The Properties and Application of an Ultrasonic-Assisted Wheat Straw Pulp having Enhanced Tendency for Ash Formation

Liyan Xing,[a] Ming Xu,[a,b] and Junwen Pu [a,*]

Ultrasonic-assisted pulping technology integrates pulping and bleaching processes in one reactor with good yields performance of approximately 60% to 70%. The properties of ultrasonic-assisted wheat straw pulp were compared, *i.e.*, composition, strength, and whiteness, with those of other conventionally pulps. The ash content of ultrasonic wheat straw pulp (the content is 27.81%) was much higher than that of traditional wheat straw pulp (the content was approximately 15%). Upon comparison with the ash content of the raw material, X-ray diffraction analysis, and thermogravimetry, the authors believe that some heat-resistant material was produced during the ultrasonic pulping process. The strength and whiteness performance of ultrasonic wheat straw pulp was better than that of traditional wheat straw pulp and was close to that of reed soda-anthraquinone (soda-AQ) pulp. Offset paper was successfully made using ultrasonic wheat straw pulp.

Keywords: Ultrasonic-assisted wheat straw pulp; High yield; Tensile strength; Heat-resistant

*Contact information: a: Beijing Key Laboratory of Lignocellulosic Chemistry, Beijing Forestry University, 100083, P.R. China; b: Beijing Zhongke Audible and Ultrasound Technology Institute, 100083, P.R. China; *Corresponding author: 13681243864@126.com*

INTRODUCTION

Over the past few decades, the increasing consumption of paper and paperboard products has resulted in a rising demand for wood raw materials in the pulp and papermaking industry. Various non-wood fibers, such as wheat straw, rice straw, and reeds, have been used as alternative materials in Asia, especially in China. The soda-AQ pulping method is the dominant process used to produce pulp from non-wood fibers. However, the traditional pulping methods have many shortcomings, including poor alkali recovery performance, high-energy consumption, and more wastewater emission. All of these drawbacks have become major obstacles in the development of non-wood pulping processes.

Ultrasonic technology has shown broad application prospects in the pulp and papermaking industry. The applications of this technology in the process of pulping (Healey *et al.* 1956; Subhedar and Gogate 2014), beating, preparation of nanocrystalline cellulose (Li *et al.* 2011) and carboxymethyl cellulose (Hivechi *et al.* 2015), and treatment of recycled fiber (Tatsumi *et al.* 2000; Subhedar *et al.* 2015) have been well documented. It has been proposed that the ultrasound-assisted reactions in aqueous solutions involve free-radical mechanisms because of the formation of hydroxyl radicals (HO·) with oxidation capabilities (Weissler *et al.* 1959, 1962; Anbar and Pecht 1964; McKee *et al.* 1977; Reifsneider and Spurlock 1973; Mead *et al.* 1975). These free-radical formations have been confirmed *via* spin-trapping techniques (Makino *et al.* 1982, 1983). Ultrasound

produces transient cavities, and these cavities successively collapse. During the final stages of collapse, the partial temperature and pressure in the cavities may even exceed 3000 K and 1000 bar, respectively, leading to the dissociation of water into hydroxyl free radicals (HO·) and hydrogen atoms. Furthermore, the resultant hydroxyl free radicals (HO·) can be transformed into H_2O_2 (Apfel 1981; Riesz and Christman 1986). Many studies (Kim *et al.* 2001; Kubo *et al.* 2003; Hata *et al.* 2005; Xiaojun and Guangjie 2010; Johnson and Faber 2011) have confirmed that cellulosic carbons could be converted to amorphous carbon or graphitic carbon at high temperature and pressure. The partial high temperature and pressure are critical to the ultrasonic pulping process and potentially contribute to good separation of the fibers and the bleaching of the pulp (He *et al.* 2017). Meanwhile, they could also cause pulp graphitization.

The ultrasonic pulping process is conducted in high-capacity equipment within the liquid phase, and its effect is based on the subsequent processes of cavitation, mechanical, and thermal processes. Compared with the traditional chemical pulping technologies, ultrasonic technology integrates the pulping and bleaching processes inside one reactor. Therefore, other processes, such as the soda recovery process, bleaching process, bleaching solution preparation step, and coal-fired steam supply system, are eliminated. The ultrasonic pulping method produces a high yield of approximately 60% to 70% (while the yield of the soda-AQ pulping method is approximately 40%). The fixed investment, the operating cost, and the comprehensive energy consumption of ultrasonic pulping technology are much lower than the traditional pulping methods. The amount of chemical oxygen demand (COD), biochemical oxygen demand (BOD), and suspended solids (SS) in wastewater sharply drops, and the formation of absorbable organic halogens (AOX), dioxins, *etc.*, is prevented. Additionally, the strength and optical properties of ultrasonic pulp are equal to and even higher than those of soda-AQ pulping.

In this study, the preparation and properties of ultrasonic wheat straw pulp were studied through scanning electron microscopy (SEM), X-ray diffraction analysis, thermogravimetric analysis, Fourier transform infrared (FTIR) spectral analysis, and strength testing. Additionally, the process parameters of the offset paper that was prepared were also examined.

EXPERIMENTAL

Materials

Ultrasonic-assisted wheat straw pulp (UWP) with a Kappa number of 11.2 was formed *via* ultrasonic pulping technology. The wheat straw was first cut into small pieces with lengths of approximately 1 to 3 cm) and pulped in a covered ultrasonic reactor (Self-products, volume 30 L). The reaction was performed at a mass concentration of 8% to 20%, a time of 2 h, and an ultrasonic frequency of 18 kHz, at room temperature and pressure with constant stirring. In the late reaction stages, the temperature rose to approximately 70 to 90 °C. The chemical additives used consisted of ammonia (5%), sodium dodecyl sulfonate (0.2%), silicate (2%), and magnesium sulfate (1%). The percentages of these were relative to the dried material. All chemicals are commercial products and were used as received. The reaction equipment that was used is shown in Fig. 1.

Reed soda-AQ pulp (RSP) was supplied by Xinjiang Bohu Reed Co., Ltd (Korla, China). Eucalyptus bleached pulp (EBP) and aspen alkaline peroxide mechanical pulp (APMP) were supplied by Sun Paper Co., Ltd (Yan Zhou, China).

The China National Pulp and Paper Research Institute supplied the traditional wheat straw pulp (TWP) with a Kappa number of 10.5, made by the traditional soda-AQ pulping process.

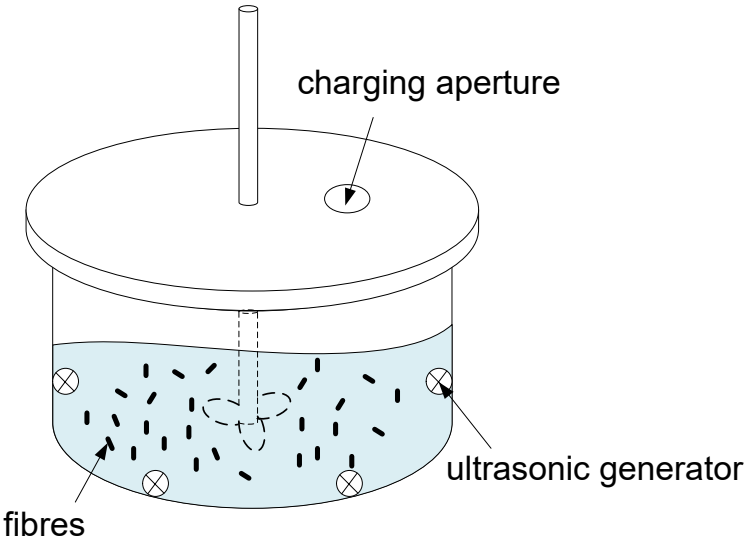

Fig. 1. Simplified reaction device for the ultrasonic pulping process

All of these pulps were stored at a temperature of 5 °C. The UWP, RSP, EBP, and APMP were beaten to 40 °SR freeness during the experiment. All of the chemicals used were commercial products and used without modification.

Methods

The chemical compositions and Kappa number of UWP were measured according to ISO standards (GB/T2677.20 (1995); GB/T742 (2008); GB/T2677.6 (1994); GB/T2677.6 (1994); GB/T10337 (2008); GB/T745 (2003)).

The SEM analysis was performed on the US 8020 model scanning electron microscope (Hitachi, Kyoto, Japan) by grinding the pulp samples into a fine powder. The ground pulp was then adhered to the sample stage and sprayed with gold (Hitachi E1030 Sputter Coater, Kyoto, Japan). Then, the surface morphology of fibers was observed.

The X-ray diffraction (Shimadzu model XRD 6000, Kyoto, Japan) analysis was performed on similar pulp samples, and the scanning speed, step size, and scanning ranges were 0.5 s/step, 0.04°, and 5° to 60°, respectively (Jiebin *et al.* 2011).

The thermal behavior of pulp was explored using a STA449 thermal analyzer (Netzsch, Germany), and the thermogravimetric curves were obtained at 5°C to 500 °C in air with a heating rate of 10 °C/min.

The FTIR spectral analysis was performed on similar pulp samples. For this purpose, approximately 2.0 mg of pulp powder was mixed with 100 mg of KBr. The mixture was pressed into a 13-mm-diameter tablet. The tablet was tested to obtain the FTIR spectra using a VERTEX 70V spectrometer (Bruker, Germany), equipped with a deuterated triglycine sulfate (DTGS) detector. The scanning range used was 4000 to 500 cm^{-1}, with a resolution of 4 cm^{-1}, and a total accumulation of 32 scans.

The pulps UWP, RSP, EBP, APMP, and TWP were beaten to 40 °SR freeness. Handsheets (60 g/m^2) were formed using a KRK semi-auto sheet machine (Kyoto, Japan). Then, the physical and optical properties of the UWP were compared with the other four pulps.

After repeated exploration, the optimum process parameters and the additive amounts of offset paper were obtained as follows: the freenesses of UWP and EBP were 45° SR and 35° SR, respectively, and the UWP and EBP additive ratio was 1:1. Moreover, the order of the addition of chemical additives was calcium carbonate (8.0% to 12.0%, $m_{Calcium\ carbonate}/m_{pulp}$), cationic polyacrylamide (CPAM) (0.2%, m_{CPAM}/m_{pulp}), and alkyl ketene dimer (AKD) (0.8%, m_{AKD}/m_{pulp}). Surface sizing was performed on an ST-1 type surface-sizing machine (Beijing, China) under a pressure of 10 kgf/m^2 and at a temperature of 25 °C. During the process, the CS1 surface-sizing agent (the ratio of additive amount was 2 kg/t), aluminum sulfate (concentration of 30%, and the ratio of the additive amount was 2 kg/t), and oxidized starch (concentration of 10%, and the ratio of the additive amount was 25 kg/t) were used. The press polishing was conducted on a KRK Super Calendar (Kyoto, Japan), and the line pressure, pressure roller speed, and steam roll venting pressure were 1.5 to 2.0 kN/cm, 15 m/min, and 80 to 120 kPa, respectively.

And then all kinds of properties of pulp were measured according to ISO standards (GB/T12914 (2008); GB/T455 (2002); GB/T454 (2002); GB/T7974 (2002)). The zeta potential was measured on the FPA Fiber zeta Potential Analyzer (AFG Analytic GMBH, Germany). The pH of the pulps was approximately 7.

RESULTS AND DISCUSSION

Component Analysis of UWP

The composition of the raw materials contributed an important basis for evaluating quality.

Table 1. Component Analysis of UWP

Ash (%)	Extraction from Benzyl Alcohol (%)	Holocellulose (%)	Pentosan (%)	Lignin (%)	Kappa Number
27.81	1.08	66.04	5.28	4.31	11.71

The yield of UWP was approximately 70%, and its Kappa number was 11.71. Table 1 also shows that the lignin content of UWP was between that of chemical pulp and chemo-mechanical pulp.

The ash content of UWP, which was up to 27.81%, was clearly much higher than that of the TWP. The ash content of the raw material (wheat straw) was then measured, which was only about 7%. Hence, it was assumed that all of the ash remained in the pulp. Although the yield of the UWP obtained was 70%, its ash content was only 10%. Even after including the chemical additives, the ash content of UWP did not exceed 15%, which was much lower than the measured value. Therefore, the authors speculated that a new heat-resistant material was produced in the partial temperature and pressure of the ultrasonic pulping process.

SEM of Two Kinds of Pulp

During the ultrasonic pulping process, no strong acid and alkali were used. Therefore, damage to the fibers was trivial and the obtained fiber was more complete. From

Fig. 2, it is evident that there were many protrusions on the fibers of UWP, which may have been due to the deposition of inorganic substances or derivatives.

The fiber surface of the TWP was smoother, but the stratification phenomenon was clear, which weakened the strength of the fiber. This may be one of the reasons that the UWP had better performance compared with the TWP.

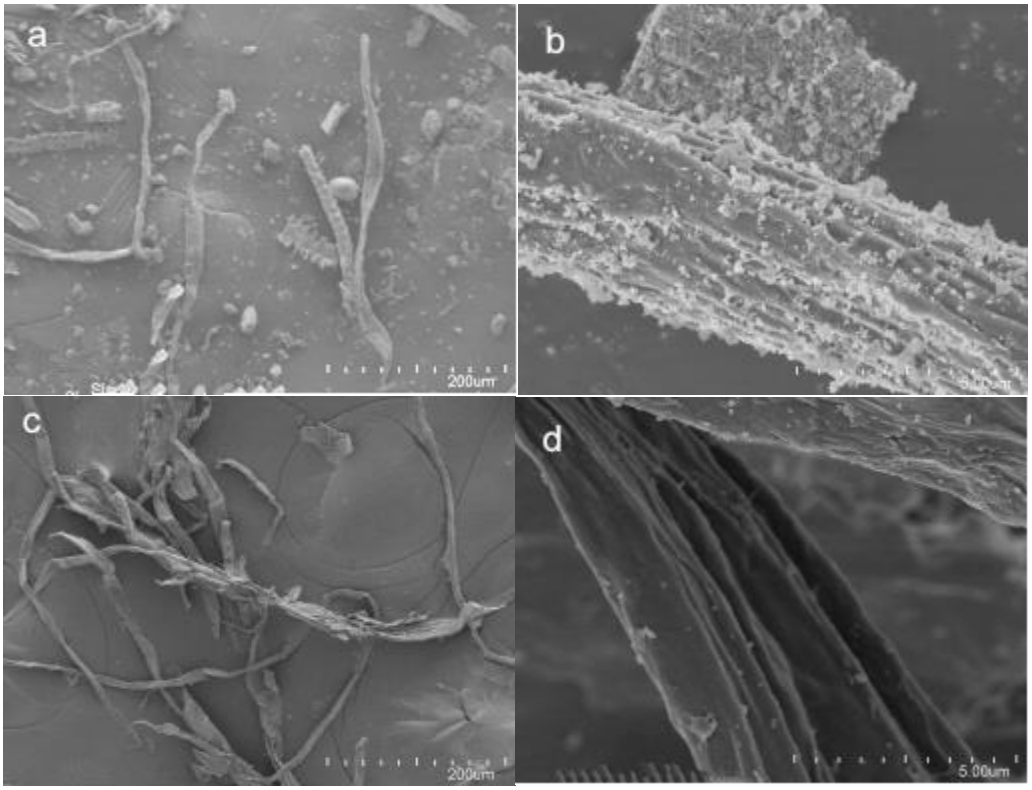

Fig. 2. The SEM of the UWP (a,b) and TWP (c,d)

X-Ray Diffraction Analysis of UWP and TWP

The curves corresponding to UWP and TWP were essentially identical. The Y-axis of such output tends to be different for each specimen, which may be related to the mass and density. The higher mass of the specimen, the stronger will be the signal strength.

Both types of specimen exhibited a typical cellulose-I structure. The crystallinities of UWP and TWP were 35.4% and 32.0%, respectively, which suggests that the orderly arrangement of UWP was stronger than that of TWP, which was one of the reasons for its good performance.

Additionally, there was an obvious peak at $2\theta = 26.5°$ on the curve of UWP that was not present for TWP. This peak appeared repeatedly in several experiments. Therefore, this phenomenon was not a mere coincidence but was due to a new material that was produced during the ultrasonic pulping process. This can be attributed to carbon deposition, as observed by other researchers (Aurbach *et al*. 1999; Sun *et al*. 2008; Hartono *et al*. 2009).

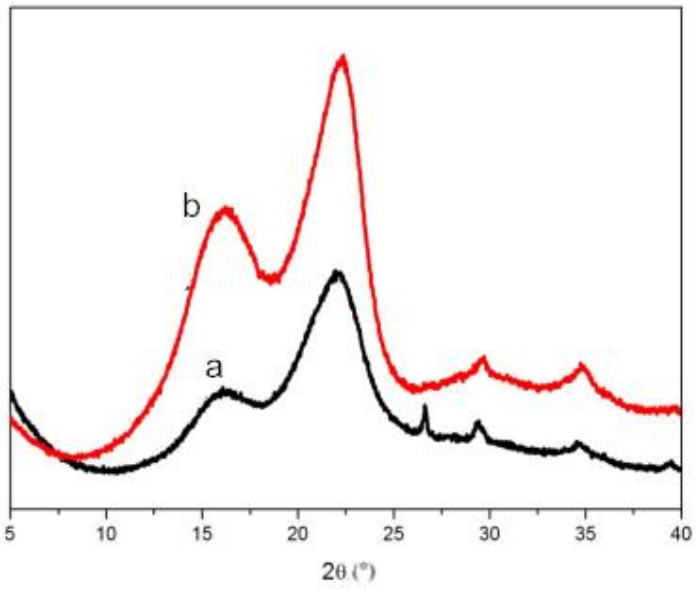

Fig. 3. XRD spectra of the UWP (a) and TWP (b)

Thermogravimetric Analysis of UWP and TWP

The mass loss before 100 °C was due to the evaporation of water. As shown in Fig. 4, both pulps began to decompose at 280 °C and the decomposition was approximately completed around 450 °C.

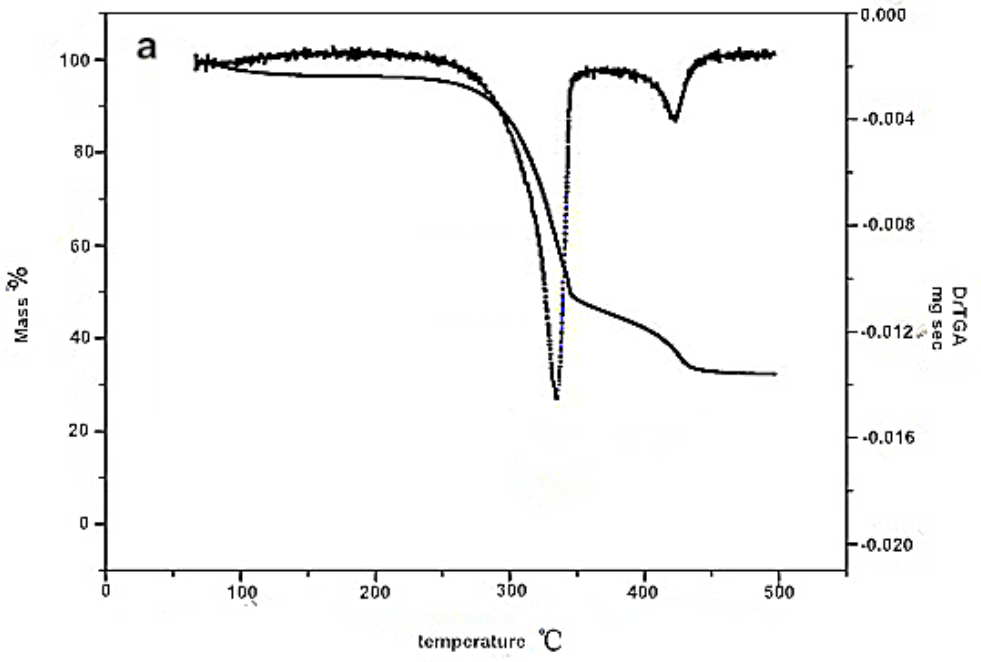

Fig. 4(a). The thermal analysis of UWP

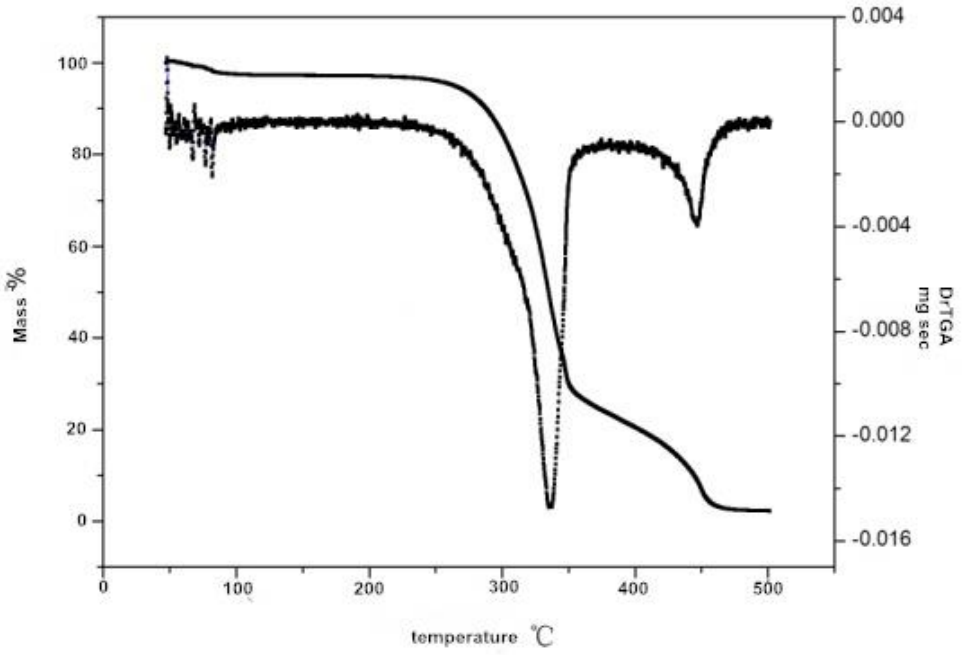

Fig. 4(b). The thermal analysis of TWP

Most of the C and H elements volatilized at this stage. The total loss of UWP and TWP were 70% and 95%, respectively. The final residue obtained from the UWP and TWP were 31% and 2%, respectively, which was consistent with the ash content and further suggests that there was some heat-resistant material formed during the ultrasonic wheat straw pulping process.

FTIR Spectral Analysis of UWP and TWP

Previous FTIR spectral studies (Lu *et al.* 2005) of wheat straw pulp showed that absorption peaks at 1226.6, 2918.2, 1637.5, 1400.3, and 1060.8 cm^{-1} belong to O-H stretching vibration, C-H stretching, water bending, CH_2 symmetric bending, and C-O-C pyran ring skeleton vibrations, respectively.

As shown in Fig. 5, the peak near 804 cm^{-1} in the FTIR spectra of UWP exhibited a strong intensity; however, the corresponding wavelength region in the FTIR spectra of the TWP showed almost no absorption peak. The stretching at 804 cm^{-1} were characteristic peaks of substituted substituents of the benzene ring, mostly from lignin. It was speculated that the lignin content of UWP was larger than that of TWP because of the milder reaction conditions and a smaller amount of chemicals used.

Properties of five kinds of pulp

The pulps RSP, EBP, APMP, and TWP were made *via* traditional pulping methods, and the advantages of UWP were well presented through the comparison with these four kinds of conventional pulps. The properties of UWP, RSP, EBP, APMP, and TWP are presented in Table 2.

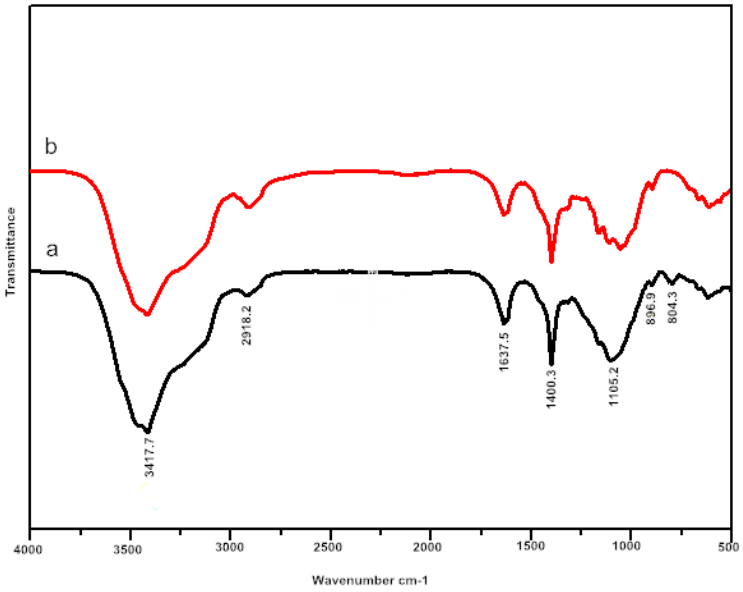

Fig. 5. Infrared spectra of (a) UWP and (b) TWP

Table 2. Pulp Properties of UWP, RSP, EBP, APMP, and TWP

	EBP	RSP	UWP	APMP	TWP
Tensile Index (N·m/g)	41.41	34.35	30.49	9.09	30.1
Tear Index (mN·m^2/g)	3.24	4.83	3.98	1.99	2.95
Burst Index (kPa·m^2/g)	4	3.74	3.08	1.63	2.74
Whiteness (%)	83.5	78.6	77.8	64.7	75.3
Zeta Potential (mV)	-21.3	-22.6	-18.3	-29.3	-24.1

*Notes: EBP: Eucalyptus bleached pulp;
RSP: Reed soda-AQ pulp
UWP: Ultrasonic wheat straw pulp
APMP: Aspen alkaline peroxide mechanical pulp
TWP: Traditional wheat straw pulp

Table 2 indicates that the performance of UWP was better than that of TWP and APMP, and very close to that of RSP. The tensile index of UWP was 30.49 N·m/g, while that of TWP was 30.1 N·m/g, that of APMP was only 9.09 N·m/g, and that of RSP was 34.35 N·m/g. The zeta potential of UWP (-18.3 mV) was higher than that of TWP (-24.1 mV), while the zeta potential of APMP was just -29.3 mV, which corresponded with the conclusions from the strength properties. In comparison with the whiteness of TWP (75.3%), the UWP (without a separate bleaching process) indicated 77.8% whiteness. Additionally, the length and width of UWP were measured and were determined to be 0.754 mm and 29.5 μm, respectively.

In addition, the production costs of UWP were much lower than those for TWP, APMP, and RSP because of its high yield and simple preparation process. If the UWP is used as an alternative for RSP and APMP in the paper and papermaking industry, it would have a great economic benefit.

Table 3. Comparison of Sample and Industry Standard

	Quantitative (g/m^2)	Thick-ness (mm)	White-ness (%)	Opacity (%)	Water absorption (g/m^2)	Tensile index (N·m/g)	Folding -time (s)	Smooth-ness	Printing surface strength (m/s)
Industry Standard (Excellence)	70	0.075	≥80	≥84	≤ (30±5)	≥35	≥15	≥30	≥1
Sample	70	0.078	80.9	93	29.8	54.5	115	40	2.8

The test values of paper reached or exceeded the industry standards for offset paper. The authors strongly believe that the use of UWP instead of TWP to make offset paper would be as economically beneficial.

CONCLUSIONS

1. The ultrasonic-assisted pulping technology integrates the pulping and bleaching processes inside one reactor with a high yield of approximately 60% to 70%.
2. A material having an enhanced tendency to produce a thermostable ash component was produced during the ultrasonic pulping process. This observation will require further research to understand its nature.
3. The UWP had a good performance and the offset paper has been made successfully using UWP. All kinds of parameters reached or exceeded the industry standard.

ACKNOWLEDGMENTS

This work was sponsored by the Special Fund for Beijing Common Construction Project and Beijing Forestry University, Grant No. 2015HXKFCLXY002.

REFERENCES CITED

Anbar, M., and Pecht, I. (1964). "On the sonochemical formation of hydrogen peroxide in water," *The Journal of Physical Chemistr*y 68(2), 352-355. DOI: 10.1021/j100784a025

Apfel, R. E. (1981). "Acoustic cavitation," *Methods in Experimental Physics* 19, 355-411. DOI: 10.1016/S0076-695X(08)60338-5

Aurbach, D., Markovsky, B., Weissman, I., Levi, E., and Ein-Eli, Y. (1999). "On the correlation between surface chemistry and performance of graphite negative electrodes for Li ion batteries," *Electrochimica Acta* 45(1), 67-86. DOI: 10.1016/S0013-4686(99)00194-2

GB/T2677.20 (1995). "Fibrous material - Determination of holocellulose,"

Standardization Administration of China, Beijing, China.
GB/T742 (2008). "Fibrous raw material, Pulp, Paper, and board - Determination of ash," Standardization Administration of China, Beijing, China.
GB/T2677.6 (1994). "Fibrous raw material – Determination of solvent extractives," Standardization Administration of China, Beijing, China.
GB/T2677.6 (1994). "Fibrous raw material – Determination of acid-insoluble lignin," Standardization Administration of China, Beijing, China.
GB/T10337 (2008). "Fibrous raw material – Determination of acid-soluble lignin," Standardization Administration of China, Beijing, China.
GB/T745 (2003). "Pulps – Determination of pentosan content," Standardization Administration of China, Beijing, China.
GB/T12914 (2008). "Paper and board – Determination of tensile properties," Standardization Administration of China, Beijing, China.
GB/T455 (2002). "Paper and board determination of tearing resistance," Standardization Administration of China, Beijing, China.
GB/T454 (2002). "Paper determination of bursting strength," Standardization Administration of China, Beijing, China.
GB/T7974 (2002). "Paper, board and pulp – Measurement of brightness – Diff/Geometry," Standardization Administration of China, Beijing, China.
Hivechi, A., Bahrami, S. H., Arami, M., and Karimi, A. (2015) "Ultrasonic mediated production of carboxymethyl cellulose: Optimization of conditions using response surface method-ology," *Carbohydrate Polymers* 134, 278-284. DOI: 10.1016 /j.carbpol. 2015. 07.045
Hartono, T., Wang, S., Ma, Q., and Zhu, Z. (2009). "Layer structured graphite oxide as a novel adsorbent for humic acid removal from aqueous solution," *Journal of Colloid and Interface Science* 333(1), 114-119. DOI: 10.1016/j.jcis.2009.02.05
Hata, T., Ishimaru, K., Fujisawa, M., Bronsveld, P., Vystavel, T., De Hosson, J., Kikuchi, H., Nishizawa, T., and Imamura, Y. (2005). "Catalytic graphitization of wood-based carbons with alumina by pulse current heating," *Fullerenes, Nanotubes, and Carbon Nanostructures* 13(S1), 435-445. DOI: 10.1081/FST-200039431
Healey, H. J., Patterson, S. S., and Van Huysen, G. (1956). "Pulp reaction to ultrasonic Cavity preparation; Preliminary report," *United States Armed Forces Medical Journal* 7(5), 685.
He, Z., Wang, Z., Zhao, Z., Yi, S., Mu, J., and Wang, X. (2017). Influence of ultrasound pretreatment on wood physiochemical structure. *Ultrasonics Sonochemistry*, 34, 136-141.DOI:10.1016/j.ultsonch.2016.05.035
Jiebin, T., Kefu, C., Jun, X., and Jun, L. (2011). "Isolation and characterization of cellulose from Chinese alpine rush," *CIESC Journal* 6, 040.
Johnson, M. T., and Faber, K. T. (2011). "Catalytic graphitization of three-dimensional wood-derived porous scaffolds," *Journal of Materials Research* 26(1), 18-25. DOI: 10.1557/jmr.2010.88
Kim, D. Y., Nishiyama, Y., Wada, M., and Kuga, S. (2001). "Graphitization of highly crystalline cellulose," *Carbon* 39(7), 1051-1056. DOI: 10.1016/S00086223(00)00221-9
Kubo, S., Uraki, Y., and Sano, Y. (2003). "Catalytic graphitization of hardwood acetic acid lignin with nickel acetate," *Journal of Wood Science* 49(2), 188-192. DOI: 10.1007/s100860300030
Li, W., Wang, R., and Liu, S. (2011). "Nanocrystalline cellulose prepared from softwood

kraft pulp *via* ultrasonic-assisted acid hydrolysis," *BioResources* 6(4), 4271-4281. DOI: 10.15376/biores.6.4.4271-4281

Lu, J., Shi, S. L., Yang, R. N., Niu, M. H., and Song, W. J. (2005). "Modification of reed cellulose microstructure and IT change in enzymatic hydrolysis of reed pulp," *Trans. China Pulp Pap.* 20(2), 85-90.

Makino, K., Mossoba, M. M., and Riesz, P. (1982). "Chemical effects of ultrasound on aqueous solutions. Evidence for hydroxyl and hydrogen free radicals (·OH and ·H) by spin trapping," *Journal of the American Chemical Society* 104(12), 3537-3539. DOI: 10.1021/ja00376a064

Makino, K., Mossoba, M. M., and Riesz, P. (1983). "Formation of and in aqueous solutions by ultrasound using clinical equipment," *Radiation Research* 96(2), 416-421. DOI: 10.1021/ja00376a064

McKee, J. R., Christman, C. L., O'Brien Jr., W. D., and Wang, S. Y. (1977). "Effects of ultrasound on nucleic acid bases," *Biochemistry* 16(21), 4651-4654. DOI: 10.1021/bi00640a019

Mead, E. L., Sutherland, R. G., and Verrall, R. E. (1975). "The ultrasonic degradation of thymine," *Canadian Journal of Chemistry* 53(16), 2394-2399. DOI: 10.1139/v75-338

Reifsneider, S. B., and Spurlock, L. A. (1973). "Chemistry of ultrasound. II. Irradiative behavior of aliphatic aldehydes and carboxylic acids in an aqueous medium," *Journal of the American Chemical Society* 95(2), 299-305. DOI: 10.1021/ja00783a001

Riesz, P., and Christman, C. L. (1986). "Sonochemical free radical formation in aqueous solutions," in: *Federation Proceedings* 45(10), 2485-2492.

Sun, G., Li, X., Qu, Y., Wang, X., Yan, H., and Zhang, Y. (2008). "Preparation and characterization of graphite nanosheets from detonation technique," *Materials Letters* 62(4), 703-706. DIO: 10.1016/J.matlet.2007.06.035

Subhedar, P. B., and Gogate, P. R. (2014) "Alkaline and ultrasound assisted alkaline pre-treatment for intensification of delignification process from sustainable raw-material," *Ultrasonics Sonochemistry* 21(1), 216-225. DOI: 10.1016/j.ultsonch.2013.08.001

Subhedar, P. B., Babu, N. R., and Gogate, P. R. (2015). "Intensification of enzymatic hydrolysis of waste newspaper using ultrasound for fermentable sugar production," *Ultrasonics Sonochemistry* 22, 326-332. DIO:10.1016/j.ultsonch.2014.07.005

Tatsumi, D., Higashihara, T., Kawamura, S. Y., and Matsumoto, T. (2000). "Ultrasonic treatment to improve the quality of recycled pulp fiber," *Journal of Wood Science* 46(5), 405-409. DOI: 10.1007/BF00776405

Weissler, A. (1959). "Formation of hydrogen peroxide by ultrasonic waves: Free radicals," *Journal of the American Chemical Society* 81(5), 1077-1081. DOI: 10.1021/ja01514a015

Weissler, A. (1962). "Ultrasonic hydroxylation in a fluorescence analysis for microgram quantities of benzoic acid," *Nature* 193, 1070. DOI: 10.1038/1931070a0

Xiaojun, M., and Guangjie, Z. (2010). "Preparation of carbon fibers from liquefied wood," *Wood Science and Technology* 44(1), 3-11. DOI: 10.1007/s00226-009-0264-3

Article submitted: October 16, 2016; Peer review completed: November 28, 2016; Revised version received and accepted: November 30, 2016; Published: December 7, 2016.
DOI: 10.15376/biores.12.1.871-881