**BIODIVERSITAS**
Volume 23, Number 3, March 2022
Pages: 1490-1494

ISSN: 1412-033X
E-ISSN: 2085-4722
DOI: 10.13057/biodiv/d230337

# Production and characterization of *Imperata cylindrica* paper using potassium hydroxide as pulping agent

**ZULIANA MADUNG, SABRINA SOLOI♥, MOHD HAFIZ ABD MAJID, MOHD SANI SARJADI**
Faculty of Science and Natural Resource, Universiti Malaysia Sabah. Jl. UMS, Kota Kinabalu 88400, Sabah, Malaysia. Tel./fax.: +60-195308613, ♥email: sabrinas@ums.edu.my

Manuscript received: 17 January 2021. Revision accepted: 24 February 2022.

**Abstract.** *Madung Z, Soloi S, Majid MHA, Sarjadi MS. 2022. Production and characterization of* Imperata cylindrica *paper using potassium hydroxide as pulping agent. Biodiversitas 23: 1490-1494.* Non-woody plants have become an alternative fiber in paper production as woods, the primary sources of papers have become limited due to increasing demand of paper year by year. *Imperata cylindrica* or cogon grass as the source of fiber for the pulp and paper industry in replacing the wood fiber because it has high cellulose and low lignin content. In this study, cogon grasses were treated with various concentrations of potassium hydroxide, KOH (5%, 8%, 10% and 12%). The effects of alkali treatment on the properties of the papers were investigated in the present study. Scanning Electron Microscope (SEM) image of the paper sheet indicates that the amount of cellulose fiber and other materials were lessening toward a higher concentration of KOH treatment. This explained that most of the lignin was entirely removed and some cellulose probably degraded at higher KOH concentrations. Fourier Transform Infa-Red (FTIR) analysis showed that the peaks at range 1650 $cm^{-1}$-1630 $cm^{-1}$ attributed to the presence of $v$(C=C) aromatic ring of lignin disappeared at 10% and 12% of KOH. The mechanical strength of the papers decreased with an increasing concentration of KOH, while 5% of KOH produced the highest value of tensile strength.

**Keywords:** *Imperata cylindrica*, non-woody plant, potassium hydroxide (KOH) pulping, surface morphology

## INTRODUCTION

Paper is mainly known as an essential material and is omnipresent all around the world. The demand for paper is steadily increasing year by year and is expected to double by 2050 (Höller et al. 2021). Globally, papers are used for packaging, writing and printing, sanitary, newsprint and others. The non-wood-based paper has slowly become attention nowadays as people are starting to be concerned about the environmental effects due to deforestation (Abd El-Sayed et al. 2020). (Kamoga et al. 2013) mentioned that countries like China and India had taken an initiative to use non-woody plants such as straw, rice straw, wheat, hemp, bagasse, sugarcane, bamboo and switchgrass to replace woods in their paper production (Judt 1993; Tsalagkas et al. 2021). In Malaysia, previous studies by (Kassim et al. 2016) revealed that *I. cylindrica* has the most effective characteristics as a pulp source as compared to other non-woody plants like switchgrass and elephant grass due to its low lignin content (5.67%), solubility in hot water and rich with long thin fiber which is essential for mechanical properties of paper. In addition, *I. cylindrica* grass is a perennial and aggressive type of plant because it is tolerant of shade, poor soils, high salinity, moisture and drought (Mohd Kassim et al. 2015). In addition, it is also considered a plant without any economic values, which can also be found easily along the roadside throughout Malaysia.

In the papermaking industry, mechanical or chemical pulping can be employed to remove the lignin and other non-cellulosic components. However, chemical pulping has become the leading choice in most industries as compared to mechanical pulping. The paper produced has a higher strength due to delignification process that occurs through chemical method (Mcdonald et al. 2004). Chemical pulping consists of several types such as sulfide, kraft, alkaline and organosolv. Alkali treatment is the first chemical process patented in 1845 (Doherty and Rainey 2006). This process involved using an alkali solution to detach lignin from the cellulose and hemicellulose fiber to obtain pulp. The critical step in this process is delignification which refers to the removal of lignin by the alkalinity of the pulping liquor that weakens the intermolecular hydrogen bond between the cellulose and the lignin, allowing for easy detachment. The efficiency of delignification mainly depends on the chemical ability of the pulping agent to break ether bonds in lignin into molecules that are small enough to dissolve in the liquor (Doherty and Rainey 2006).

Sodium hydroxide (NaOH) was the most common alkali solution used to remove lignin. However, a recent study using potassium hydroxide (KOH) as pulping solution showed that KOH treatment produces better pulp yield and greater brightness of paper when compared to NaOH treatment (Sutradhar et al. 2018; Popy et al. 2020). Therefore, this study was conducted to investigate the properties of the *I. cylindrical* paper using KOH as a pulping agent.

## MATERIALS AND METHODS

### Materials and instrument

*I. cylindrica* was collected at the area of Kg Rampayan, Kota Kinabalu, Sabah. The samples were cut into 4-5 cm and washed with tap water to remove dirt and impurities and it was stored at room temperature until further used. All chemicals used were analytical grade.

### Pulping process and paper making

The cogon grass was treated with various concentrations of KOH (5%, 8%, 10% and 12%) at 100°C with a 75-minute pulping time. Then, the black liquor solution containing the pulp was let to cool and washed with running water to clean the excess KOH. The treated cogon grasses were then ground to get a uniform pulps slurry. The pulps were separated from water using a sieve before being manually compressed by a dry cloth so that a uniform sheet of paper can be formed using a mold and deckle. The compressed pulps were then dried under the sun for approximately 3 hours until a constant weight was obtained. The dried paper was labeled as S1, S2, S3 and S4, respectively, based on their concentration.

### Surface morphology

The surface morphology of the paper was examined using a Hitachi S 3400 N Scanning Electron Microscope (SEM) at 100x and 500x of magnification level. A small piece of the sample was first coated with a thin layer of gold before analyzed to improve the image quality.

### Fourier Transform Infra-Red (FTIR) analysis

The presence of important functional groups of the cellulose and lignin were analyzed using FTIR Spectrum 100. The IR spectra of all samples were analyzed at the wavelength of 4000-400 $cm^{-1}$.

### Tensile strength test

GOTECH AI - 7000 M was used to measure the tensile strength of paper according to TAPPI T 494 om-01. The samples were cut into 1.5 cm x 1.0 cm size and fastened by two clamps extended at a constant speed of 2 mm/min until the papers were pulled apart. The average reading of each tensile test was calculated and recorded.

### Tearing resistance test

Tearing test was done based on TAPPI T 414 om-98 using a DC-SLY13K tearing test machine. A 6.3 cm x 7.5 cm size of paper was positioned and clamped in place before a pendulum was released from a 4.3 cm far from the edge of the paper to create a slit on it. The tearing force needed to rupture the sample was recorded. The grammages of S1, S2, S3 and S4 were calculated respectively to determine the tearing index according to Equation 1 and Equation 2 (Theliander 2009):

$$\text{Grammage } (g/m^2) = \frac{Weight\ (g)}{Length \times Width\ (m^2)} \quad \text{Equation 1}$$

$$\text{Tearing Index } (mN.m^2/g) = \frac{Tearing\ force\ (mN)}{Grammage\ of\ sample\ (g/m^2)} \quad \text{Equation 2}$$

## RESULTS AND DISCUSSION

### Delignification process

The major reaction involved during alkaline treatment is the dissociation of lignin from sugar molecules in hardwood and softwoods. According to (Kassim et al. 2016), the lignin content of *I. cylindrica* is considerably low, with only 5.67%. Lignin is the third lignocellulosic component which contains a profuse number of carbons after cellulose. Four types of bonds made up linkages between individual components in lignin which are ester bond, ether bond, C-C bond and hydrogen bond (Tarasov et al. 2018). Most of the components, especially lignin, are removed through delignification to obtain pure pulps that consist of cellulose and some hemicelluloses fibers during the treatment process. The key reaction in the delignification is the cleavage of the ether bond (Soloi and Hou 2019). Chen and Wang 2017 classified delignification into initial, bulk and residual stages. At the initial stage of KOH treatment, the ether bonds which are phenolic α-O-4 and some ß-O-4 linkages of lignin are cleaved. In the bulk stage, the most rapid reaction is the breakage of ß-O-4 linkages of lignin, as shown in figure 1. While in the residual stage, the rest of the C-C bond linkages are cleaved, and the carbohydrates are degraded. Furthermore, in a high alkaline state, there are particular bonds in lignin that are cleaved due to the presence of plenty $OH^-$ ions which are α-aryl ether bonds in phenolic units and ß-aryl ether bonds in non-phenolic units (Brännvall 2017).

### Physical observation

The brightness of the paper reflects the efficiency of lignin removal by KOH. As shown in figure 2, the papers became brighter when higher KOH concentrations are used. The factor that leads to the change of brightness in papers is the presence of lignin. When lignin content is low, the paper will appear brighter. Moreover, when the papers are pressed and folded by hand, paper using 10% and 12% KOH are softer than paper using 5% and 8% KOH. A higher concentration of KOH soften cellulose fiber. The color of the paper produced is yellowish since no bleaching agent like hydrogen peroxide ($H_2O_2$) was used in this study.

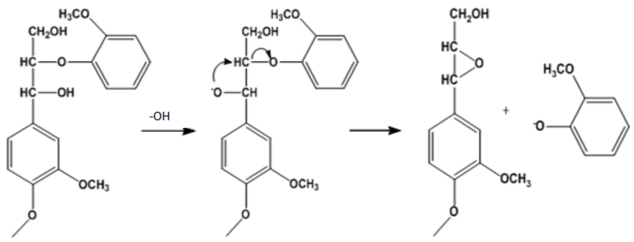


**Figure 1.** Cleavage of β-O-4 linkages of lignin at the bulk stage of the delignification process

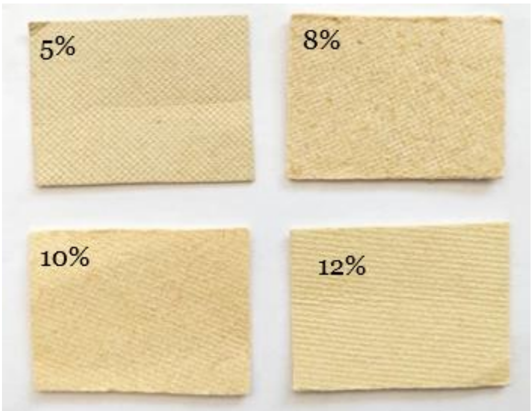


**Figure 2.** Physical observation of cogon grass paper prepared using different concentrations of potassium hydroxide (KOH)

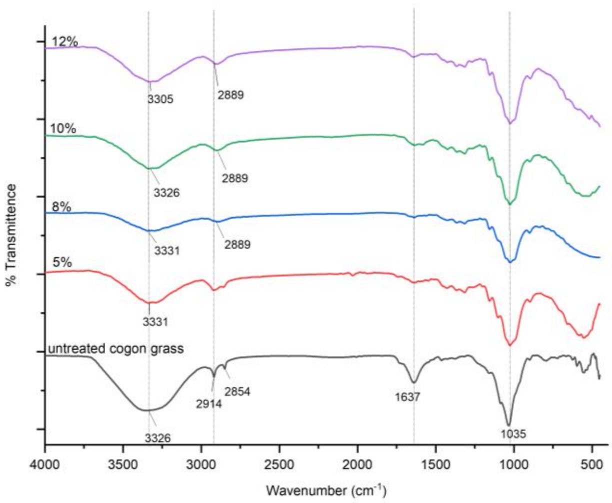


**Figure 3.** FTIR spectra of *I. cylindrica* paper sheet

## Fourier Transform Infra-Red (FTIR) analysis

The delignification efficiency is crucial in preparing the pulp for paper (Laftah and Rahaman 2015). High delignification means a better appearance and properties of the paper produced. As lignin is a complex structure of the polyphenolic compound, the most prominent functional group that indicates the delignification occurs efficiently is the absence/presence of C=C stretching aromatic ring, which usually appears with strong intensity at around 1609-1640 $cm^{-1}$ (Ibrahim et al. 2007; Todorciuc et al. 2009) and bending vibration inside the aromatic ring at 1038-1035 $cm^{-1}$ (Ibrahim et al. 2007; Todorciuc et al. 2009). Based on the spectra in Figure 3, those peaks were absent after the cogon grass was treated with 5% KOH. This implies that KOH was very effective in breaking the cogon grass lignin into its phenylpropane monomer. Another prominent peak that also resembles the presence of lignin is the CH stretching of $CH_2$, $CH_3$ and $CH_3O$ group (Todorciuc et al. 2009; Liu et al. 2014) and CH vibration of aromatic methoxyl group (Lupoi et al. 2014; Sathawong et al. 2018) at 2914 $cm^{-1}$ and 2854 $cm^{-1}$ respectively as shown in the untreated cogon grass spectrum. Meanwhile, for the cogon grass paper, the weak intensity observed at around 2889 $cm^{-1}$ was due to the CH methylene stretching of all the cellulose and hemicellulose hydrocarbon constituents (Hospodarova et al. 2018). The absence of 2914 $cm^{-1}$ indicates that the lignin was entirely removed during the treatment. The broad and strong intensity at the range 3300 $cm^{-1}$ corresponds to the stretching vibration of O-H groups owned by the cellulose chain. As the KOH concentration increased, the intensity peak was reduced due to the cellulose chain's degradation during the delignification.

## Surface morphology

The surface morphology of the cogon grass paper is shown in Figure 4. The cellulose fiber of cogon grass appears to be a high abundance of long and thin fibers, as in agreement with previous study (Mohd Kassim et al. 2015). Some broken fibers and larger voids between fibers were also observed as the percentage of KOH increased. These explained that different concentrations of KOH give different levels of treatment toward cogon grass fibers. Generally, non-cellulosic materials such as hemicelluloses and lignin are more soluble in an alkaline condition. So, more materials will be removed when a strong alkali is applied to cogon grass. This matter had caused voids between fibers due to the degradation of some cellulose in high alkali treatment and could affect the strength of paper (Przybysz et al. 2016). This is because, better strength properties of paper can be achieved from a strong inter-bonded membrane between cellulose and hemicelluloses (Bochek 2003). During the manual pressing process of paper, the fibers are drawn toward each other, thus creating a more robust fibers network that contributes to good paper strength. Karlsson (2010) stated that hydrogen bonds between cellulose and hemicellulose are a major force that built up a paper's web. These bonds are mainly formed between the hydroxylic group of cellulose and hemicelluloses fibers where the hydrogen oscillates two oxygen atoms. Therefore, a small amount of those fibers can lead to the low strength of papers.

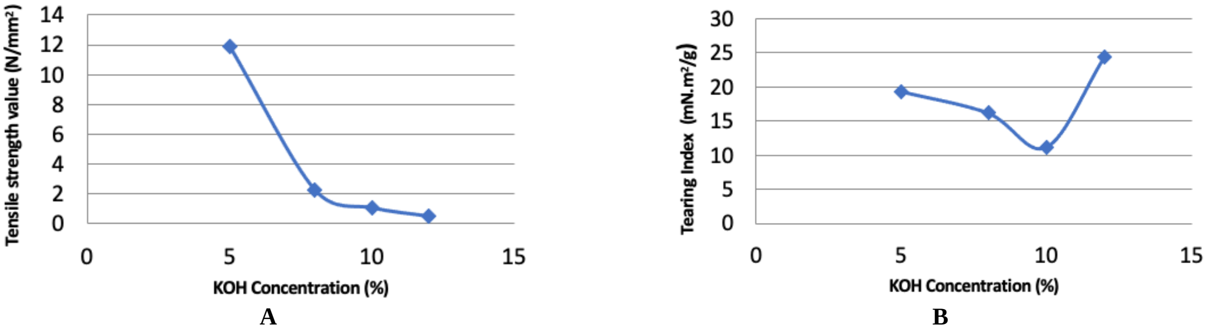


**Figure 4.** Graph of tensile strength (A) and tearing index (B) of *I. cylindrica* paper sheet

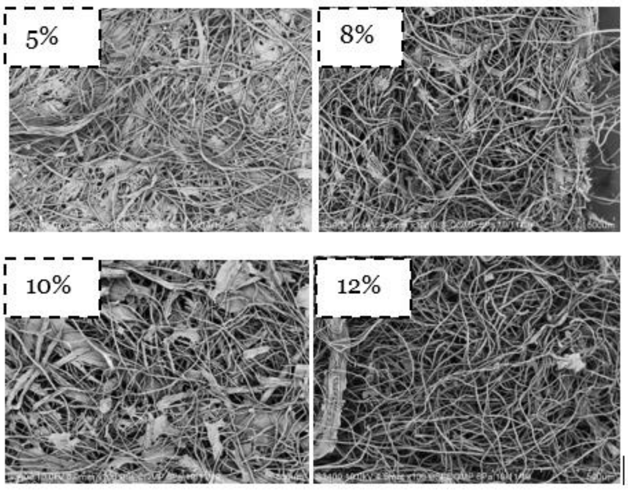


**Figure 5.** SEM images of cogon grass paper at 100x magnification

### Mechanical properties

The result plotted in figure 5 showed an inversely proportional line of tensile strength value against the KOH concentration. When the sample was treated with 5% KOH, it shows an optimum value of 11.90 N/mm$^2$. This happened due to the removal of impurities and lignin during the delignification process that made the bond between fibers strong (Oushabi et al. 2017). However, the trend seems decreasing as the KOH concentration was raised to 8% (2.23 N/mm$^2$), 10% (1.04 N/mm$^2$) and 12% (0.47 N/mm$^2$). (Zaman et al. 2009) mentioned that the tensile strength test is generally dependent on bonding and individual fibers strengths. At the rupture area of the samples, some of the fibers are broken and others are pulled out from the fiber networks (Theliander 2009). In a concentrated alkaline condition, (Brännvall 2017) stated that $OH^-$ ions in KOH probably attack the cellulose, reducing sugar and peels off the terminating sugar units, leading to the dissolution of cellulose in cooking liquor. Hence, the amount of cellulose is lessening and the bonding between fibers become weaker due to degradation, making the paper easier to be ruptured and consequently, gives a low tensile strength of paper.

In conclusion, various concentrations of alkali treatment influence the paper quality. The chemical and mechanical strength of paper produced decreases when a high KOH concentration is used. Results from SEM show that voids between fibers become bigger with increasing KOH percentage. The spectra from FTIR analysis explain that lignins can be fully eliminated by using as low as 5% KOH. The highest tensile strength was observed using 5% of KOH, while 12% KOH recorded the highest tearing index.

## ACKNOWLEDGEMENTS

The authors would like to acknowledge Universiti Malaysia Sabah for FRGS RACER-25 2019 research grant and the Faculty of Sains and Natural Resource for the laboratory facility.

## REFERENCES

Abd El-Sayed ES, El-Sakhawy M, El-Sakhawy MAM. 2020. Non-wood fibers as raw material for pulp and paper industry. Nord Pulp Pap Res J 35 (2): 215-230. DOI: 10.1515/npprj-2019-0064.

Bochek A. 2003. Effect of hydrogen bonding on cellulose solubility in aqueous and nonaqueous solvents. Russ J Appl Chem 6 (11): 1711-1719. DOI: 10.1023/B:RJAC.0000018669.88546.56.

Brännvall E. 2017. The limits of delignification in kraft cooking. BioResources 12: 2081-2107. DOI: 10.15376/biores.12.1.Brannvall.

Chen H, Wang L. 2017. Pretreatment Strategies for Biochemical Conversion of Biomass. In: Chen H, Wang L. (Eds.). Technologies for Biochemical Conversion of Biomass. Academic Press, Oxford.

Doherty B, Rainey T. 2006. Bagasse Fractionation by The Soda Process. In: Hoghart DM (Eds.). Proceeding of The Australian Society of Sugar Cane Technologist 2006, Australia.

Höller M, Lunze A, Wever C, Deutschle A, Stücker A. 2021. Meadow hay, *Sida hermaphrodita* (L.) Rusby and *Silphium perfoliatum* L. as potential non-wood raw materials for the pulp and paper industry. Ind Crops Prod 67: 113548. DOI: 10.1016/j.indcrop.2021.113548.

Hospodarova V, Singovszka E, Stevulova N. 2018. Characterization of cellulosic fibers by FTIR spectroscopy for their further implementation to building materials. Am J Analyt Chem 9 (6): 303-310. DOI: 10.4236/ajac.2018.96023.

Ibrahim MNM, Yusof NNM, Hashim A. 2006. Comparison studies on soda lignin and soda-anthraquinone lignin in terms of physico-chemical properties and structural features. J Appl Sci 6 : 292-296. DOI: 10.3923/jas.2006.292.296.

Zaman I, Ismail AE, Awang MK. 2009. Influence of fiber volume fraction on the tensile properties and dynamic characteristics of coconut fiber reinforced composite. J Sci Technol 1 (1): 55-78.

Judt M. 1993. Non-wood plant fibres, will there be a come-back in paper-making?. Ind Crops Prod 2 (1): 51-57. DOI: 10.1016/0926-6690(93)90011-W.

Kamoga OLM, Byaruhanga JK, Kirabira JB. 2013. A review on pulp manufacture from non wood plant materials. Intl J Chem Eng Appl 4 (3): 144-148 DOI: 10.7763/IJCEA.2013.V4.281.

Karlsson HK. 2010. Strength Properties of Paper produced from Softwood Kraft Pulp : Pulp Mixture, Reinforcement and Sheet Stratification. [Thesis]. Karlstad University. [Sweeden]

Kassim ASM, Aripin AM, Ishak N, Zainulabidin MH. 2016. Potential of cogon grass (*Imperata cylindrica*) as an alternative fibre in paper-based industry. ARPN Journal of Engineering and Applied Sciences 11(4): 2681-2686.

Laftah WA, Rahaman WAWA 2015. Chemical pulping of waste pineapple leaves fiber for kraft paper production. J Mater Res Technol 4 (3) : 254-261. DOI: 10.1016/j.jmrt.2014.12.006.

Liu Y, Hu T, Wu Z, Zeng G, Huang D, Shen Y, He X, Lai M, He Y. 2014 Study on biodegradation process of lignin by FTIR and DSC. Environ Sci Pollut Res 21 (24): 14004-14013. DOI: 10.1007/s11356-014-3342-5.

Lupoi JS, Singh S, Simmons BA, Henry RT. 2014. Assessment of lignocellulosic biomass using analytical spectroscopy: An evolution to high-throughput techniques. Bioenerg Res 7: 1-23 DOI: 10.1007/s12155-013-9352-1.

Mcdonald D, Miles K, Amiri R. 2004 The Nature of the Mechanical Pulping Process. Pulp&Paper, Canada.

Mohd Kassim AS, Ashuvila MA, Ishak N, Zainulabidin MH. 2015. Cogon grass as an alternative fibre for pulp and paper-based industry: on chemical and surface morphological properties. In: International Integrated Engineering Summit 2014. Trans Tech Publications Ltd AMM 773-774: 1242-1245. DOI: 10.4028/www.scientific.net/AMM.773-774.1242.

Oushabi A, Sair S, Hassani FO, Abboud Y, Tanane O, Bouri AL. 2017. The effect of alkali treatment on mechanical, morphological and thermal properties of date palm fibers (DPFs): Study of the interface of DPF-Polyurethane composite. S Afr J Chem Eng 23: 116-123. DOI: 10.1016/j.sajce.2017.04.005s.

Popy RS, Nayeem J, Arafat KMY, Rahman MM, Jahan MS. 2020. Mild potassium hydroxide pulping of straw. Curr Opin Green Sustain Chem 3: 100015. DOI: 10.1016/j.crgsc.2020.100015.

Przybysz P, Dubowik M, Kucner MA, Przybysz K, Buzała KP. 2016. Contribution of hydrogen bonds to paper strength properties. PloS One 11 (5): e0155809. DOI: 10.1371/journal.pone.0155809.

Sathawong S, Sridach W, Techato K. 2018. Lignin: Isolation and preparing the lignin based hydrogel. J Environ Chem Eng 6 (5): 5879-5888. DOI: 10.1016/j.jece.2018.05.008.

Soloi S, Hou EKZ. 2019. The potential of oil palm leaf fibre in paper-making industry. J Physics Conf Ser 1358: 012005.

Sutradhar S, Sarkar, A, Nayeem J, Jahan M, Tian C. 2018. Potassium hydroxide pulping of four non-woods. Bangladesh J Sci Ind Res 53: 1-6. DOI: 10.3329/bjsir.v53i1.35903.

Tarasov D, Leitch M, Fatehi P. 2018. Lignin-carbohydrate complexes: Properties, applications, analyses, and methods of extraction: A review. Biotechnol Biofuels 11: 269-269. DOI: 10.1186/s13068-018-1262-1.

Todorciuc T. 2009. Characterization of non-wood lignin and its hydoxymethylated derivatives by spectroscopy and self-assembling investigations. Cellul Chem Technol 43 (9-10): 399-408.

Tsalagkas D, Börcsök Z, Pásztory Z, Gogate P, Csoka L. 2021. Assessment of the papermaking potential of processed *Miscanthus* × *giganteus* stalks using alkaline pre-treatment and hydrodynamic cavitation for delignification. Ultrason Sonochem 72: 105462. DOI: 10.1016/j.ultsonch.2021.105462.