The Degumming Effect on Kenaf by Different Residence Times of Steam Explosion Treatment

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Steam explosion (STEX) is an effective method of degumming kenaf and separating kenaf fibers. The residence time has a strong effect on the STEX process, and its mechanism of degumming kenaf was studied in this paper. In this research, five different residence times were chosen to treat kenaf at 1 MPa STEX pressure. The morphological changes were recorded using an optical camera and scanning electron microscopy (SEM). The chemical and physical properties of kenaf fiber were analyzed using wet chemistry analysis and a standard mechanical test. The cellulose content increased with increasing residence time. The breaking strength of kenaf fiber reached the highest level at 5 min residence time. It was also found that four different levels of kenaf fiber exist in the steam explosion process. This finding is very helpful for the degumming method development.

Keywords: Kenaf; Fiber; Degumming; Steam explosion; Residence time

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INTRODUCTION

Natural cellulosic fiber derived from lignocellulosic biomass has received increased interest in the textile and fiber reinforcement composite area (Shahid ul *et al.* 2013; Zheng and Wang 2014; Yan *et al.* 2016). The characteristics of biomass, such as abundance, sustainability, and reproducibility, contribute greatly to the increasing demand (Zhao and Li 2016). Traditional fiber resources including cotton, linen, flax, and ramie have been investigated thoroughly and are widely used in the world (Peets *et al.* 2017). However, they still cannot meet the requirement of the growing demands of the cellulose fiber industry (Kohli and Gupta 2015). Therefore, research on new fiber resources is important to industry.

Kenaf is a cellulosic fiber raw material that has been used for 2,000 years (Gao *et al.* 2015). However, the production of kenaf fiber is low because its diameter is too high (Amel *et al.* 2013). Research found that the gum content of kenaf is higher than 40%, while its lignin content is approximately 20%, which leads to the difficulty of degumming kenaf (Gao *et al.* 2015). New degumming technology could bring a potential way to make fine kenaf fibers.

Steam explosion has been shown to be a good pretreatment method for destroying the structure of biomass and pre-degumming (Rocha *et al.* 2012). This method is widely used on rice straw (Chen *et al.* 2011), sugarcane (Rocha *et al.* 2012), corn stover (Liu *et*

al. 2013), and wood (Martin-Sampedro *et al.* 2011) for making sugars and bioethanol. The above research demonstrates that steam explosion has a strong ability to degrade non-cellulose polysaccharides and to destroy the structure of biomass that facilitates the following treatment (Martin-Sampedro *et al.* 2011). Therefore, steam explosion could also be a good tool for pretreating kenaf for degumming kenaf fibers.

The authors' previous study investigated the effect of steam pressure on the chemical and structural properties of kenaf fibers during the steam explosion process (Zhang *et al.* 2016). This previous research offers good guidance on degumming kenaf using steam explosion. It is known that the pressure and residence time are the two main parameters of steam explosion for pretreating biomass (Vivekanand *et al.* 2013). This study focuses on the degumming effect of different residence times in steam explosion treatments on kenaf.

EXPERIMENTAL

Materials

Kenaf bast was supplied by CHTC Helon CO., Ltd. All the samples were harvested from Xinjiang Province, China. Kenaf bast was cut to obtain a uniform size of approximately 5-cm-long for the following treatment.

Steam explosion treatment

The kenaf was first soaked in distilled water at room temperature for 12 h. Then, 10 g of water-treated kenaf was placed in a steam explosion machine (QB-200, Gentle Bioenergy, Hebi City of Henan Province, China). The steam explosion was performed at 1 MPa pressure with different residence times (1 min, 2 min, 3 min, 5 min, 10 min, and 15 min). After the pressure was released, the sample was collected and rinsed using water. The kenaf fibers were oven-dried at 75 °C for 24 h.

Methods

Scanning electron microscopy (SEM)

The SEM images of kenaf before and after treatment were acquired using a JSM 6390LV scanning electron microscope (JEOL, San Diego, USA).

Chemical composition determination

The acid-soluble and acid-insoluble lignin content of kenaf fibers was determined following the National Renewable Energy Laboratory (NREL) method (Hansen *et al.* 2016). The water-soluble matter, pectin, hemicellulose, and cellulose content of the kenaf fibers were determined using the same method as the authors' previous study (Zhang *et al.* 2016).

Kenaf fiber properties testing

The kenaf fiber was processed and characterized using the following methods. Raw kenaf bast and kenaf fiber with steam explosion treatment were treated with the traditional soda boil degumming method to extract the kenaf fibers. This method is described in the authors' previous study (Gao *et al.* 2015). The extracted fibers were then oven-dried for the subsequent characterization.

Fiber length, diameter, and breaking tenacity are three of the most important indices for textile fibers. The length, diameter, and breaking tenacity were measured according to the Chinese National Standard GB/T 18147.3 (2000), GB/T 18147.4 (2000), and GB/T 18147.5 (2000), respectively (Jie *et al.* 2014). The fiber bundle tensile tester of Y162 (Depp Textile Technology Co., Ltd., Changzhou, China) was used for testing the breaking tenacity of kenaf fiber (Baiardo *et al.* 2004). In this study, "dtex" was chosen as the unit for evaluating fiber diameter based on the standard.

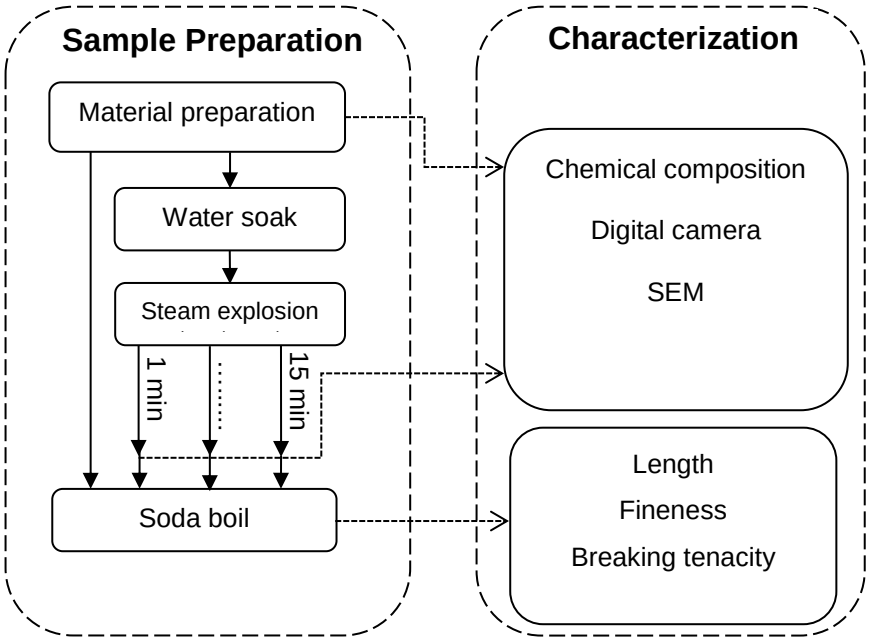

Fig. 1. Schematic diagram for the degumming and characterization process

RESULTS AND DISCUSSION

Morphological Changes with Different Residence Time Treatment

The separation effect of the steam explosion with different residence times was recorded by an optical camera and is shown in Fig. 2. The results revealed that the steam explosion had a strong separation effect; the kenaf was well separated into coarse fiber after the steam explosion treatment as seen in Figs. 2a and 2b. As the residence time increased, the coarse fiber was further separated, and the color of the kenaf fiber darkened, as compared from Fig. 2b through 2f. The total energy applied to the kenaf increased with the residence time increase, which could help degum more non-cellulose matters and further separate the fiber. It also made the lignin oxidize more, which led to the dark fiber.

The SEM images showed the steam explosion treatment on a μm scale. Coupled with Fig. 2a, it was found that the width of kenaf fiber can be divided into four levels, which are 1 cm to 10 cm level (level 1), 30 μm to 50 μm level (level 2), 10 μm to 20 μm level (level 3), and 3 μm to 10 μm level (level 4).

As shown in Fig. 3, raw kenaf has a large amount of gum on the surface of the coarse fiber. After the steam explosion treatment and the separation of the coarse fiber,

the coarse fiber had a width of approximately 30 μm to 50 μm (level 2) as displayed in Fig. 3, but there was still a lot of gum on the fiber (1 min residence time).

Fig. 2. Photograph of kenaf (fiber) after different residence time steam explosion treatment (a) raw kenaf, (b) 1 min, (c) 3 min, (d) 5 min, (e) 10 min, and (f) 15 min

When the residence time was increased from 1 min to 5 min, the gum was removed from the surface of the coarse fiber, and the level 3 of coarse fiber with a width of 10 μm to 20 μm was achieved (5 min residence time). With the residence time further increased, the final basic unit of kenaf appeared, which is also called the fiber cell (level 4). The gum on and between level 4 fibers had been partially removed, and the width of the level 4 fiber was 3 μm to 10 μm.

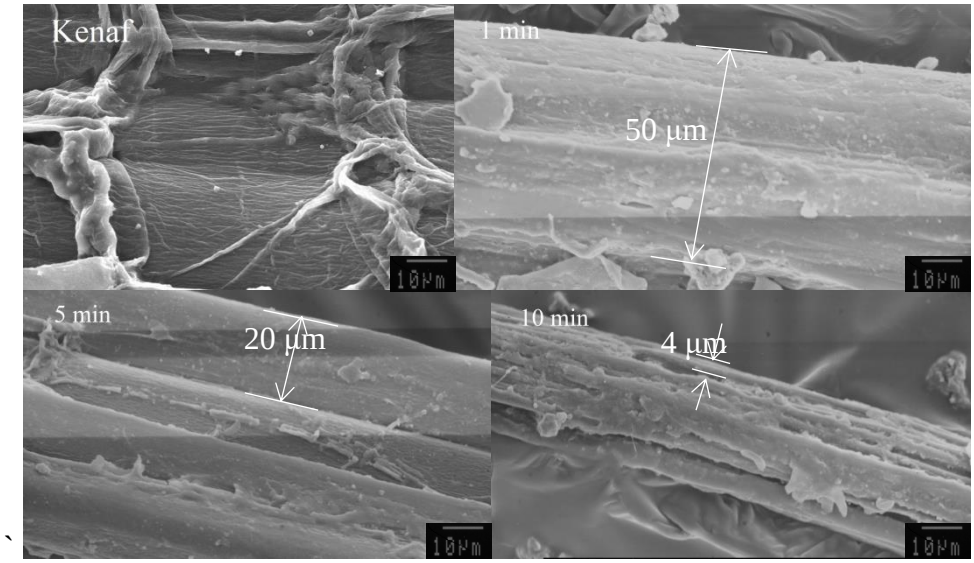

Fig. 3. SEM images of kenaf (fiber) after different residence time steam explosion treatment Note: the SEM images of 2 min and 3 min samples were similar to 1 min sample; the SEM image of 15 min sample was similar to 10 min sample, therefore 2 min, 3 min, and 15 min samples are not shown in this paper.

Figure 3 also implies that the steam explosion treatment can separate kenaf to level 3 coarse fibers, but it cannot go further. Other treatment methods should be combined if the level 4 fiber needs to be produced from kenaf. Even though steam explosion was proven as not enough for degumming completely, the finding of level 4 fibers during the treatment has cardinal importance. The investigation of the structure, chemical composition, and binding style of each unit could be a good guidance for further degumming method development.

Effect of Residence Time on Chemical Composition of Kenaf Fibers

The change of the chemical composition of kenaf fiber with different residence times was also investigated. As shown in Fig. 4, when the residence time increased, the relative amounts of hemicellulose, pectin, and acid soluble (AS) lignin decreased, while cellulose, lignin, and water-soluble matter (WSM) increased. Much of the literature demonstrates that the steam explosion treatment can degrade the structure of pectin and hemicellulose and then result in a weight loss of hemicellulose and pectin during the pressure release period (Dreyer *et al.* 2002; Sun and Cheng 2002; Alvira *et al.* 2010). Research also found that organic acids can be produced by pectin and hemicellulose degradation (Hongzhang and Liying 2007), the organic acid soluble part of acid soluble lignin. This was the reason for the decrease of AS lignin content. Cellulose and lignin have a high degree of polymerization and strong structures. They remained stable in steam explosion treatments. With the removal of gum content (pectin and hemicellulose), the weight of raw fiber decreased. Therefore, the cellulose and lignin content increased. The WSM content decreased rapidly at first (1 min) and should have decreased further with increased time. However, Fig. 3 shows that the WSM content increased after 1 min. Pectin and hemicellulose could be degraded to low molecular weight polysaccharides (Sun *et al.* 2005). Parts of them were removed in the course of the pressure release process, while the others were retained in kenaf fiber. They can be extracted by hot water, which is why WSM content increased. This mechanism can be verified by further study.

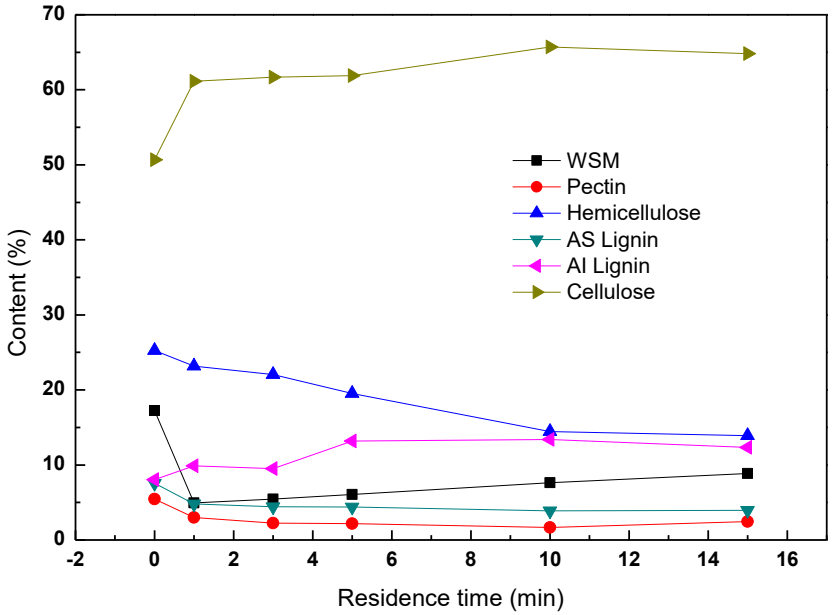

Fig. 4. Chemical composition of kenaf after different residence time steam explosion treatments; Note: WSM represents water soluble matter, AS represents acid soluble, AI represents acid insoluble

It was also found that all of the chemical compositions of the contents were similar when comparing the 10 min and 15 min samples. This indicated that the increase of residence time from 10 min to 15 min had no evident effect on the chemical composition of the fibers. Therefore, 10 min was better than 15 min for degumming kenaf.

Kenaf Fiber Properties

Both the fiber length and diameter decreased with the increase of the residence time as described in Fig. 5.

As stated in the Chinese textile standard "FZ/T 31003 (2009)," the bundle fibers should have a length and diameter standard. Any fiber whose length is smaller than 30 mm (length limit) or has a diameter higher than 28.6 dtex (fineness limit) can be considered unusable for spinning (China 2009). It was found that all samples' lengths were longer than the length limit (black line), which met the requirement of the standard; but their diameter was higher than fineness limit (blue line), such that they did not meet the requirement of the standard. This implied that all of the samples need a further degumming process, which would need to degum the current fiber to a thinner diameter. Therefore, a fineness limit*2 line (red line) is required to select the samples.

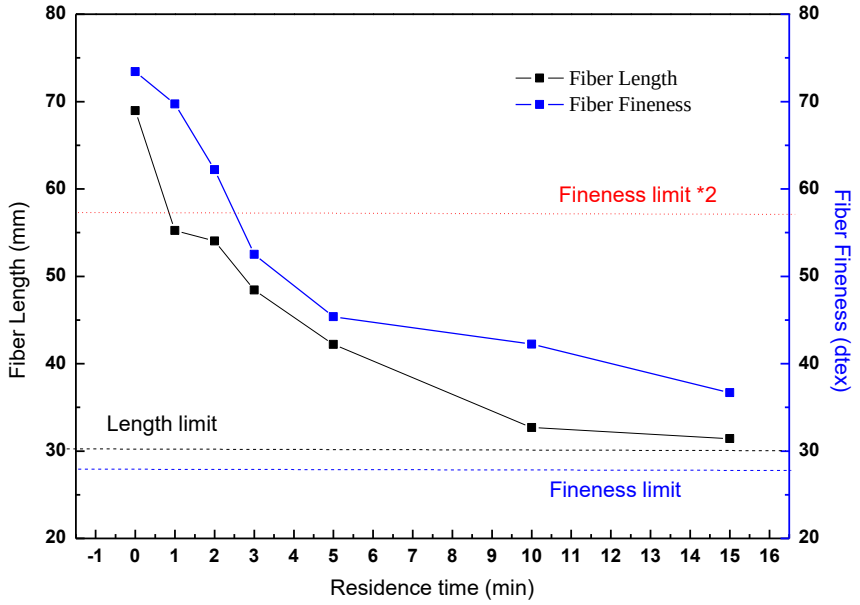

Fig. 5. Fiber length and diameter of kenaf fiber after different residence time steam explosion treatments

The raw kenaf and kenaf with 1 min and 2 min steam explosion treatment samples had a diameter higher than fineness limit*2 line, which demonstrated that they were difficult to degum to fiber and was lower than the diameter limit. The diameter of 3 min, 5 min, 10 min, and 15 min samples had a fineness of 52.5 dtex, 45.38 dtex, 42.22 dtex, and 36.67 dtex, respectively. They have the potential ability to be degummed to 28.6 dtex. However, with the decrease of diameter, the length of fiber decreased as well. The fiber length of 10 min and 15 min samples were 32.68 mm and 31.41 mm, respectively. They were very close to the 30 mm length limit, and they could be decreased to lower than 30 mm with a further degumming treatment. Therefore, the 10 min and 15 min samples

cannot be degummed for textile usage fibers. The kenaf fibers with 3 min and 5 min residence time steam explosions were the best in this research for textile spinning resources. All of the other samples could only be considered as a material for the fiber reinforcement industry.

As shown in Fig. 6, the breaking tenacity of kenaf fiber increased first and then decreased as the residence time increased. The kenaf fiber of 5 min residence time showed the highest breaking tenacity. It is known that the fibers from the bast materials are mostly fiber bundles. They are coarse fibers that consist of several fiber cells combined together using gum. The breaking tenacity of bast fibers (fiber bundle) is the comprehensive strength property between the fiber cell and gum. With the diameter decrease, the fiber changed from large bundle fibers to small bundle fibers, and the gum content decreased as well. The breaking tenacity of fiber is mostly from the mechanical property of fiber cells, which is much higher than the gum mechanical property. Therefore, with the decrease of fiber diameter, the breaking tenacity of fiber should increase. From 0 min (kenaf without steam explosion treatment) to 5 min, the fiber diameter decreased, while the breaking tenacity increased. This finding is consistent with the above discussion. However, the breaking tenacity of fiber from 5 min to 15 min decreased with the decrease of the fiber diameter. It demonstrated that there was another effect on fiber when the residence time increased to 10 min. As investigated in a previous study, the degree of polymerization of kenaf fiber will decrease if the energy is too high in the steam explosion treatment. The decrease of the degree of polymerization implies that the cellulose structure weakened, which led to a decrease of the mechanical properties of the fiber cell, which made the fiber breaking tenacity decrease further. In this research, Fig. 6 clearly shows that the degree of polymerization of kenaf fibers decreased when the residence time was 10 min or higher. This can be demonstrated *via* a gel permeation chromatograph (GPC) test.

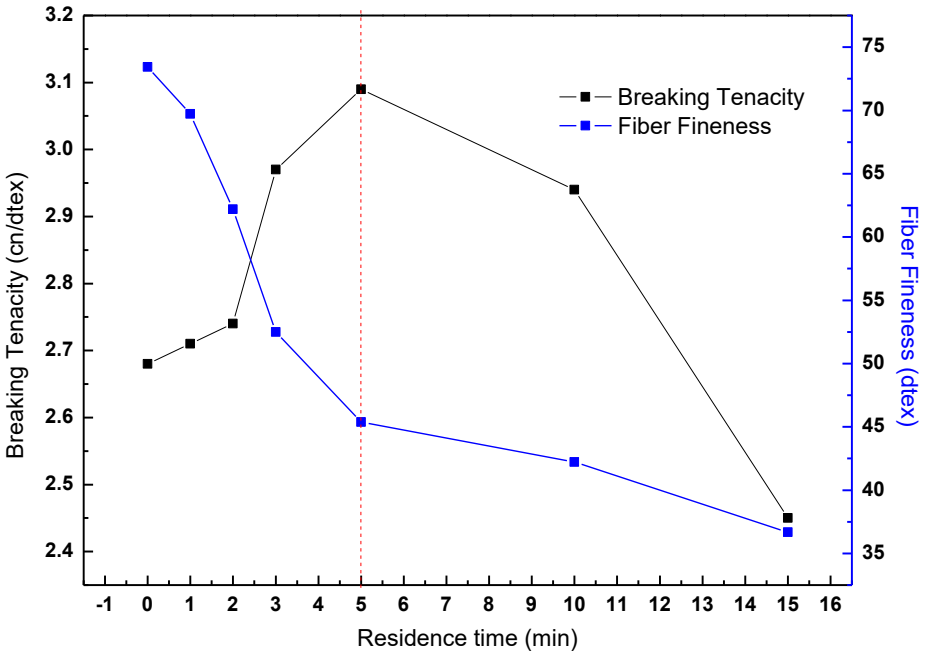

Fig. 6. Breaking tenacity and fiber diameter of kenaf fiber after different residence time steam explosion treatments

CONCLUSIONS

1. The raw kenaf was treated for different residence times using steam explosion. It was found that four different levels of kenaf fiber exist in this process. This finding can be helpful for the further development of degumming methods.
2. When the residence time was 10 min or higher, the degumming effect was low, based on the chemical composition changes study. The fibers were smaller than 30 mm, which was not enough for the spinning process; the breaking tenacity decreased too.
3. In conclusion, a 5 min residence time steam explosion was the best time for treating kenaf. It resulted in the best combination of length, diameter, and mechanical properties. For textile usage purpose, a 10 min or higher residence time is not suggested.

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REFERENCES CITED

Alvira, P., Tomás-Pejó, E., Ballesteros, M., and Negro, M. J. (2010). "Pretreatment technologies for an efficient bioethanol production process based on enzymatic hydrolysis: A review," *Bioresource Technol.* 101(13), 4851-4861.DOI: 10.1016/j.biortech.2009.11.093

Amel, B. A., Paridah, M. T., Sudin, R., Anwar, U., and Hussein, A. S. (2013). "Effect of fiber extraction methods on some properties of kenaf bast fiber," *Ind. Crop. Prod.* 46, 117-123. DOI: 10.1016/j.indcrop.2012.12.015`

Baiardo, M., Zini, E., and Scandola, M. (2004). "Flax fibre-polyester composites," *Compos. Part A-Appl. S.* 35(6), 703-710. DOI: 10.1016/j.compositesa.2004.02.004

Chen, W. H., Pen, B. L., Yu, C. T., and Hwang, W. S. (2011). "Pretreatment efficiency and structural characterization of rice straw by an integrated process of dilute-acid and steam explosion for bioethanol production," *Bioresource Technol.* 102(3), 2916-2924. DOI: 10.1016/j.biortech.2010.11.052

Dreyer, J., Müssig, J., Koschke, N., Ibenthal, W. D., and Harig, H. (2002). "Comparison of enzymatically separated hemp and nettle fibre to chemically separated and steam exploded hemp fibre," *Journal of Industrial Hemp* 7(1), 43-59. DOI: 10.1300/J237v07n01_05

FZ/T 31003 (2009). "The standard for refine of Jute," China Standard Press, Beijing, China.

Gao, S., Han, G., Jiang, W., Zhang, Y., and Zhang, X. (2015). "Steam explosion and alkali-oxygen combined effect for degumming of kenaf fiber," *BioResources* 10(3), 5476-5488. DOI:

GB/T 18147.3 (2000). "Test method for hemp fiber- Part 3: Test method for length of hemp fiber," China Standard Press, Beijing, China.

GB/T 18147.4 (2000). "Test method for hemp fiber- Part 4: Test method for fineness of hemp fiber," China Standard Press, Beijing, China.

GB/T 18147.5 (2000). "Test method for hemp fiber- Part 5: Test method for rupture strength of hemp fiber," China Standard Press, Beijing, China.

Hansen, B., Kusch, P., Schulze, M., and Kamm, B. (2016). "Qualitative and quantitative analysis of lignin produced from beech wood by different conditions of the organosolv process," *J. Polym. Environ.* 24(2), 85-97. DOI: 10.1007/s10924-015-0746-3

Hongzhang, C., and Liying, L. (2007). "Unpolluted fractionation of wheat straw by steam explosion and ethanol extraction," *Bioresource Technol.* 98(3), 666-676. DOI: 10.1016/j.biortech.2006.02.029

Jie, Z., Hua, Z., and Jianchun, Z. (2014). "Effect of alkali treatment on the quality of hemp fiber," *Journal of Engineered Fibers and Fabrics.* 9(2), 19-24.

Kohli, P., and Gupta, R. (2015). "Alkaline pectinases: A review," *Biocatal. Agric. Biotechnol.* 4(3), 279-285. DOI: 10.1016/j.bcab.2015.07.001`

Liu, Z. H., Qin, L., Jin, M. J., Pang, F., Li, B. Z., Kang, Y., Dale, B. E., and Yuan, Y. J. (2013). "Evaluation of storage methods for the conversion of corn stover biomass to sugars based on steam explosion pretreatment," *Bioresource Technol.* 132, 5-15. DOI: 10.1016/j.biortech.2013.01.016

Martin-Sampedro, R., Capanema, E. A., Hoeger, I., Villar, J. C., and Rojas, O. J. (2011). "Lignin changes after steam explosion and laccase-mediator treatment of eucalyptus wood chips," *J. Agr. Food Chem.* 59(16), 8761-8769. DOI: 10.1021/jf201605f

Peets, P., Leito, I., Pelt, J., and Vahur, S. (2017). "Identification and classification of textile fibres using ATR-FT-IR spectroscopy with chemometric methods," *Spectrochim. Acta. A.* 173, 175-181. DOI: 10.1016/j.saa.2016.09.007

Rocha, G. J. M., Gonçalves, A. R., Oliveira, B. R., Olivares, E. G., and Rossell, C. E. V. (2012). "Steam explosion pretreatment reproduction and alkaline delignification reactions performed on a pilot scale with sugarcane bagasse for bioethanol production," *Ind. Crop. Prod.* 35(1), 274-279. DOI: 10.1016/j.indcrop.2011.07.010

Shahid ul, I., Shahid, M., and Mohammad, F. (2013). "Perspectives for natural product based agents derived from industrial plants in textile applications - A review," *Journal of Cleaner Production.* 57, 2-18. DOI: 10.1016/j.jclepro.2013.06.004

Sun, X., Xu, F., Sun, R., Fowler, P., and Baird, M. (2005). "Characteristics of degraded cellulose obtained from steam-exploded wheat straw," *Carbohyd. Res.* 340(1), 97-106. DOI: 10.1016/j.carres.2004.10.022

Sun, Y., and Cheng, J. (2002). "Hydrolysis of lignocellulosic materials for ethanol production: A review," *Bioresource Technol.* 83(1), 1-11. DOI: 10.1016/S0960-8524(01)00212-7

Vivekanand, V., Olsen, E. F., Eijsink, V. G. H., and Horn, S. J. (2013). "Effect of different steam explosion conditions on methane potential and enzymatic saccharification of birch," *Bioresource Technol.* 127, 343-349. DOI: 10.1016/j.biortech.2012.09.118

Yan, L., Kasal, B., and Huang, L. (2016). "A review of recent research on the use of cellulosic fibres, their fibre fabric reinforced cementitious, geo-polymer and polymer composites in civil engineering," *Compos. Part B-Eng.* 92, 94-132. DOI: 10.1016/j.compositesb.2016.02.002

Zhang, X., Han, G., Jiang, W., Zhang, Y., Li, X., and Li, M. (2016). "Effect of steam pressure on chemical and structural properties of kenaf fibers during steam explosion process," *BioResources* 11(3), 6590-6599.

Zhao, X. G., and Li, A. (2016). "A multi-objective sustainable location model for biomass power plants: Case of China," *Energy* 112, 1184-1193. DOI: 10.1016/j.energy.2016.07.011

Zheng, Y., and Wang, A. (2014). "Kapok fiber: Applications," in: *Biomass and Bioenergy: Applications*, R. K. Hakeem, M. Jawaid, and U. Rashid (eds.), Cham: Springer International Publishing, pp. 251-266. DOI: 10.1007/978-3-319-07578-5.

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