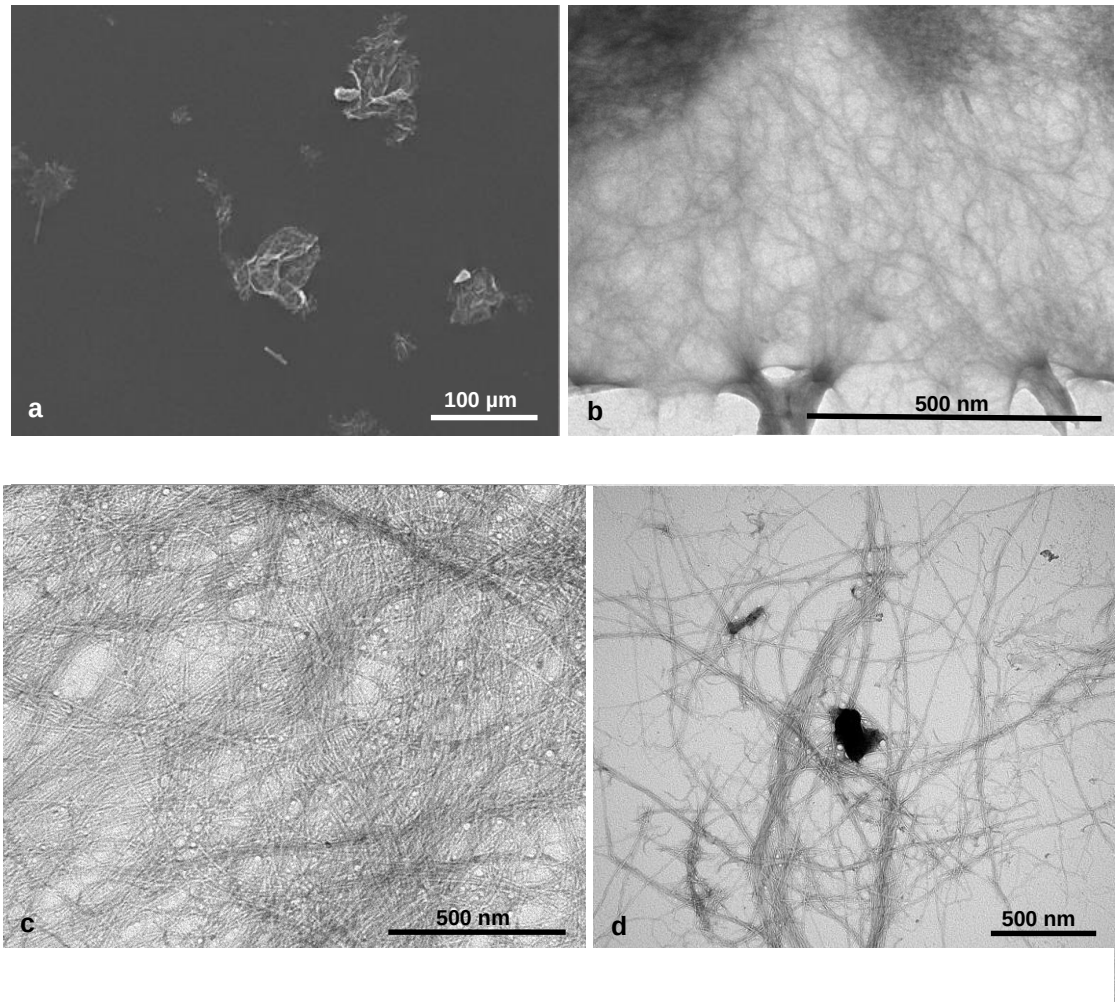

Fig. 5. (a) Scanning electron micrograph of AV pulp fibers without cuticle; (b) Transmission electron micrograph of the "cell ghost" cell wall; (c) and (d) Transmission electron micrograph of AVRNF without cuticle after 25 passes

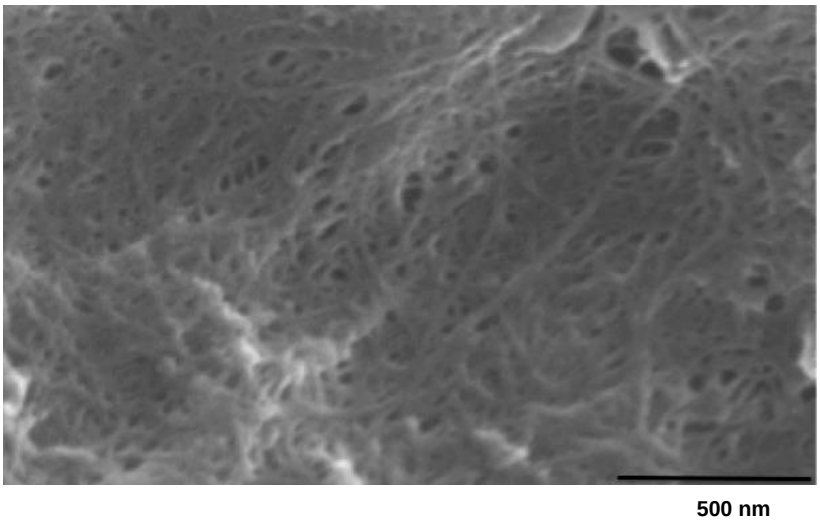

Fig. 6. Scanning electron micrograph of AV rind nanofibrous film prepared without press (nanofibers without cuticle, after 25 passes through the grinder)

Figure 6 shows an SEM image of a piece of AV rind nanofibrous film prepared without press using the nanofibers without cuticle after 25 passes through the grinder. The size of the observed nanofibers was smaller than 30 nm in diameter and approximately 10 to 15 μm in length. The results further supported the assertion that the AV rind nanofibers without cuticle had a high aspect ratio, and most of the observed fibers were in nanometer scale.

CONCLUSIONS

The following conclusions were drawn from this study.

1. The presence of cuticle in the aloe vera (AV) rind is the root of the lower strength characteristics of the AV rind nanofibre (AVRNF) films reported in earlier work by the authors. The cuticle needs to be peeled off before the chemical treatments. The cuticle can be easily peeled off after boiling for 2 to 3 h in water.
2. Mild chemical treatments are sufficient for the isolation of cellulose fibrils from AV rind.
3. Tensile properties of AV rind nanofibre films prepared using AVRNF with and without cuticle after similar chemical treatments and same number of passes through the grinder (number of passes 15) showed that presence of cuticle lowered the strength of the film; tensile strength of 170 MPa and a modulus of 11 GPa for the films prepared with AVRNF without cuticle *vs.* a tensile strength of 110 MPa and a modulus of 10 GPa for the films prepared with AVRNF with cuticle.
4. The nanofiber films prepared using the AV rind without cuticle showed a maximum tensile strength (234.5 MPa) and Young's modulus (12.6 GPa)
5. As evidenced by morphological analysis, the observed microfibrils in the "cell-ghosts" wall (isolated pulp fibers) were organized in a random interwoven network consisting of individual elements. Most of the nanofibers had diameters smaller than 20 nm. The aspect ratio was possibly around 1000 to 1500 or even larger.

ACKNOWLEDGEMENT

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