

Extraction and characterization of holocellulose fibers by microwave-assisted selective liquefaction of bamboo

Jiulong Xie,^{1,3} Chung-Yun Hse,² Todd F. Shupe,³ Hui Pan,⁴ Tingxing Hu¹

¹Department of Wood Science, College of Forestry, Sichuan Agricultural University, Chengdu, Sichuan 611130, China

²Southern Research Station, USDA Forest Service, Pineville, LA, 71360

³Louisiana Forest Products Development Center, School of Renewable Natural Resources, Louisiana State University Agricultural Center, Baton Rouge, LA, 70803

⁴College of Chemical Engineering, Nanjing Forestry University, Nanjing, Jiangsu 210037, China

Correspondence to: H. Pan (E-mail: hpan@njfu.edu.cn)

ABSTRACT: Microwave-assisted selective liquefaction was proposed and used as a novel method for the isolation of holocellulose fibers. The results showed that the bamboo lignin component and extractives were almost completely removed by using a liquefaction process at 120 °C for 9 min, and the residual lignin and extractives in the solid residue were as low as 0.65% and 0.49%, respectively. Increasing the reaction temperature or time could decrease the solid yield, but they can also enhance the removal of lignin and extractives from bamboo particles and increase the holocellulose content in the solid residue. The absorbance bands that characterized functional groups of lignin on the Fourier transform infrared spectra of the solid residue weakened or disappeared. The solid residue showed high crystallinity, indicating the removal of noncellulosic material. Small cracks were observed on the SEM images of the residue, which indicated that the fibers from liquefaction may be susceptible to chemical access or enzyme attack. © 2016 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* **2016**, *133*, 43394.

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INTRODUCTION

Global concerns regarding fossil fuel depletion and associated adverse environmental impacts have encouraged research in renewable biofuels.¹ Moreover, the demand for environmentally friendly products has renewed interest in finding new biobased materials that are biodegradable and environmentally compatible.² Lignocellulosic biomass represents a renewable, abundant, and inexpensive source of raw materials for the chemical industry to develop biofuels, renewable chemicals, and biomaterials. Conversion of abundant lignocellulosic biomass to ethanol as a transportation fuel provides an important opportunity to improve energy security, rural development, and environmental stewardship.

Cellulosic fibers isolated from lignocellulosic biomass can be used as reinforcing elements in polymeric matrices and are considered highly promising fillers in preparing sustainable composite materials. During the bioconversion of lignocellulosic biomass to biofuels via enzymatic hydrolysis, the carbohydrate component needs to be exposed to enzymatic action, and the lignin in the biomass acts as a barrier and hinders the access of

the enzymatic molecules to the cellulose.³ Thus, lignin removal is necessary. In addition, to generate natural cellulose-enriched fibers or microfibrillated cellulose (MFC), wood should be treated by various chemical processes to remove lignin and other substances before it is used as a raw material for the production of MFC.⁴ Moreover, in the manufacturing of wood-plastic composites (WPC) by Ou *et al.*,^{5,6} the removal of lignin and extractives in wood flour could increase the tensile modulus. Therefore, large-scale production of holocellulose fibers is necessary for paper pulping, bioconversion, MFCs, and high-performance biobased composites industries, and thus an efficient pretreatment process of lignocellulosic biomass is required.

In recent decades, a number of different pretreatment processes, such as acidic or alkaline pretreatment,⁷ wet oxidation,⁸ liquid hot water,⁹ steam explosion,¹⁰ and ultrasound¹ have been applied in the treatment of different lignocellulosic biomass materials. However, to achieve industrial-scale production, these pretreatment techniques will need to become more efficient, inexpensive, and environmentally friendly.

Glycerol, a high-boiling-point solvent, is presently the main byproduct of the oleo chemicals industry and can be as high as

10% of the total biodiesel production.⁸ With the increase in global biodiesel production, glycerol production has rapidly moved into oversupply, resulting in a crash in the price for crude glycerol. Thus, the commercial utilization of this compound has become a challenge. It is, therefore, desirable to find a novel, economical utilization method for crude glycerol to improve the economics of biodiesel production. Recent advancements in techniques for treatment of woody materials using organic solvents have stimulated research on the use of glycerol pulping as a potential utilization approach. Several studies have been conducted on the pretreatment and delignification of biomass using glycerol.¹¹ Although study of lignocellulosic biomass pretreatment using glycerol for enzymatic hydrolysis or renewable chemicals has shown encouraging results, an economically viable technology is yet to be realized because of the high cost and time-consuming nature of the biomass pretreatment process.

Compared to conventional heating, microwave energy penetrates and produces a volumetrically distributed heat source: heat is generated throughout the material and leads to faster heating rates and improved kinetics. Therefore, the development of a reaction process that combines the greater microwave heating efficiency and the byproduct glycerol from biodiesel production to replace petroleum-based solvents is a promising method to enhance the economic viability of the process and benefit agricultural producers, biobased processing industries, and the environment. Based on the potential benefits of microwave heat, we developed a new microwave-assisted liquefaction system using glycerol as the main solvent.^{12–15} In the investigation on the liquefaction behavior of bamboo, lignin in bamboo could be easily decomposed in glycerol with sulfuric acid as catalyst using microwave energy under mild reaction conditions. Moreover, the addition of methanol in glycerol has been proven effective in avoiding recondensation reactions of lignin in liquefaction of bamboo, and the benefit of using methanol in liquefaction is that it can be easily recovered after liquefaction. Moreover, in the decomposition of lignin, the methanol —OH group functions as the nucleophile and attacks electrophilic C adjacent to the glycosidic bond in the liquefaction system, creating good leaving groups and a neutral hydroxyl group by cleaving the C—O bond.¹²

From this point of view, we proposed that this microwave-assisted liquefaction system could also be applied in biomass main component separation if the reaction conditions are properly controlled. Accordingly, in this study, microwave-assisted selective liquefaction was introduced for the production of holocellulose fibers to enhance the paper pulping, bioethanol, and biomaterials industry. The aim of the research was to achieve a rapid process for the production of holocellulose fibers. The specific objective of this study was to evaluate the effect of reaction temperature and time on the physicochemical properties of the extracted holocellulose fibers.

EXPERIMENTAL

Materials

Three-year-old bamboo culms (*Phyllostachys pubescens*) were harvested from the Kisatchie National Forest, Pineville, LA. The bamboo culms were reduced to particles using a Thomas Wiley

(Swedesboro, New Jersey, USA) laboratory mill. The particles were screened to collect particles that passed through a 20-mesh sieve and then retained on a 40-mesh sieve and then dried to a constant weight in an oven maintained at 80 °C. The dried particles were stored in polyethylene bags and used without further treatment. All acids, glycerol, and methanol used were of reagent grade and obtained from commercial sources.

Microwave-Assisted Selective Liquefaction

Selective liquefaction of bamboo was carried out in a Milestone (Shelton, Connecticut, USA) MEGA laboratory microwave oven. Mixed glycerol and methanol at a ratio of 2/1 (w/w) was used as the solvent in a solvent-to-bamboo ratio of 4/1 (w/w). Sulfuric acid (1.75% of solvent weight) was used as the catalyst. A typical reaction mixture consisting of 2 g of bamboo particles, 8 g of solvent, and 0.14 g of sulfuric acid was loaded in 100 mL Teflon vessels with a magnetic stirring bar. The Teflon vessels were then placed on the rotor tray inside the microwave cavity. The temperature was monitored using an ATC-400FO (Shelton, Connecticut, USA) automatic, fiber optic temperature-control system. Based on the monitored temperature, the output power was auto-adjusted during liquefaction. In this study, the temperature was increased from room temperature to 120 °C and then was kept constant for 4 min, unless otherwise noted. At the end of the reaction, the vessels were taken from the microwave cavity and cooled for 15 min. Afterward, the resulting reaction mixtures were dissolved in methanol and then vacuum-filtered through Whatman No. 4 filter paper. The solid retained on the filter paper, which was designated as holocellulose fibers in this study, was washed with methanol and oven-dried at 105 °C. The yield is the mass percentage of the residue solid to the original bamboo and was calculated as follows:

$$\text{Yield (\%)} = \frac{\text{weight of solid residue}}{\text{weight of raw material}} \times 100 \quad (1)$$

Preparation of Jeffrey's Solution Fiber

Complete bamboo fiber was obtained using Jeffrey's solution in order to have a comparison with the fiber obtained by microwave liquefaction. The Jeffrey's solution fiber was used as a control because it keeps the original structure of the fiber and is usually used for fiber morphology analysis.¹⁶ Bamboo samples were softened in hot water for 3 h, then bamboo splints were macerated in a binary solution of chromic acid and nitric acid (10% chromic acid and 10% nitric acid mixture of 1:1). The mixture was maintained at 50 °C for 1 h. Thereafter, the macerated splints were washed carefully with distilled water. The splint fibers were air-dried and then collected for further use.

Chemical Component Analysis

The holocellulose, Klason lignin, and alcohol-toluene extractive of the samples were determined according to ASTM standards D 1104-56, D 1106-96, and D 1110-96, respectively.^{17–19} Each test was conducted in duplicate, and the average value was reported.

Fourier Transform Infrared Spectra

The Fourier transform infrared (FTIR) analysis was performed by a Nicolet (Ramsey, Minnesota, USA) Nexus 670 spectrometer equipped with a Thermo Nicolet Golden Gate MKII Single

Table I. Yield and Chemical Composition of Solid Residue from Different Liquefaction Conditions

Sample	Yield (%)	Holocellulose (%)	Lignin (%)	Extractives (%)
Control	100 ± 0.00	63.08 ± 1.03	20.91 ± 0.24	7.24 ± 0.29
75 °C/7 min	81.44 ± 1.55	65.79 ± 1.32	14.60 ± 0.28	4.75 ± 0.89
90 °C /7 min	65.75 ± 1.77	71.07 ± 1.29	11.31 ± 0.48	2.60 ± 0.13
105 °C /7 min	53.52 ± 1.90	81.28 ± 1.35	4.87 ± 0.53	2.13 ± 0.21
120 °C /7 min	42.44 ± 2.17	93.00 ± 1.73	1.66 ± 0.15	0.59 ± 0.04
3 min/120 °C	59.35 ± 1.95	78.34 ± 1.14	7.44 ± 0.61	2.26 ± 0.19
5 min/120 °C	45.95 ± 1.06	86.64 ± 1.33	3.82 ± 0.05	1.07 ± 0.03
9 min/120 °C	37.85 ± 1.33	94.37 ± 0.48	0.65 ± 0.27	0.49 ± 0.05

Reflection ATR accessory. Data collection was performed with a 4 cm^{-1} spectral resolution, and 32 scans were taken per sample.

X-ray Diffraction Analysis

The crystallinity of the original bamboo and solid residue samples was analyzed using wide-angle X-ray diffraction (Bruker (BillERICA, Massachusetts, USA) D 5000). The data were generated by a diffractometer with Cu K α radiation ($\lambda = 1.54\text{ \AA}$) at 40 kV and 30 mA over the angular range $2\theta = 5\text{--}40^\circ$ and a step time of 2.0 s. A focusing powder diffraction method was applied. The crystallinity index (CrI) is determined using the following equation:

$$\text{CrI (\%)} = \frac{I_{002} - I_{\text{am}}}{I_{002}} \times 100 \quad (2)$$

where I_{002} is the intensity of the diffraction from the (200) plane at $2\theta = 22.1^\circ$, and I_{am} is the intensity for amorphous material taken at $2\theta = 18^\circ$.²⁰

Morphology Analysis

The surface morphology of the bamboo particles and solid residue samples were observed using scanning electron microscopy (SEM, JCM-5000). Test samples were coated with gold using a vacuum sputter coater before being subjected to the SEM analysis.

Thermal Stability Analysis

The thermogravimetric and differential thermogravimetric (TG/DTG) analysis was conducted with a thermal analyzer Q50 TGA (TA Instruments, New Castle, DE) to simultaneously obtain thermogravimetric data. About 2 mg of sample was analyzed by the thermal analyzer. Pyrolysis was terminated at 800 °C with a heating rate of 20 °C/min under a flow of 60 mL/min of nitrogen gas.

Gas Chromatograph/Mass Spectrometer (GC-MS)

The general profile for the liquefied liquor was obtained using electron-ionization mass spectrometry (EIMS). Analysis of the product was conducted on a mass spectrometer (Agilent (Santa Clara, California, USA) 5975C VL MSD), and the products were separated into their components using a gas chromatograph (Agilent 7890A) equipped with a fused capillary column (DP-5 (Santa Clara, California, USA), $L = 30\text{ m}$, i.d. 0.32 mm, film thickness 0.25 μm) with 5% phenyl and 95% dimethylpolysiloxane as the stationary phase. The carrier gas was helium at a flow rate of 1.8 mL/min. The conditions for the analysis were as follows: injection mode was split at split rate 35; the column was held at 50 °C for 2 min and then heated to 250 °C at the rate of 10 °C/min, and

the injector temperature was 250 °C. The identification of the product components was confirmed using total ion chromatograms as well as fragmentation patterns.

Statistical Analysis

The statistical analysis was carried out using SAS software (version 9.1, SAS Institute, Cary, NC). Analysis of variance (ANOVA) was performed to determine the significant difference ($\alpha = 0.05$) among the different samples.

RESULTS AND DISCUSSION

Microwave-Assisted Selective Liquefaction

The yield of solid residue with respect to reaction temperature and time is presented in Table I. As shown in Table I, the yield dramatically decreased from 81.4 to 39.65% as the reaction temperature increased from 75 to 120 °C, indicating that the increase in temperature combined with the microwave heating showed significant ability to enhance the decomposition of bamboo substances. In the research on selective liquefaction of wheat straw in phenol using conventional heating, the reaction conditions for obtaining about 40% of liquefied wheat straw were 125 °C, 60 min, and a sulfuric acid loading of 3%.²¹ For comparison, the introduction of microwave energy in selective liquefaction of bamboo allowed the temperature to be lower and shortened the reaction time, and it reduced sulfuric acid usage. This may be because microwave irradiation intensified the heat and mass transfer during the reactions. The highly efficient liquefaction of this process may also be due to the fact that extractives and lignins in bamboo could be easily liquefied under lower reaction temperatures with microwave irradiation.

A selective liquefaction of bamboo using conventional liquefaction was conducted in a Parr (Moline, Illinois, USA) reactor in order to have a rough comparison in energy demands with microwave selective liquefaction. The reaction temperature and time for a solid residue yield similar to the microwave liquefaction process of 120 °C and 9 min were 120 °C and 35 min for conventional liquefaction. A referenced method was used to estimate the energy requirement for the conventional liquefaction.²² The heating power of the Parr reactor was 1500 W at the maximum temperature of 400 °C, and the power required to maintain the temperature of 120 °C was determined to be 450 W (120/400 multiplying 1500). The energy requirement was calculated as the power multiplied by time and determined to be $9.45 \times 10^5\text{ J}$. Since microwave power was automatically adjusted based on

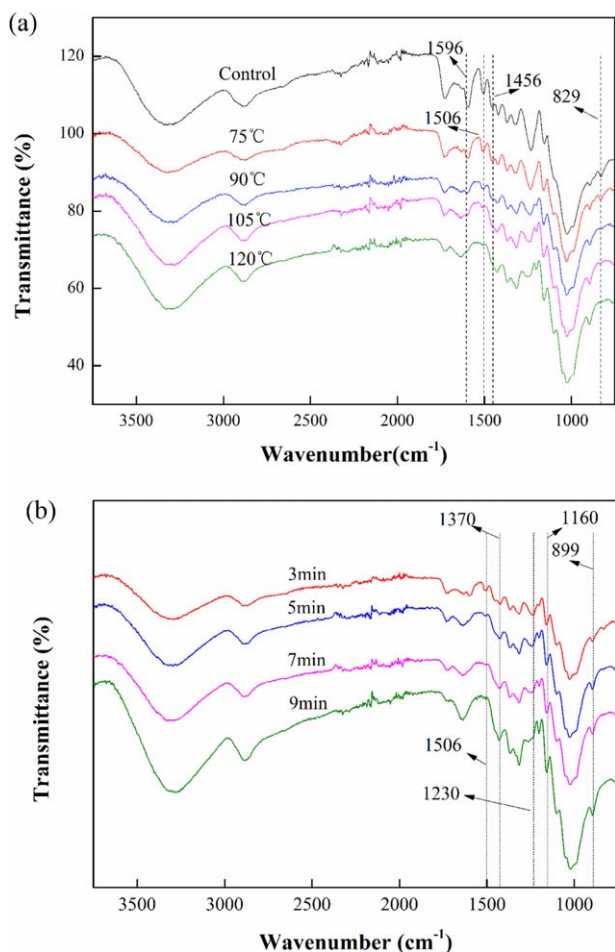


Figure 3. FTIR spectra of solid residues from microwave selective liquefaction (a) temperature (time, 7 min) and (b) reaction time (temperature, 120 °C). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

(*p*-)hydroxycinnamic acids and lignin in the fibers.¹⁰ The dissociation of the key ester linkages between lignin and carbohydrates promoted the dissolution of lignin into the solvents. This result was in accord with the results of the GC–MS analysis.

The absorbances at 1596 cm⁻¹ and 1506 cm⁻¹ arising from the aromatic skeletal vibration, the absorbance at 1456 cm⁻¹ assigned to C–H deformation combined with aromatic ring vibration, and the band at 1230 cm⁻¹ corresponding to methoxyl groups of lignin were all shown to be strong peaks in the spectra of the bamboo particles. These peaks were weakened in the spectra of the solid residue samples at 75, 90, and 105 °C. As the temperature increased to 120 °C, the absorbance bands at 1596, 1456, and 1230 cm⁻¹ disappeared, and the band at 1506 cm⁻¹ became a small shoulder. This result indicates that lignin functional groups such as aromatic rings were almost completely dissociated and dissolved with only a small amount of associated lignin retained on the solid residue (indicated by the band at 1506 cm⁻¹), which was consistent with the wet chemistry analyses discussed above.

The spectra of the solid residue as a function of reaction time are shown in Figure 3(b). By prolonging the reaction time, the absorbance bands assigned to lignin decreased (3–7 min). By fur-

ther prolonging the reaction time to 9 min, the absorbance bands that characterized functional groups of lignin disappeared on the spectrum of the solid residue, leaving visible intensity bands assigned to carbohydrates. This was in good agreement with the holocellulose content for the solid residues, which was more than 94% composed of holocellulose. The bands for different functional groups of carbohydrates, such as the methylene groups at 1420 and 1371 cm⁻¹, the C–O band at 1162 cm⁻¹ and 1108 cm⁻¹, and CH deformation at 899 cm⁻¹, became much more intensive in the spectra of the solid residues from different liquefaction conditions, which also gave evidence of the high holocellulose content in the solid residues (88–94.28%).

The ratio of relative intensities of the three characteristic absorbance bands of holocellulose (1370, 1160, and 899 cm⁻¹) to the two characteristic absorption ones of lignin (1506 and 1230 cm⁻¹) were calculated to further clarify the chemical changes during the liquefaction process. As shown in Figure 4, the ratio increased by prolonging the reaction time, revealing the decrease in lignin content and increase in holocellulose content. It was interesting to find that good linear correlations existed between the ratio of band intensities of the

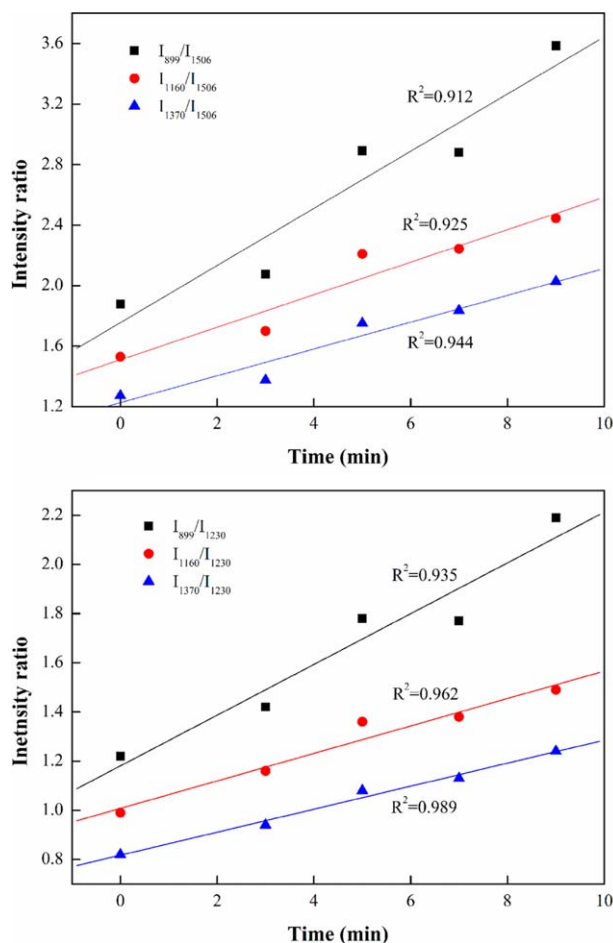


Figure 4. Relationship between FTIR intensity ratio of solid residues and microwave selective liquefaction time. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

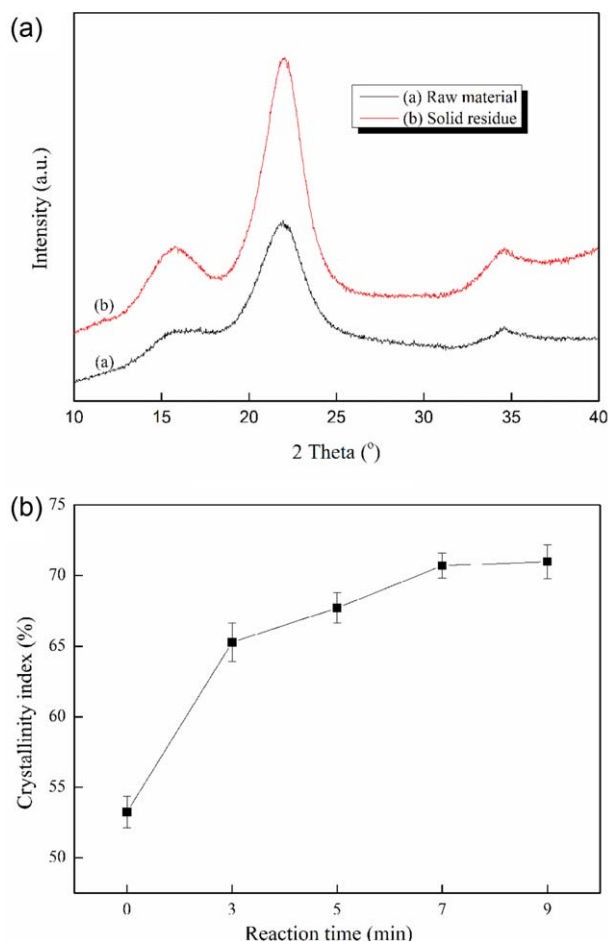


Figure 5. (a) Typical X-ray diffraction spectra of original bamboo, solid residue, and (b) effect of reaction time on the crystallinity value of solid residue from the microwave liquefaction process at 120 °C. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

holocellulose/lignin (I_{899}/I_{1506} , I_{1160}/I_{1506} , I_{1370}/I_{1506} , I_{899}/I_{1230} , I_{1160}/I_{1230} , and I_{1370}/I_{1230}) and reaction time ($R^2 = 0.91-0.99$).

Crystallinity Analysis

Figure 5(a) shows the wide-angle X-ray diffraction (XRD) pattern of the original bamboo and the solid residue from 120 °C and 9 min. Both of the XRD patterns displayed peaks at $2\theta = 14.9$ and 22.1° , indicating the typical cellulose I crystalline structure. The calculated crystallinity index (CrI) values were 52.3 for the original bamboo.²⁰ After liquefaction, the CrI value was significantly increased. With increasing reaction time, the CrI value displayed an increasing trend and reached a maximum value for the solid residue from 120 °C, 9 min [Figure 5(b)]. This significant increase of crystallinity in the solid residue in comparison to the original bamboo was undoubtedly attributed to the efficient dissolution and removal of noncellulosic materials such as lignin and extractives from the amorphous regions and enrichment in cellulose content in the solid residue during microwave liquefaction. This was in good agreement with the chemical results and FTIR analysis shown in Table I and Figure 3, respectively. This result further confirmed the efficient

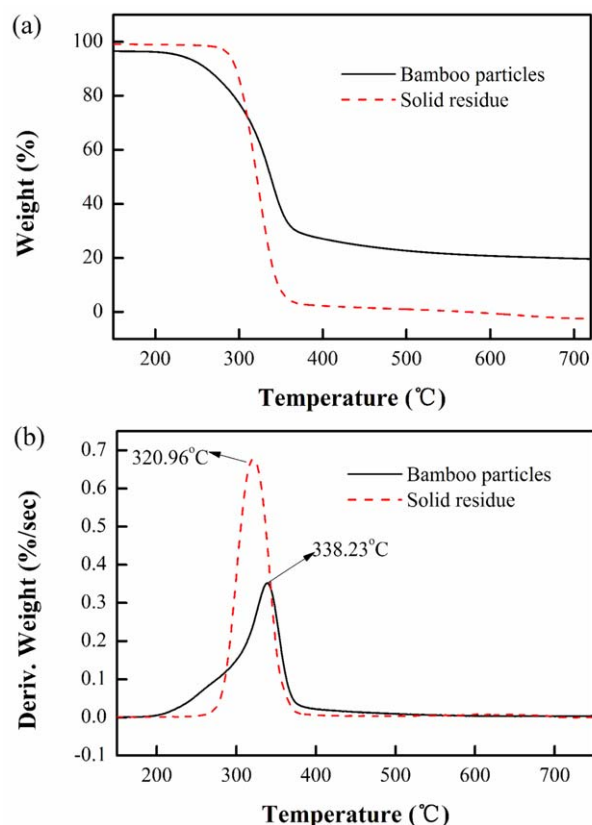


Figure 6. (a) TG and (b) DTG curves of bamboo particles and solid residues from microwave selective liquefaction process at 120 °C for 9 min. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

removal of noncrystalline components and that microwave selective liquefaction could be used for prepurification of lignocellulosic biomass for cellulose fiber production.

Thermal Stability Analysis

Figure 6 presents the thermal degradation curves of the original bamboo particles and the solid residue from 120 °C, 9 min. As shown in Figure 6, the samples showed a more significant weight loss below 200 °C. A slight shoulder was observed on the TG curve of bamboo particles but was absent on the TG curve for the solid residue. This may be attributed to the removal of extractives and degradation of a portion of hemicellulose during the microwave liquefaction process, which was also indicated by the weakened absorbance band at 1735 cm^{-1} assigned to hemicellulose on the FTIR spectrum of holocellulose fibers. The onset temperature, T_o , is considered as the

Table II. Thermal Degradation Temperatures for the Original Bamboo Particles and Solid Residue from 120 °C and 9 min

Sample	T_o (°C) Initial weight loss	T_1 (°C) Shoulder	T_2 (°C) DTG peak
Bamboo particles	220.21	280	320.96
Solid residue	288.61	— ^a	338.23

^aNo shoulder was observed.

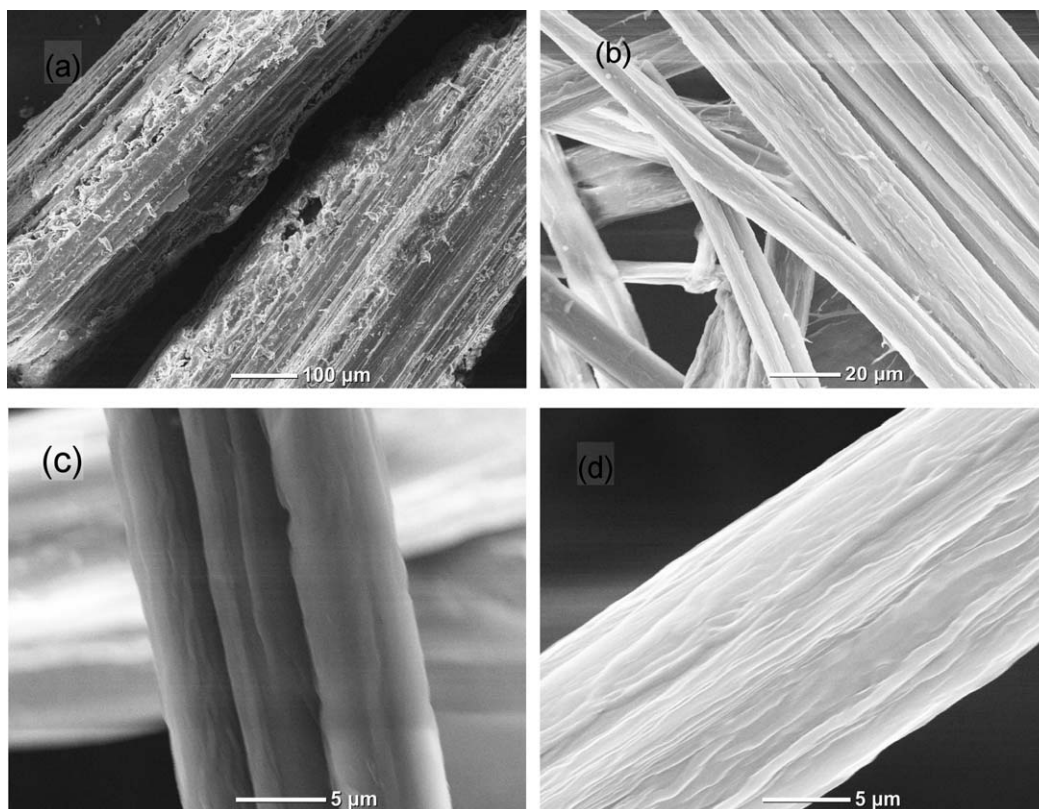


Figure 7. SEM images of (a) bamboo particles, (b) split fasciculus, (c) single fiber from microwave selective liquefaction at 120 °C for 9 min, and (d) Jeffrey's solution fiber.

temperature at which the sample loses 5% of its weight. The T_5 value for the bamboo particles was particularly lower than that for the solid residues (Table II). It has been reported that extractives could promote ignition of woody materials at lower temperature.²³ Thus, in this study, the removal of extractives during the liquefaction process contributed to the higher initial weight loss temperature, and the extractive component in the bamboo particles accelerated the degradation process of the other components, resulting in a lower initial weight loss temperature. Prominent peaks corresponding to the maximum decomposition rate were observed on both the bamboo particles and the solid residues. The DTG peak temperature for the bamboo particles was about 20 °C higher than that for the solid residue (320.96 and 338.23 °C for bamboo particles and solid residue, respectively). As for the three components of woody materials, hemicellulose was the easiest to pyrolyze, followed by cellulose then lignin based on TG analysis.²⁴ Consequently, the lower DTG peak temperature for the solid residue was mainly due to the removal of lignin by the microwave liquefaction process. The result obtained in this study was also in agreement with the results of Chen *et al.*,²⁵ who

reported that liquefaction could significantly decrease the maximum degradation rate temperature (temperature at DTG peak). The differences in thermal stability between the bamboo particles and liquefied bamboo solid gave evidence of the efficient removal of lignin and extractives by this liquefaction process.

SEM Images

The SEM images of bamboo particles and the solid residue samples from 120 °C within 9 min are shown in Figure 7. The bamboo particles [Figure 7(a)] showed large bundles with many small fragments and rough surfaces. As can be seen in Figure 7(b), microwave-assisted selective liquefaction led to split fasciculus with smooth surfaces, indicating that lignin components in the middle lamella were completely removed, resulting in the collapse of the compact structure of bamboo. Figure 7(c) shows small cracks 1–2 μm inwidth on the exterior surface. Jeffrey's solution fibers [Figure 7(d)] were prepared in order to verify the specific character of the fibers obtained in this study, and no cracks were observed. The Jeffrey's solution fibers were used as a control because fibers obtained by this method retain their

Table 3. Length, Width, and Aspect Ratio of Original Bamboo Particles and Solid Residue from 120 °C and 9 min

Sample	Dimension		
	Length (μm)	Width (μm)	Aspect ratio
Bamboo particle	2312.01 ± 594.99	567.67 ± 162.36	4.08 ± 1.77
Solid residue	891.19 ± 256.43	17.02 ± 6.93	65.08 ± 10.44

original structure and show little damage. This comparative observation indicated that fibers isolated from the microwave liquefaction process may be more susceptible to chemical access or enzyme attack. One possible explanation for the cracks on the fibers from liquefaction is the physical effect of the microwave treatment on the properties of lignocellulosic materials. Studies have shown that microwave pretreatment can open pathways in wood, dramatically increase permeability, and accelerate moisture migration in wood.^{26,27} Thus, the cracks observed may be created by the solvent-absorption behaviors induced by microwave energy heating during the liquefaction process. Moreover, the physical changes in wood properties with microwave energy facilitate favorable conditions for the solvents to react. Accordingly, the changes in wood properties resulting in the uniform distribution of solvents along with the uniform temperature distribution induced by microwave energy heating also enhanced the bamboo chemical component degradation.

The dimensions of the original bamboo and liquefied solid residues from 120 °C, 9 min were also evaluated, and the results are listed in Table 3. A significant difference ($P < 0.01$) in the dimensional size between the bamboo particles and the solid residues was observed. The average length and width for the original bamboo particles and the solid residues were 2312.01 and 891.19, and 567.67 and 17.02 μm , respectively. The solid residues had a high aspect ratio compared to that of the original bamboo particles (4.08 and 65.08 for the bamboo and the solid residues, respectively). The dimension and morphology results of the solid residues revealed that the solid residues were micro-sized fibers and were comparable to that of the original single fibers in terms of morphology. This result indicated that the solid residues in this study may have potential in fabrication of microfiber-reinforced composites.²⁸

CONCLUSIONS

The proposed microwave-assisted selective liquefaction process could efficiently remove lignin and extractives from bamboo and produce good-quality fibers with high holocellulose content. The holocellulose, lignin, and extractive contents of the solid residues from the reaction at 120 °C, 9 min were 94.28, 0.65, and 0.49%, respectively. The FTIR results revealed that aromatic rings of lignin could be completely dissociated with this liquefaction process. The improvement in crystallinity for the solid residue confirmed the efficient removal of noncellulosic materials by microwave selective liquefaction. TG/DTG analysis gave further evidence of the liquefaction of bamboo since the removal of lignin could significantly decrease the thermal stability of bamboo. The SEM images of the solid residue provide further information to better understand the effect of microwave energy heating on the liquefaction process.

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