Preparation and Characterization of Nanofibrillated Cellulose from Waste Sugarcane Bagasse by Mechanical Force

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The effect of different mechanical force application methods was investigated relative to the structure and properties of nanofibrillated cellulose (NFC) from sugarcane bagasse. The NFC was prepared by grinding, high pressure homogenization, and ultrasonication with chemical pretreatment. Fiber morphology, crystalline, chemical structures, and tensile property were analyzed to reveal the mechanisms of NFC production behind different mechanical treatments. The results showed that compared with grinding treatment, high pressure homogenization and ultrasonic treatment can obviously increase the aspect ratio and size uniformity of NFC. Ultrasonic treatment gave the best results. The combination of grinding, high pressure homogenization, and ultrasonic treatment resulted in NFC with the average diameter of 23.18 nm. With the shearing action of grinding, high pressure homogenization, and ultrasonic treatment, the mechanical properties, crystallinity and thermal stability of NFC were gradually enhanced. This results demonstrated that a very low-value agricultural waste product can be easily converted to a high performance nanocomposite with tensile strength of 153.6 MPa and strain at break of 8.83%.

Keywords: Cellulose nanofibers; Sugarcane bagasse; Grinding; High pressure homogenization; Ultrasonic

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INTRODUCTION

With the emphasis on environment protection, the harm of petroleum-based materials to the environment has received increasing attention. This consideration has prompted much research into the development of bio-based polymers to replace non-biodegradable materials (Tang *et al*. 2012). As the most abundant biopolymer, cellulose has received increasing attention due to its wide availability, excellent performance, biocompatibility, low density, thermal stability, and environmental benefits. It is a homopolysaccharide, composed of (β-1,4)-linked-glucopyranose units (Abraham *et al*. 2011) with polymer chains connected by hydrogen bonds and van der Waals forces conferring strength and stability to the plant cell walls. In addition to its widespread use in fiber, paper, films, and polymers, the utilization of natural biomass for processing of novel material has recently attracted increasing interest (Mazalevska *et al*. 2011). Due to superior functionalities, such as extremely large, active surface area, and low cost, the production of NFC from renewable cellulose has gained attention.

NFC consists of particles with nano-sized dimension generated from cellulose macromolecules comprising of both ordered crystallites domains and disordered amorphous domains. The advantages of NFC are its inherent biodegradability, renewability, high tensile strength (10 GPa) and Young's modulus (100 to 160 GPa), large specific surface area, high aspect ratio, and unique morphology (Afra *et al*. 2013; Chen *et al*. 2013). There is a wide variety of wood and non-wood resources suitable for producing cellulose nanostructures.

The development of non-wood sources of cellulose is important because of the shortage of wood resources (Alila *et al*. 2013). By contrast, a serious challenge associated with agricultural intensification is the millions of tons of agricultural wastes generated each year. Approximately 180 million tons of bagasse is produced each year in the world (de Moraes Rocha *et al*. 2015). As an abundant lignocellulosic agro-industrial by-product obtained during sugar manufacturing, sugarcane bagasse fibers represent approximately 30% of the total weight, which are composed of approximately 40 to 50% semicrystalline cellulose, 25 to 35% amorphous hemicellulose, and 10 to 14% lignin (Ju *et al*. 2011). With the rapid progress in material science and engineering in recent year, many new processes and technologies have been developed to produce high-performance new materials using sugarcane bagasse as raw materials (Rossignolo *et al.* 2018; Khonngam and Salakkam 2019); particularly, in the production of pulp and paper and fermentation-based products (Bautista *et al*. 2019). Other products are obtained from processes related to bagasse including chemicals and metabolites such as alcohol and alkaloids, fungi, and animal food enriched with proteins and enzymes (Gil-López *et al.* 2019). Recently, the isolation of nanocellulose from sugarcane bagasse has been the focus of studies (Liu *et al.* 2020; Macías-Almazán *et al.* 2020). The mechanical performance of NFC can be tuned *via* modulation of their geometric shapes and stacking patterns in the matrix (Sun *et al.* 2018).

Mechanical defibrillation (mainly including ultrafine grinding, homogenization, microfluidization, and cryocrushing) is considered to be an essential step for NFC production, during which NFC particles are isolated by delamination of fiber cell wall under successive applications of mechanical force (Jiang *et al.* 2019). The use of conventional methods to extract biopolymers from agricultural wastes at high temperatures may lead to degradation of biopolymers and worsen their performance, which forces industry and researchers to replace traditional methods with alternative technologies. With simplicity, high efficiency and the lack of a requirement for organic solvents, high pressure homogenization (HPH) treatment is an efficient technology for isolation of cellulose fibers from biomass (Li *et al*. 2012; Wang *et al*. 2015; Ninomiya *et al*. 2018). The insolubility of natural cellulose in water and most organic solvents causes clogs in the valve of the homogenizer, and for this reason it is difficult to refine it uniformly. Therefore, a pretreatment of cellulose is essential prior to homogenization to avoiding clogging the homogenizer (Li *et al*. 2012).

In this study, NFC from sugarcane bagasse was successfully isolated by mechanical forces treatment generated from grinding, high pressure homogenization (HPH), and ultrasonication. The structural and physicochemical properties of NFC were studied by field emission scanning electron microscope (FE-SEM), X-ray diffraction (XRD), thermogravimetric analysis (TG), Fourier transformed infrared (FTIR) spectroscopy, and tensile property test. The main objective of the present study was to explore the morphological changes and mechanism of NFC under different mechanical treatments.

EXPERIMENTAL

Materials

The sugarcane bagasse was obtained in the southern region of Anhui Province of China, during the harvest of September 2018. Hydrochloric acid (HCl) was purchased from Aladdin Biochemical Technology Co., Ltd. (Shanghai, China). Acetic acid (CH_3COOH), hexan (C_6H_{14}), sodium chlorite ($NaClO_2$), and potassium hydroxide (KOH) were supplied from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China).

Preparation of Nanocellulose from Sugarcane Bagasse

The bagasse sample was washed thoroughly in running water to remove all sugar residues from freshly collected sugarcane bagasse. The dry bagasse was manually shredded into small pieces (1 to 3 cm). A hot air oven drying (at 60 °C) was used to further dry the cut bagasse. The bagasse was pulverized using a mill and then sieved to gain a bagasse powder with particle size of 420 to 630 μm.

The dried and ground bagasse samples were first defatted using a Soxhlet apparatus with C_6H_{14} for 6 h. Cellulose fibers was extracted from sugarcane bagasse using a three-step process. The defatted bagasse powder in 0.7 wt.% $NaClO_2$ solution was boiled under reflux at pH 4 (adjusted by CH_3COOH) for 3 h to remove lignin. Subsequently, the material was treated by 2 wt.% KOH solution for 2 h at 90 °C to remove most hemicelluloses. Finally, the whole mixture was treated $NaClO_2$ at pH 4 to 5 for 2 h, 5 wt.% of KOH at 90 °C for 2 h, and 1 wt.% of HCl solution at 85 °C for 2 h, to obtain purified cellulose. To obtain NFC from the bagasse cellulose, the method of mechanical force step by step refinement was employed. The 1 wt.% NFC suspension was prepared by dispersing the purified cellulose into water. The purified cellulose suspension was poured into a grinder (IKN CMSD2000, Neustadt, Germany) with a rotational speed of 1500 rpm/min and a grindstone gap of -9 for three times to prepare grinding treated NFC suspension (GT). Next, the 1 wt.% NFC suspension prepared by grinding was treated five times in a high pressure homogenizer (SCIENTZ Scientz-150, Ningbo, China) under the condition of an average pressure of 1000 bar to obtain NFC prepared by the combination of grinding and high pressure homogenization treatment (GT-HPH). Finally, the 1 wt.% GT-HPH treated NFC suspension was placed in the ultrasonic cell pulverizer (LAWSON DH-1200E, Ningbo, China) and treated by ultrasonic for 30 min at 900 W to obtain NFC prepared by the combination method of grinding, high pressure homogenization, and ultrasonic waves (GT-HPH-U).

Preparation of NFC Films

The NFC suspensions obtained by different mechanical forces treatment were filtered through a 0.22 μm microporous filter membrane to obtain an NFC wet film. After removal from the vacuum filtration device, the two sides of the NFC wet film were covered with microporous filter membranes and coarse filter papers in turn. The sample was sandwiched between two glass plates with the pressure of 400 N and oven-dried at 80 °C for 24 h to obtain NFC.

Characterisations and Measurements

Morphological properties

The morphology of the NFC was analyzed with a scanning electron microscope (Hitachi S-4800, Tokyo, Japan) after being coated with platinum using sputtering technique to make them conductive. The SEM images were taken at the acceleration voltage of 3 kV and under different magnifications.

The longitudinal surface of fibers was analyzed; the diameter of the NFC was measured from the SEM images using ImageJ software. The diameters of approximately 150 NFC was measured for each sample.

X-ray diffraction analysis

The X-ray diffractometer (Panalytical X `Pert MPD, Almelo, Netherlands) equipped with an X-ray generator (λ = 0.154 nm) and with Cu-Kα radiation (K=1.54056 $\mathring{A}$) was operated over the 2θ range of 5 to 40° with a scan step of 0.02°, at a scan rate of 5 °/min.

The acceleration voltage and current were set to 40 kV and 30 mA, respectively. The degree of crystallinity was measured by empirical peak-height method (Segal *et al*. 1959), as represented in Eq. 1,

$$Cr = \frac{I_{200} - I_{am}}{I_{200}} \times 100 \tag{1}$$

where C_r is the crystallinity, I_{200} is the intensity value for the crystalline cellulose, and I_{am} is the intensity value for the amorphous cellulose. X-Ray diffraction data were analyzed using Origin 8.5 software (Northampton, MA, USA), based on the Gaussian function in order to do de-convolution and find individual peak regions.

Thermogravimetric analysis

Thermogravimetric analysis measurements (TG) were carried out with a Synchronous Thermal Analysis (Mettler TGA/DSC 1/1100, Zurich, Switzerland). The thermal stability of the samples was evaluated at heating rate of 10 °C/min, from 50 to 600 °C. All measurements were performed under a nitrogen atmosphere with a gas flow of 20 mL/min in order to prevent thermoxidative degradation.

FIIR spectral analysis

The FTIR spectra of original cellulose and NFC treated by different treatment methods were measured using a Bruker Optic Tensor 27 spectrometer (Karlsruhe, Germany). The samples were dried at 80 °C under vacuum for 12 h before analysis. The mixture of finely ground NFC and KBr powder at a mass ratio of 1:49 was compressed to form a disc.

All infrared spectra were recorded by an accumulation of 32 scans for each sample, with a resolution of 4 cm^{-1} at 4000 to 400 cm^{-1}. The background spectrum in the absence of any sample was subtracted from the spectra of the individual samples.

Mechanical properties

Tensile tests of specimens were conducted on a universal testing machine (Labthink XLW-PC, Jinan, China) at room temperature. Gauge length was determined in the specimens at 20 mm and widths were 4 mm. The rates for load cell and extension rate were 100 N and 1 mm/min, respectively. The tensile strength and modulus were determined from the obtained stress-strain curves.

RESULTS AND DISCUSSION

Morphological Characterization

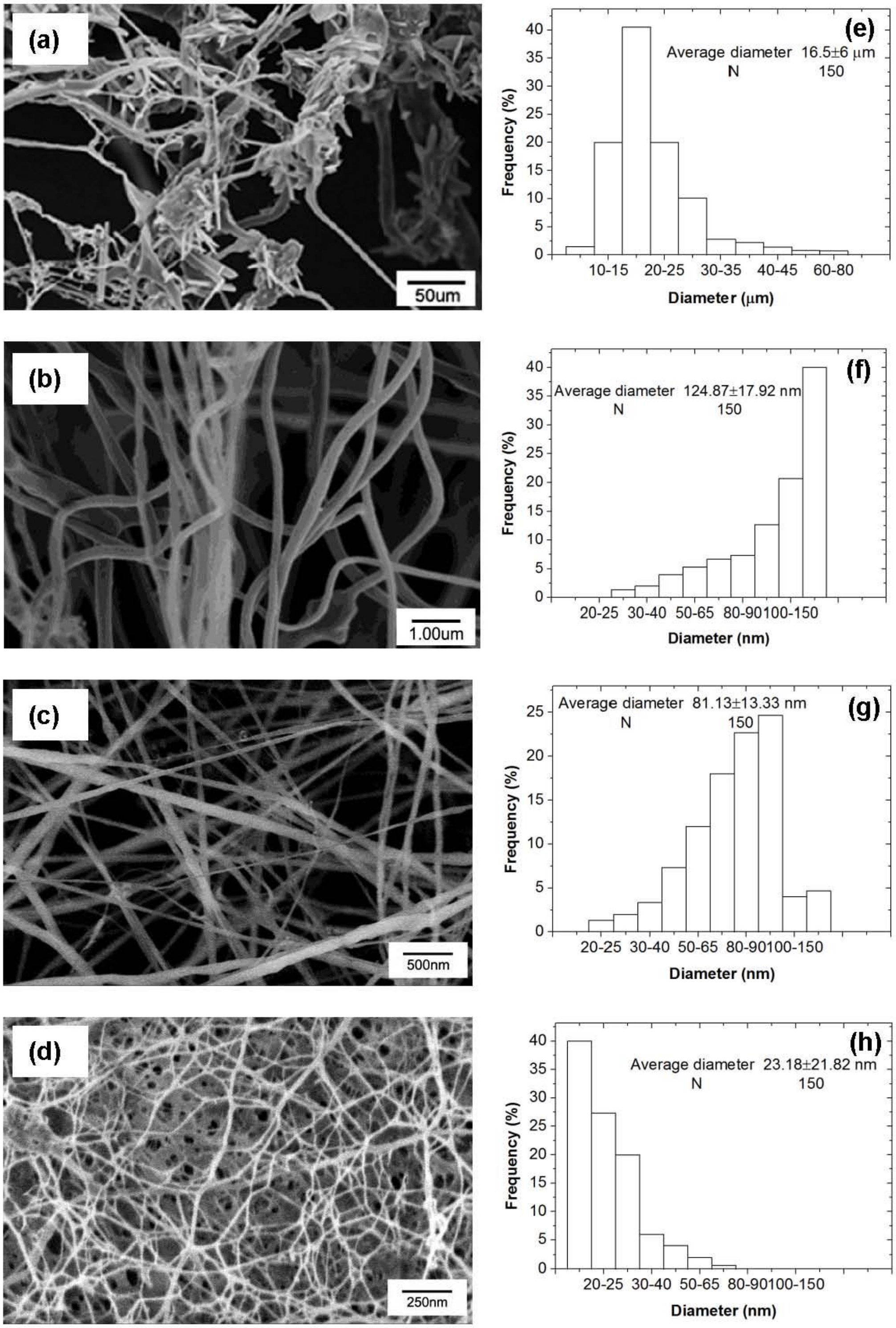

Fig. 1. FE-SEM micrographs of (a) chemical pretreated cellulose, (b) grinding treated NFC, (c) GT-HPH treated NFC, and (d) GT-HPH-U treated NFC. The diameter distribution and number of measured micro- and NFC are shown on Fig. 1 (e), (f), (g) and (h) accordingly.

The structure and strengthening effect of NFC depend on their length, shape, and degree of nanocrystallization (Huq *et al*. 2012). Due to the extensive network *via* numerous intermolecular and intramolecular hydrogen bonds, the aggregated nanofibers should be pretreated by a mechanical force to obtain the homogeneous NFC without destroying in inherent structure (Liu *et al.* 2007). Selected NFC films were analyzed through SEM images to observe morphology and microstructural aspects (Fig. 1 a-h). The NFC in film show a rod-like morphology (Fig. 1 a-d). The diameter distributions of cellulose and nanofiber produced by different mechanical treat method are shown in Fig. 1(e-h). The image in Fig. 1a shows that chemical pretreatment can remove the non-cellulosic impurities on the surface of cellulose and cause partial separation of the celluloses into the micro-sized fiber and a compact structure. Figures 1(b-d) show that the conversion from cellulose to NFC had been realized with the action of mechanical force. Comparing with Fig. 1 a, b and c, it can be found that the degree of nanocrystallization of cellulose increased with the enhancement of mechanical force. With the treatment of HPH on NFC produced by grinding, the aggregated NFC was further converted to refined NFC with an average diameter of 81.13 nm under the high shearing action in the homogenizer. The diameter of most NFC ranged from 60 to 100 nm. Under the treatment of ultrasonic, the diameter of NFC decreased to 23.18 nm on average and the uniformity of NFC was improved. Approximately 40% of the NFC with diameter less than 20 nm, and the NFC with a diameter less than 30 nm accounted for about 90%. Compared with grinding treatment and HPH treatment, ultrasonic treatment can better destroy the amorphous part of cellulose and weaken the hydrogen bonding between cellulose chains.

X-ray Diffraction Analysis

The XRD patterns of NFC treated by chemical pretreatment, GT, GT-HPH, and G-HPH-U are shown in Fig. 2. The diffractograms displayed the same typical cellulose-I crystal structure with the broad peaks at 2θ = 15° corresponding to the (110) crystallographic plane, and a sharp peak at 22.6° corresponding to the (200) crystallographic plane.

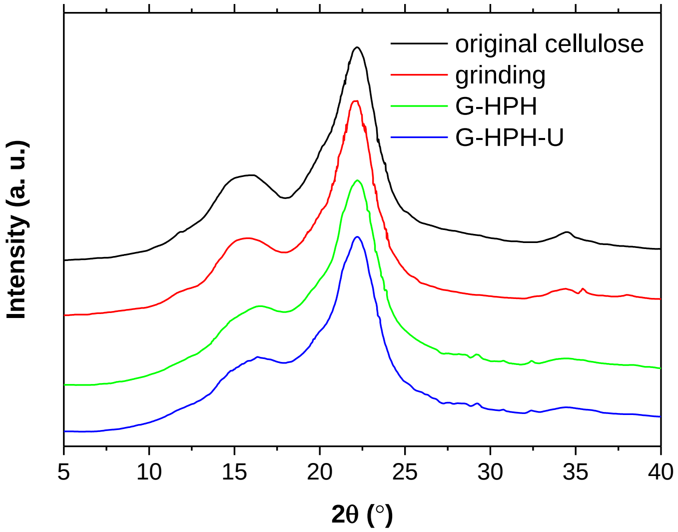

Fig. 2. X-ray diffraction pattern of (a) chemical pretreated cellulose, (b) GT treated NFC, (c) GT-HPH treated NFC, and (d) GT-HPH-U treated NFC

In all cases, the cellulose-I crystal structure was maintained in the mechanically treated NFC. This result indicates that the treatments such as grinding, high pressure homogenization, and ultrasonic had no obvious influence on the crystal structure of natural cellulose. By contrast, the degree of crystallinity of NFC was affected greatly by the mechanical treatment. In the process of GT, GT-HPH, and GT-HPH-U treatment on cellulose, these forces caused cleavage of the crystallites and an increase in non-crystalline parts (Ahmadzadeh *et al*. 2018). The crystallinity indexes of GT-, GT-HPH-, and GT-HPH-U- treated NFC were 64.2%, 61.7%, and 60.8%, respectively, which is much higher than about 45% of original cellulose and lower than 65% of chemical pretreated cellulose. This indicates that the breakage of the intermolecular hydrogen bonds in NFC caused the collapse of crystal structure and the emergence of amorphous region during mechanical treatment.

Thermostability Analysis

The effect of structural properties on the thermal degradation of cellulose can be evaluated using the thermogravimetry technique; because the different polymers with various structures behave differently when undergo thermal degradation (Ahmadzadeh *et al.* 2018). The TGA curves of chemical pretreated cellulose and NFC are shown in Fig. 3. As can be seen, the onset decomposition temperature of original cellulose was 210°C. By contrast, the thermostability of NFC produced by grinding, GT-HPH, and GT-HPH-U treatments were increased. Due to the thermal decomposition of aromatic hydrocarbons in residual lignin, the chemical pretreated bagasse cellulose degraded obviously at the initial stage of heating. Rapid quality degradation occurred in the range of 250 °C to 380 °C, and the weight loss rate reached 86% when heated to 600 °C. Correspondingly, NFC produced by grinding, GT-HPH, and GT-HPH-U treatments exhibited the high thermal stability with the onset decomposition temperature of near 300 °C. When heated to 600 °C, the thermal weight loss rates of the samples produced by GT, GT-HPH, and GT-HPH-U treatments were 69.2%, 73.3%, and 74.4%, respectively. This indicated that the destruction of crystallization in NFC caused the decrease of thermal stability under the shear action of mechanical forces, which is not significant.

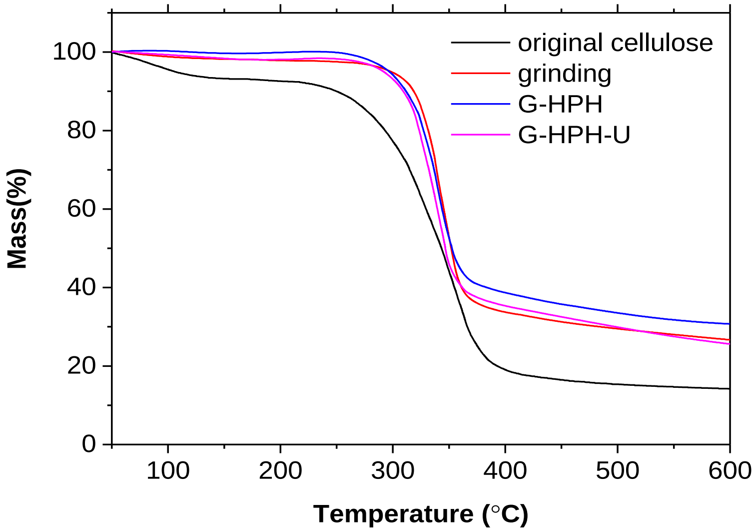

Fig. 3. TGA curves of (a) chemical pretreated cellulose, (b) GT treated NFC, (c) GT-HPH treated NFC, and (d) GT-HPH-U treated NFC

FTIR Analysis

The FTIR characteristic peaks assigned to the functional groups identify the presence or removal of these components (Ahmadzadeh *et al.* 2018). FTIR spectra of chemically pretreated cellulose and NFC are shown in Fig. 4.

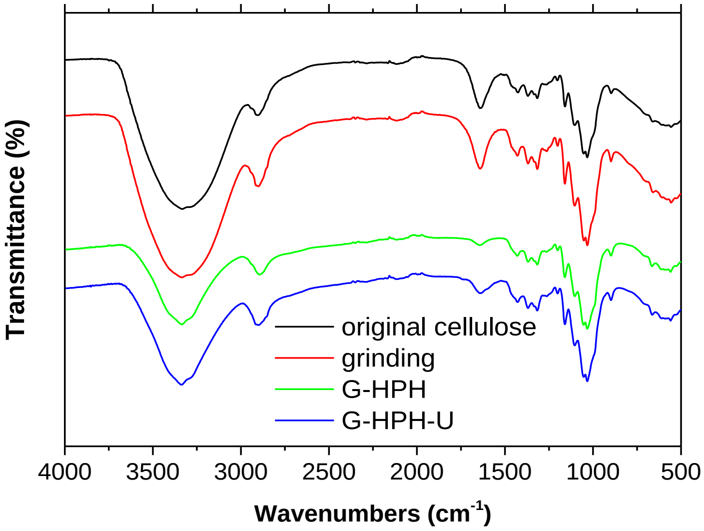

Fig. 4. IR spectra of (a) chemical pretreated cellulose, (b) GT treated NFC, (c) GT-HPH treated NFC, and (d) GT-HPH-U treated NFC

The dominant peaks of OH-stretching and CH-stretching at approximately 3334 cm^{-1} and 2900 cm^{-1} were observed in the spectrum of all specimens. The absorbance band attributed to the stretching vibration of C=O appeared at around 1640 cm^{-1}. The shear vibration of $–CH_2$ appeared as an absorption peak at 1420 cm^{-1}. The absorption band at around 1023 cm^{-1} is related to the stretching vibration of C-O in the alcohol hydroxyl groups of cellulose. The absorption peak near 895 cm^{-1} is related to the C-H deformation vibration of cellulose. The above absorption peaks related to cellulose are all observed in the FTIR spectra of chemical pretreated cellulose and GT-, GT-HPH- and GT-HPH-U-treated NFC. Under the action of mechanical pretreatment, hemicellulose and lignin are almost removed, and highly purified NFC is produced (Ghaderi *et al.* 2014). Compared with the FTIR spectrum of chemical pretreated cellulose, the absorption peak located at 1731 cm^{-1} related to the stretching vibration of C=O on the xylan acetyl group $CH_3C=O$ in hemicellulose and the absorption peak at 1460 cm^{-1} related to the bending vibration of C-H on the $–CH_2$ of lignin disappeared in the FTIR spectra of NFC. Two absorption bands at 1640 cm^{-1} and 1508 cm^{-1} are related to the stretching vibration peak of carbonyl C=O in the side chain of lignin and the vibrational absorption peak of benzene ring skeleton, respectively. These two bands were detected in the spectrum of chemical pretreated cellulose; however, their intensity was reduced in the spectra of NFC. There were no obvious differences in FTIR spectrum among NFC, which indicates that no other chemical reaction occurred during the processes of cellulose pretreated by grinding, high pressure homogenization, and ultrasonication.

Mechanical Property Analysis

Figure 5 shows the effect of mechanical pretreatment on tensile strength of NFC. The mechanical performances of NFC films strongly depend on the size and morphology of their corresponding constituent NFC, which can be controlled by the mechanical treatments (Liu *et al.* 2020).

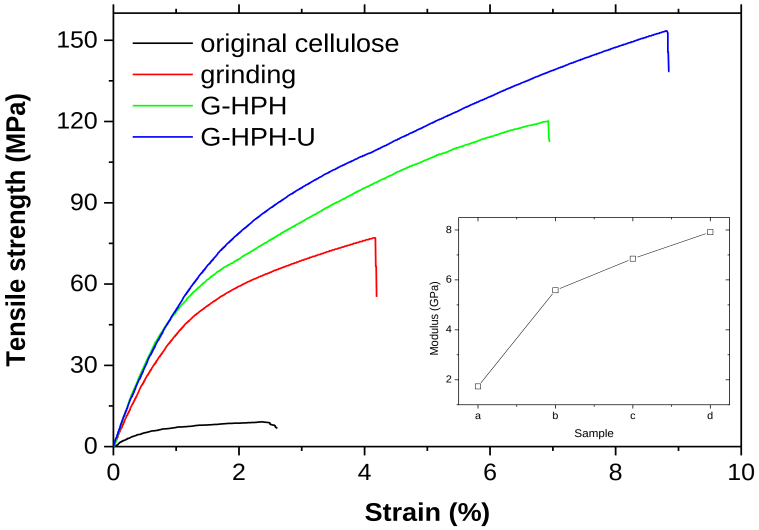

Fig. 5. Stress-strain curves and modulus curve of (a) chemical pretreated cellulose, (b) GT treated NFC, (c) GT-HPH treated NFC, and (d) GT-HPH-U treated NFC.

The tensile strength of NFC films increased with the increase of mechanical force. Compared with the chemical pretreated cellulose fiber, the tensile strength and the strain at break of NFC film treated by grinding increased by 765% and 64.2%, and reached 77 MPa and 4.18%, respectively. Under the shear action of HPH treatment, the aspect ratio and the size of NFC were further adjusted. Moreover, the tensile strength and strain at break of NFC treated by GT-HPH-U increased to 153.6 MPa and 8.83%, respectively. These results indicate that the strong mechanical force made the diameter distribution of NFC more uniform, the diameter smaller, and the specific surface area larger, which is verified by SEM in Fig. 1. Compared with the action of grinding process and HPH process, ultrasonic treatment can further individualized NFC (Nuruddin *et al.* 2011). A large number of hydroxyl groups on the NFC surface form a strong hydrogen bond between the adjacent fibers, which improves the interfacial bonding between the nanofibers (Ahmadzadeh *et al.* 2018). Under the action of HPH and ultrasonic treatment, the crystal region of NFC is partially destroyed, which is helpful to improve the elongation at break of NFC. The network structure formed by the interweaving of NFC and the uniformly distributed diameter reduce the stress concentration of NFC, which makes the NFC film have excellent mechanical properties. The prepared NFC can be used as reinforcement materials and matrix materials to provide good mechanical support for the matrix.

CONCLUSIONS

1. Nanofibrillated cellulose (NFC) from sugarcane bagasse was prepared by grinding, high pressure homogenization, and ultrasonication coupled with simple chemical pretreatment. Compared with the complex chemical treatment, the grinding-high pressure homogenization-ultrasonic process is more convenient in the preparation of NFC, which resulted in NFC with the average diameter of 23.18 nm and higher thermal stability and crystallinity.
2. A very low-value agricultural waste product can be converted to a high performance NFC with tensile strength of 153.6 MPa and strain at break of 8.83%.
3. Under the action of mechanical force produced by GT-HPH-U, the multilayer structure of the cellulose was destroyed, and NFC with small diameter, uniform distribution, and large specific surface area was obtained. The strong hydrogen bonding interaction produced by a large number of hydroxyl groups on the surface of the nanofiber and the decrease of stress concentration caused by the uniformly dispersed fiber network structure greatly enhanced the mechanical properties of the NFC and improved the crystallinity and thermal stability of the fiber. By using the ultrasonic treatment, part of the cellulose crystal region was further destroyed, which increased the elongation at break while maintaining the high tensile strength of NFC, thus expanding the application range of NFC.

ACKNOWLEDGMENTS

The authors are grateful for the support of the Anhui Provincial Natural Science Foundation, Grant No. 1908085ME154; and the Open Fund of Fujian Universities and Colleges Engineering Research Center of Soft Plastic Packaging Technology for Food, Grant No. G1-KF1708.

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Article submitted: April 25, 2020; Peer review completed: June 4, 2020; Revised version received and accepted: July 4, 2020; Published: July 9, 2020.
DOI: 10.15376/biores.15.3.6636-6647