Carbohydrate Polymers 85 (2011) 245–250

Contents lists available at ScienceDirect

Carbohydrate Polymers

journal homepage: www.elsevier.com/locate/carbpol

Study on structure and thermal stability properties of cellulose fibers from rice straw

Xiaolang Chen[a,b,*], Jie Yu[c,**], Zhibin Zhang[d], Canhui Lu[b]

[a] Key Laboratory of Advanced Materials Technology Ministry of Education, School of Materials Science and Engineering, Southwest Jiaotong University, Chengdu 610031, China
[b] The State Key Laboratory of Polymer Materials Engineering, Polymer Research Institute of Sichuan University, Chengdu 610065, China
[c] National Engineering Research Center for Compounding and Modification of Polymer Materials, Guiyang 550014, China
[d] School of Bioscience and Engineering, Southwest Jiaotong University, Chengdu 610031, China

ARTICLE INFO

Article history:
Received 9 June 2010
Received in revised form 11 February 2011
Accepted 14 February 2011
Available online 18 February 2011

Keywords:
Rice straw
Cellulose fiber
Chemical structure
Thermal properties

ABSTRACT

Cellulose fibers were extracted from the rice straw by a mechanical-high pressure steam technique. The structure, chemical composition and thermal properties of cellulose fibers were investigated by using Fourier transform infrared (FTIR) spectroscopy, wide-angle X-ray diffraction (WAXD), scanning electron microscopy (SEM), and thermogravimetric analysis (TGA). FTIR analysis and chemical composition of fibers demonstrate that this mechanical-high pressure steam treatment can result in partial removal of hemicellulose and lignin from the structure of fibers. WAXD results reveal that this results in improved crystallinity of the fibers. The rice straw fibers are determined to have diameters in the range of 5–10 μm. After mechanical-high pressure steam treatments, the thermal properties of the rice straw fibers from the TGA results are found to increase dramatically after treatments. The degradation temperature of the rice straw fibers reaches over 280 °C, which is reasonably promising for the use of these fibers in reinforced-polymer composites.

© 2011 Elsevier Ltd. All rights reserved.

1. Introduction

In many countries, straw is an abundant cellulosic by-product from crop production such as wheat, rice, corn and soybean. Cellulose is the main component in crops straw, which is a compact structure of hemicelluloses in close association with lignin (Sun & Sun, 2002). Moreover, cellulose, the most abundant organic compound and a renewable resource, gains increasing importance as raw materials for fuels, chemicals, paper industry, building board, and food industries because of its low cost, wide availability, and low content in non-fibrous materials (Ebeling et al., 1999; Gousse, Chanzy, Cerrada, & Fleury, 2004; Mohanty, Drzal, Askeland, & Misra, 2004; Puglia, Tomassucci, & Kenny, 2003; Roncero, Colom, & Vidal, 2003; Samir, Alloin, Sanchez, & Dufresne, 2004). As well known, rice straw has traditionally been used as animal feed for cattle and horse breeding, feedstock for paper industry, or organic fertilizer by burning it on the open field or burying it into the soil (Caviglioli et al., 2002; Vlasenko, Ding, Labavith, & Shoemaker, 1997). However, recent points of animal breeding practices and a growing attention to environmental problems have tended to limit these uses. In order to develop its new uses and increase its potential value, the isolation and characterization of rice straw have been extensively studied in recent years (Assavanig, Amornkitticharoen, Ekpaisal, Meevootisom, & Flegel, 1992; Jin & Chen, 2007; Sun and Cheng, 2002; Sun, Tomkinson, Mao, & Sun, 2002).

Rice straw is mainly composed of cell walls in which cellulose is one of the main components. In the cell walls, the cellulose chains bind together by hydrogen bonds to form microfibrils of several nanometers in diameter and millimeters in length. These high tensile strength crystalline microfibrils are the fundamental structural unit in straw cell walls. They make the major contribution to the mechanical strength of the straw cell walls. The microfibrils are bonded by a gel matrix composed of hemicellulose, lignin and other carbohydrate polymers to form biocomposites (Atanu, Badal, John, Shogren, & Willett, 2006; Wiiiam et al., 2005; Xu, Wang, Jiang, Yang, & Ji, 2007). However, the difficulties for rice straw to be effectively separated into renewable cellulose are due to the following two facts: the strong crystalline structure of cellulose in rice straw and the presence of a complex structure of lignin and hemicellulose with cellulose, which together limit the accessibility of rice straw. Therefore, various pretreatments of rice straw have been developed to split up the structure of cellulose and increase its exposure.

Although there are many pretreatment methods, few can be used on an industrial scale based on economical and environmental

---

* Corresponding author at: School of Materials Science and Engineering, Southwest Jiaotong University, Chengdu 610031, China. Tel.: +86 288 7602714; fax: +86 288 7602714.
** Co-corresponding author at: National Engineering Research Center for Compounding and Modification of Polymer Materials, Guiyang 550014, China. Tel.: +86 851 5845419; fax: +86 851 5845419.
E-mail addresses: chenxl612@sina.com.cn (X. Chen), yujiegz@126.com (J. Yu).

0144-8617/$ – see front matter © 2011 Elsevier Ltd. All rights reserved.
doi:10.1016/j.carbpol.2011.02.022

consideration (Sun et al., 2002). It is necessary to develop a cheap, efficient and environmental friendly pretreatment technique for the industrialization of rice straw. Physical treatments, such as crushing and grinding, steam and irradiation, have been proven to be effective in creating accessibility to chemicals and enzymes (Zhao, Li, & Qu, 2006). Treatments of the fibers result in chemical structural changes on the fiber surface and the cells. This has an effect on the properties of the fibers in application. Recently considerable work has been done on the isolation of these fibers from crops (Foyle, Jennings, & Mulcahy, 2007; Liu, Yu, & Huang, 2005; Mirjam, Gijs, Jan, Alphons, & Henk, 2007). In addition, most cellulosic materials are less crystalline. Interestingly, these amorphous celluloses are also important, since many interesting properties such as a higher chemical reactivity are associated with non-crystalline cellulose. It usually occurs on surfaces of the crystallites or in regions that periodically interrupt the cellulose crystallites. At present the isolation of highly pure cellulose from cellulosic materials by environmentally friendly techniques has been the subject of recent research e.g. (Li & Chen, 2007; Sun et al., 2004).

In this work, the mechanical-high pressure steam technique was used to separate the components of rice straw characterize them in order to evaluate their suitability as reinforcement for polymer composites. The chemical structure, morphology and physical properties of rice straw fibers were investigated by using Fourier transform infrared (FTIR) spectroscopy, wide-angle X-ray diffraction (WAXD) technique, scanning electron microscope (SEM), and thermogravimetric analysis (TGA).

2. Experimental

2.1. Materials

Rice straw was obtained from local farm (Chengdu farms, China). Absolute ethanol and all other chemical agents used in this study were of analytical grade and supplied by Chengdu Kelong Chemicals Co., Ltd. (Sichuan, China).

2.2. Preparation of the rice straw fibers

The isolation of cellulose from rice-based fibers was adopted to extract the cellulose from the rice straw according to the following steps. Firstly, the rice straw was dried at 60 °C for 16 h after it was cut into 4–5 cm lengths, and then the dried straw was crushed to pass through a 0.8 mm screen. Secondly, the rice straw powder was ground for 4 h in a ball mill and its chemical composition was established. Thirdly, the ground powder of the rice straw was soaked in water for 24 h. Fourthly, after soaking, an amount of water-saturated rice straw was streamed at 160 °C, 2 bar for 9, 18, 27, 36, 45, and 54 h with the ratio of water to rice straw 50:1 (v/w), and the value of pH is 7. In the fifth step, the steam solution was filtered and the residue was fully washed with distilled water and ethanol, respectively. Finally, it was dried in an oven at 60 °C for 16 h.

2.3. Measurements and characterization

2.3.1. Fourier transform infrared (FTIR) spectroscopy

The FTIR spectra were used to examine the structure of rice straw fibers which arose after treatment of the fibers. A Nicolet 560 spectrophotometer was used to characterize the spectra of each sample. The untreated and treated rice straw fibers were ground and mixed with KBr powders, and then the mixture was compressed into plates for FTIR analysis. FTIR spectra of samples were obtained in the range of 4000–400 cm^{-1}. 32 scans were co-added in order to achieve an acceptable signal-to-noise ratio. In all cases, spectra resolution was maintained at 4 cm^{-1}.

2.3.2. Chemical composition analysis

The chemical composition of the untreated and treated rice straw fibers was analyzed according to the procedure that was originally pioneered by Zobel and McElvee, and the Technical Association of Pulp and Paper Industry standards. The cellulose content in rice straw fibers was analyzed according to an HNO_3–ethanol method. Dry sample (1 g) was added to 200 ml of HNO_3–ethanol (10% HNO_3) and refluxed at 150 °C for 4 h. The supernatant liquor was decanted off into a beaker and the remaining mixture was filtered through a weighed sintered-glass filtering crucible. The filtered and the decanted liquor were re-filtered through the mat of cellulose that had formed on the sintered glass crucible during the first filtration step. The cellulose was then washed with hot water before washing the solid residue into a beaker with water. A certain amount of HNO_3–ethanol was added to reflux until the rice straw fibers became white. The residue was dried in the crucible at 100 °C and weighed. The cellulose concentration was calculated based on the initial weight of sample used. To determine the hemicellulose content the sample was first hydrolysed with hydrochloric acid to release pentoses, which are then converted to furfural. The furfural is determined using a bromine-bromide reagent. The sample was weighed into a 250 ml round bottomed flask. A certain amount of a 15% HCl solution was then added. The solution of HCl and sample was heated to 160 °C. The furfural that was produced as a result of hydrolysis of the pentoses was vaporized. It was condensed and collected in a receiver flask that was kept cool. The contents of the receiver flask were transferred into a 250 ml volumetric flask, rinsed with 15% HCl. The bromine-bromide solution was added dropwise to this flask, on shaking, a slight excess of bromine persisted. The flask was placed in a water bath at 25 °C for 2 h, at the end of which the bromine was destroyed by the addition of an excess of 95% ethanol which also served to precipitate the hemicelluloses. The absorbance of the solution was measured under corresponding experimental conditions, but replacing the diluted distillate with 5 ml of 15% HCl. The percent pentose was calculated from the furfural concentration. Finally, the hemicellulose content was calculated according to the pentose amount. The lignin content in rice straw fibers was determined by the 72% H_2SO_4 method (Nelson & Leming, 1957; Zobel & McElvee, 1966). Dry sample wrapped by qualitative filter paper was added to 150 ml of benzene–ethanol (2:1, v/v) and refluxed for 6 h. The sample was then dried and moved to a 250 ml conical flask. Appropriate amount of 72% H_2SO_4 was added to this conical flask. The conical flask containing the sample was put into 20 °C water bath kettle for 3 h under stirring. Deionised water was added to the conical flask and refluxed for another 4 h. Finally, the sample was washed with enough more hot water. The residue was dried to a constant weight. The total lignin content was calculated as a percentage of the original sample corrected for moisture.

2.3.3. Crystal structure measurements

The crystallinity of untreated and treated rice straw cellulose fibers was examined by wide-angle X-ray diffraction (WAXD) technique, using CuK_α radiation ($\lambda = 1.54$ Å), equipped with computerized data collection and analytical tools. The X-ray source was operated at a voltage of 40 kV and a filament current of 40 mA. All samples were scanned in 2θ ranges from 5 to 50° at a rate of 1°/min.

2.3.4. Morphology

The morphology of the rice straw fibers was performed by scanning electron microscopy (SEM) (Quanta 200, made in USA). Gold sputter coated samples were examined using a Cambridge Stereoscan 250 with an accelerating voltage of 10 kV.

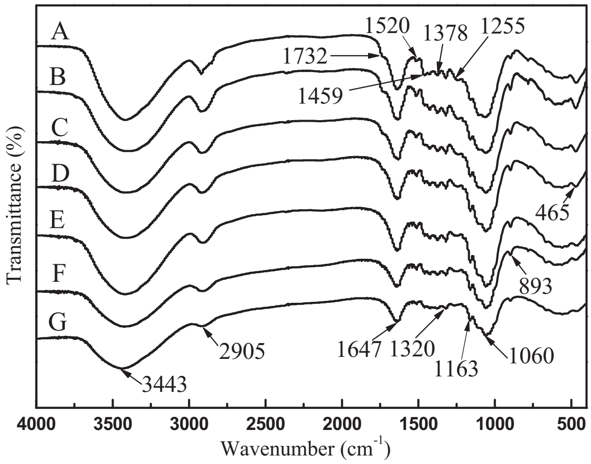

Fig. 1. FTIR spectra of (A) the untreated rice straw and (B)–(G) treated rice straw for the specified time: 9, 18, 27, 36, 45, 54 h.

2.3.5. Measurement of thermal properties

Thermogravimetric analysis (TGA) was performed to compare the degradation characteristics of the treated fibers with the untreated one. Thermal stability of each sample was determined using a TGA Q 500 series Thermogravimetric analyzer with a heating rate of 10 °C/min in a nitrogen environment.

3. Results and discussion

3.1. Structural characteristics and chemical composition

Fig. 1 shows the FTIR spectra of untreated and treated rice straw fibers. It can be seen from Fig. 1 that the dominant absorption peaks at 3443 and 2905 cm^{-1} are attributed to stretching vibrations of –OH groups and the C–H stretching, respectively. The smaller shoulder peak at 1732 cm^{-1} in the untreated rice straw is assigned to the characteristic of aliphatic esters in lignin and/or hemicellulose. The peak still appears when rice straw is treated for 9 h, however, this peak completely disappeared in the treated rice straw fibers with further increasing the treatment time because of the removal of most of the lignin and hemicellulose from the rice straw by applied high pressure steam extraction. At the same time, it can be clearly found that the peak at 1647 cm^{-1} is attributed to the bending mode of the absorbed water. The decrease of the intensity of this peak after fiber treatment is attributed to the partial removal of hemicelluloses. The peaks at 1520 and 1459 cm^{-1} in the untreated rice straw are indicative of the aromatic C═C stretch of aromatic vibrations in bound lignin (Sain & Panthapulakkal, 2006; Xiao, Sun, & Sun, 2001; Xu et al., 2006). The intensity of these peaks decreases in the treated rice straw and disappears in the FTIR spectrum when the treatment time exceeds 27 h. The results indicate that the partial lignin has been removed by the method used.

The peaks in the 1500–1000 cm^{-1} have changed with various steaming times. The absorbance peaks at 1378 and 1255 cm^{-1} originate from C–H ester bands and C–O stretching vibrations due to partial acetylation of hydroxyl groups in both polysaccharides and residual lignin (Sun, Tomkinson, Ma, & Liang, 2000). The disappearance of these bands revealed that lignin of straw was largely removed after treatment. The band at 1320 cm^{-1} is due to the C–C and C–O skeletal vibration. The peak at 1163 cm^{-1} relates to the C–O anti-symmetric stretching. The band at 1060 cm^{-1} is assigned to C–O–C pyranose ring skeletal vibration. The peak at 893 cm^{-1} represents the glycosidic C–H deformation with ring vibration contribution and OH bending in the treated rice straw, which indicates the typical structure of cellulose (Pan & Sano, 2005). Interestingly, the peaks in the 880–500 cm^{-1} showed a great change with increasing treatment time. Compared with untreated rice straw, only a wide peak appears in this region when treatment time is over 36 h, which is related to the characteristic peak of the cellulose phase. These FTIR results indicate that the hemicelluloses and lignin are removed from the rice straw fibers through the treatment used.

Table 1
Effect of pretreatment of rice straw on its main chemical composition.

Samples	Treatment time (h)	Chemical composition (%)		
		Cellulose	Lignin	Hemicellulose
A	Untreated	44.3 ± 0.4	20.4 ± 0.9	33.5 ± 0.6
B	9	47.2 ± 0.6	18.7 ± 0.7	31.8 ± 1.1
C	18	55.4 ± 0.5	14.1 ± 0.6	25.5 ± 0.2
D	27	61.6 ± 0.7	11.9 ± 1.1	22.7 ± 1.3
E	36	66.8 ± 0.8	10.1 ± 0.5	16.9 ± 0.6
F	45	68.4 ± 0.2	8.8 ± 0.2	14.6 ± 0.3
G	54	69.2 ± 0.3	8.2 ± 0.4	14.1 ± 0.5

The effect of mechanical-high pressure steam treatment of rice straw on its main chemical composition is listed in Table 1. It can be found that the chemical composition was greatly influenced by treatment time. The lignin and hemicelluloses decrease with increasing the treatment time while the cellulose increases with increasing treatment time. When the treatment time is 54 h, the cellulose content is increased from 44.3 to 69.2% while hemicelluloses and lignin content are significantly decreased to 14.1 and 8.2%, respectively. The results show that the high pressure steam treatment of rice straw fibers increases the surface area of the fibers to make polysaccharides more susceptible to hydrolysis. The increase of cellulose content is attributed to the solubilisation of their components. This indicates high pressure steam enhances the solubilisation of hemicellulose and lignin, which leads to the close contact between lignin and water and thus enhances the solubilisation of lignin (Zhu et al., 2006). The rice straw treated by mechanical-high pressure steam has higher cellulose, lower hemicelluloses and lignin. This chemical compositional change in the rice straw fibers results in a higher content of crystalline cellulose, and thus improves thermal stability and strength of the fibers.

3.2. Wide-angle X-ray diffraction (WAXD) analysis

The WAXD patterns of untreated and treated rice straw fibers are shown in Fig. 2. It can been seen from Fig. 2(A) that the diffraction peak at (0 0 2) is wide and round for untreated rice straw, however, the peak at (0 0 2) for mechanical-high pressure steam

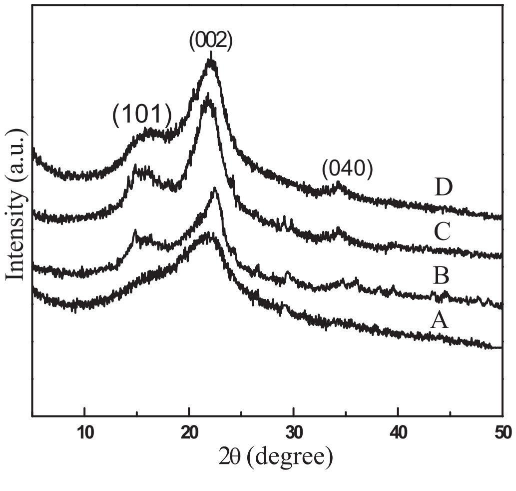

Fig. 2. X-ray diffraction patterns of untreated and treated rice straw fibers: (A) untreated; (B) treated for 9 h; (C) treated for 27 h; (D) treated for 54 h.

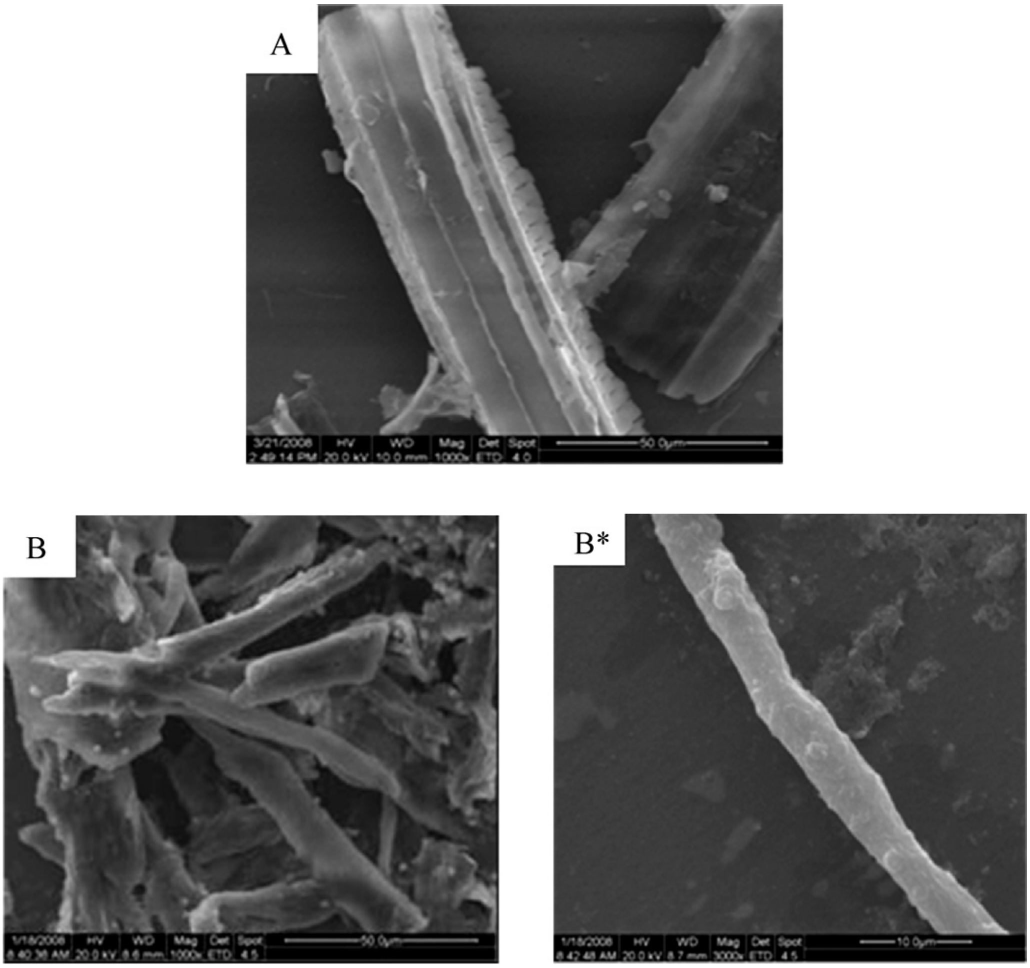

Fig. 3. SEM micrographs of the untreated and treated rice straw fibers: (A) untreated straw; (B) and (B*) treated straw.

treated rice straw fibers is sharper and narrower than untreated straw fibers. A shoulder peak at (1 0 1) and a weak peak at (0 4 0) appear after mechanical-high pressure steam treatment, which are assigned to the cellulose phase. This reveals the removal of most of lignin and hemicellulose from the rice straw. The hydrogen bonds between cellulose molecules result in an ordered system. Individual fibrillar units consist of long periods of ordered regions interrupted by completely disordered regions (Alemdar & Sain, 2008). Mechanical-high pressure steam treatments affect the crystallinity of the cellulosic fibers. The sharper diffraction peak at (0 0 2) indicates higher crystallinity degree in the treated fibers. In all cases the portion of crystalline cellulose was found to be higher for mechanical-high pressure steam treated cellulose fibers than for untreated fibers due to partial removal of the lignin and hemicelluloses by treatment. Higher crystallinity in the treated fibers is associated with a higher tensile strength of the fibers. Therefore the expected mechanical properties of the composite materials can be obtained by adding these fibers (Mirjam et al., 2007; Mohamed & Mohammad, 2007). The sample D treated for 54 h has lost some crystalline order with further increasing the steaming time and the peak at (0 0 2) becomes broader due to chemical degradation at a very long steaming times.

3.3. Morphology

The mechanical-high pressure steam treatment results in morphological changes as well as chemical changes on the rice straw fiber surfaces. SEM micrographs of the untreated and treated rice straw fibers are shown in Fig. 3. According to the above discussion, most lignin, hemicelluloses and other extractive substances were removed from rice straw, and mainly cellulose was left. It is clearly found from Fig. 3 that the morphologies of untreated and treated rice straw are different. Some parts of dense lignin, hemicelluloses, and ashes surround the fibers for untreated rice straw, while the surface of treated samples looks smoother than that in the untreated one, which is attributed to the removal of amorphous lignin and hemicelluloses. In addition, the average diameter is about 5–10 μm for the treated rice straw fibers. On other hand, it can be found that the mechanical-high pressure steam treatments also influence the separation of micro-sized fibers from the fiber bundles of rice straw. The rice straw fibers separated extensively into individual micro-sized fibers after the mechanical-high pressure steam treatments.

3.4. Thermal properties

Study on the thermal properties of crops straw fibers is very important in order to estimate their application for reinforced-polymer composites processing because the processing temperature for many polymeric materials exceeds 200 °C. Fig. 4 shows the TGA results from rice straw fibers. It is clearly seen that the degradation temperature greatly increases after treatments. In addition, the degradation temperature increases with further increasing the treatment time. Similar results were also obtained from other agricultural straw fibers (Xu et al., 2006). The curve A of Fig. 4 shows that the untreated rice straw fibers started to decom-

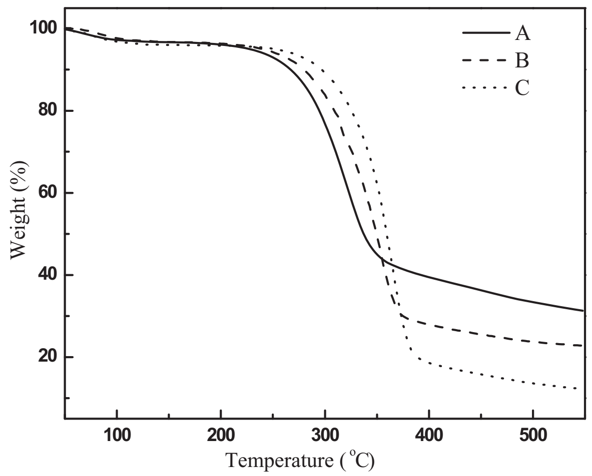

Fig. 4. TGA thermaograms of the untreated and treated rice straw fibers: (A) untreated; (B) treated for 36 h; (C) treated for 54 h.

Table 2
Thermal stability data of untreated and treated rice straw fibers.

Samples	$T_{5\%}$ (°C)	$T_{20\%}$ (°C)	$T_{50\%}$ (°C)	T_p (°C)	Residue at 530 °C (%)
A	226.9	293.4	336.6	310.5	32.1
B	239.6	308.0	349.8	339.4	23.2
C	257.7	324.9	359.6	352.7	12.5

$T_{n\%}$ represents the onset decomposition temperature of 5, 20 and 50% weight loss, respectively. T_p represents peak temperature of DTG.

pose about 200 °C, however, treated rice straw fibers with 45 and 54 h of treatment time started to decompose at 235 and 252 °C, respectively, as shown in Fig. 4(B and C). This indicates that the treated residues have a higher thermal stability than untreated ones. The fiber residue remaining after heating to 530 °C both untreated and treated rice straw fibers indicates the presence of the carbonaceous materials in the rice straw in the nitrogen atmosphere (Sun et al., 2000). There is a significant difference between the contents of the residues remaining after 530 °C for untreated and treated rice straw fibers. The relatively low amount of residue in the fibers obtained after treatments may be attributed to removal of calcium oxalate crystals of lignin and other sources of ash during the high pressure and temperature procedure. These results suggest that the higher temperature of thermal decomposition and lower residual mass of the rice straw fibers obtained after treatments is attributed to partial removal of hemicellulose and lignin. These results are very consistent with results obtained from the FTIR analysis.

The TGA data of untreated and treated rice straw fibers are listed in Table 2. The results show the decomposition temperature of 5%, 20% and 50% weight loss, the onset temperature of TGA and peak temperature of DTG. All these indicate that the thermal stability of treated rice straw fibers is visibly improved.

4. Conclusions

In the present work, cellulose fibers were isolated and extracted from rice straw followed by a mechanical-high pressure steam treatments. Chemical structure and composition, morphology and physical and thermal stability properties of the rice straw fibers were investigated by FTIR, WAXD, SEM, TGA, etc. Experimental results show that the produced rice straw fibers' diameter are within the range of 5–10 μm. FTIR analysis and chemical composition of the fibers reveal the partial removal of hemicellulose and lignin due to the success of the mechanical-high pressure treatments. The crystallinity of the fibers greatly increases for the rice straw fibers compared to the untreated rice straw fibers. The rice straw fibers exhibit improved thermal stability properties, with thermal degradation temperature increased by 40%, and thus making them promising candidates for use in polymer composites. It can be concluded from these results that the fibers obtained from rice straw have great potential applications in reinforced-polymer composites.

Acknowledgements

The authors are grateful to the Opening Project of State Key Laboratory of Polymer Materials Engineering (Sichuan University) (KF201005), Hope Stars Foundation of Southwest Jiaotong University (2008–12), and Science and Technology developing Project Foundation of Southwest Jiaotong University (2009–9) for financial supports of this work.

References

Alemdar, A., & Sain, M. (2008). Isolation and characterization of nanofibers from agricultural residues: Wheat straw and soy hulls. *Bioresource Technology*, *99*, 1664–1671.

Assavanig, A., Amornkitticharoen, B., Ekpaisal, N., Meevootisom, V., & Flegel, T. W. (1992). Isolation, characterization and function of laccase from trichoderma. *Application Microbiology Biotechnology*, *38*, 198–202.

Atanu, B., Badal, C. S., John, W. L., Shogren, R. L., & Willett, J. L. (2006). Process for obtaining cellulose acetate from agricultural by-products. *Carbohydrate Polymers*, *64*, 134–137.

Caviglioli, G., Valeria, P., Brunella, P., Sergio, C., Attilia, A., & Gaetano, B. (2002). Identification of degradation products of buprofen arising from oxidative and thermal treatments. *Journal of Pharmaceutical and Biomedical Analysis*, *30*, 499–509.

Ebeling, T., Paillet, M., Borsali, R., Diat, O., Dufresne, A., Cavaille, J. Y., et al. (1999). Shear-induced orientation phenomena in suspensions of cellulose microcrystals, revealed by small angle X-ray scattering. *Langmuir*, *15*, 6123–6126.

Foyle, T., Jennings, L., & Mulcahy, P. (2007). Compositional analysis of lignocelluloses materials: Evaluation methods used for sugar analysis of waste paper and straw. *Bioresource Technology*, *98*, 3026–3036.

Gousse, C., Chanzy, H., Cerrada, M. L., & Fleury, E. (2004). Surface silylation of cellulose microfibrils: Preparation and rheological properties. *Polymer*, *45*, 1569–1575.

Jin, S. Y., & Chen, H. Z. (2007). Fractionation of fibrous fraction from steam-exploded rice straw. *Process Biochemistry*, *42*, 188–192.

Li, D. M., & Chen, H. Z. (2007). Biological hydrogen production from steam-exploded straw by simultaneous saccharification and fermentation. *Interational Journal of Hydrogen Energy*, *32*, 1742–1748.

Liu, R. G., Yu, H., & Huang, Y. (2005). Structrue and morphology of cellulose in wheat straw. *Cellulose*, *12*, 25–34.

Mirjam, A. K., Gijs, B., Jan, Z., Alphons, G. J. V., & Henk, A. S. (2007). Effect of pretreatment severity on xylan solubility and enzymatic breakdown of the remaining cellulose from wheat straw. *Bioresource Technology*, *98*, 2034–2042.

Mohamed, E. S., & Mohammad, L. H. (2007). Physical and mechanical properties of microcrystalline cellulose prepared from agricultural residues. *Carbohydrate Polymers*, *67*, 1–10.

Mohanty, A. K., Drzal, L. T., Askeland, P., & Misra, M. (2004). Effects of alkali treatment on the structure, morphology and thermal properties of native grass fibers as reinforcements for polymer matrix composites. *Journal of Materials Science*, *39*, 1051–1054.

Nelson, L., & Leming, J. A. (1957). Evaluation of monoethanolamine method of cellulose determination for agricultural residues. *Tappi Journal*, *40*, 846–850.

Pan, X. J., & Sano, Y. S. (2005). Fractionation of wheat straw by atmospheric aectic acid process. *Bioresource Technology*, *96*, 1256–1263.

Puglia, D., Tomassucci, A., & Kenny, J. M. (2003). Processing, properties and stability of biodegradable composites based on Mater-Bi-(R) and cellulose fibres. *Polymers for Advanced Technology*, *14*, 749–756.

Roncero, M. B., Colom, J. F., & Vidal, T. (2003). Why oxalic acid protects cellulose during ozone treatments? *Carbohydrate Polymers*, *52*, 411–422.

Sain, M., & Panthapulakkal, S. (2006). Bioprocess preparation of wheat straw fibers and their characterization. *Industry Crops and Products*, *23*, 1–8.

Samir, M. A. S. A., Alloin, F., Sanchez, J. Y., & Dufresne, A. (2004). Cross-linked nanocomposite polymer electrolytes reinforced with cellulose whiskers. *Macromolecules*, *37*, 4839–4844.

Sun, Y., & Cheng, J. (2002). Hydrolysis of lignocellulosic materials for ethanol production: A review. *Bioresource Technology*, *83*, 1–11.

Sun, R. C., & Sun, X. F. (2002). Structural and thermal characterization of acetylated rice, wheat, rye, and barley straws and poplar wood fibre. *Industry Crops and Products*, *16*, 225–235.

Sun, R. C., Tomkinson, J., Ma, P. L., & Liang, S. F. (2000). Comparative study of hemicelluloses from rice straw by alkali and hydrogen peroxide treatments. *Carbohydrate Polymers*, *42*, 111–122.

Sun, R. C., Tomkinson, J., Mao, F. C., & Sun, X. F. (2002). Physicochemical characterization of lignins from rice straw by hydrogen peroxide treatment. *Journal of Applied Polymer Science*, *79*, 719–732.

Sun, X. F., Xu, F., Sun, R. C., Wang, Y. X., Fowler, P., & Baird, M. S. (2004). Characteristics of degraded lignins obtained from steam exploded wheat straw. *Polymer Degradation and Stability*, *86*, 245–256.

Vlasenko, E. Y., Ding, H., Labavith, J. M., & Shoemaker, S. P. (1997). Enzymatic hydrolysis of pretreated rice straw. *Bioresource Technology*, *59*, 109–119.

Wiiiam, J. O., Justin, S., Syed, H. I., Gregory, M. G., Mara, E. G., & Jean, F. R. (2005). Application of cellulose microfibrils in polymer nanocomposites. *Journal of Polymer Environment*, *13*, 301–306.

Xiao, B., Sun, X. F., & Sun, R. C. (2001). Chemical, structural, and thermal characterization of alkali-soluble lignins and hemicelluloses, and cellulose from maize stems, rye straw, and rice straw. *Polymer Degradation and Stability*, *74*, 307–319.

Xu, F., Liu, C. F., Geng, Z. C., Sun, J. X., Sun, R. C., Hei, B. H., et al. (2006). Characterization of degrade organosolv hemicelluloses from wheat straw. *Polymer Degradation and Stability*, *91*, 1880–1886.

Xu, Z., Wang, Q. H., Jiang, Z., Yang, X. X., & Ji, Y. Z. (2007). Enzymatic hydrolysis of pretreated soybean straw. *Biomass Bioenergy*, *31*, 162–167.

Zhao, J., Li, X. Z., & Qu, Y. B. (2006). Application of enzymes in producing bleached pulp from wheat straw. *Bioresource Technology*, *97*, 1470–1476.

Zhu, S. D., Wu, Y. X., Yu, Z. N., Wang, C. W., Yu, F. Q., Jin, S. W., et al. (2006). Comparison of three microwave/chemical pretreatment process for enzymatic hydrolysis rice straw. *Biosystems Engineering*, *93*, 279–283.

Zobel, B., & McElvee, R. (1966). Variation of cellulose in loblolly pine. *Tappi Journal*, *49*, 383–387.