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Physico-chemical properties of fibrous pulp produced by neutral sulfite digesting of birch sawdust

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Abstract: *Physico-chemical properties of fibrous pulp produced by neutral sulfite digesting of birch sawdust.* The aim of this work was to find differences in chemical composition and strength properties of fibrous pulp obtained after digesting at different conditions. The confocal microscopy and ROI method (Region of Interest) were applied to observe the delignification process. The results of confocal microscopy indicate that preimpregnations of sawdust may have an equalization effect on the distribution of lignin through the pulp fibers.

Keywords: NSSC, pulp, delignification, confocal microscopy

INTRODUCTION

Although, modern industrial papermaking uses more recycled fibers to manufacture paper products, this raw material is still not capable of eliminating virgin fibers as a main component of important paper grades. Packaging grades such as liners and fluting that make up corrugated board which is then used for food packaging, can be an example. Such products need to meet high hygienic conditions and have high strength properties. An internal layer of a corrugated medium, called fluting, is a structure of a packaging and is important to deliver properties on the expected level e.g. high tensile stiffness. Such properties are shown by NSSC fluting, manufactured with high content of pure, short fibers from hardwood treated in Neutral Sulphite Semi Chemical (NSSC) process. This high yield process and fibrous pulp have high content of residual lignin. Strength properties are created during the process of wooden chips cooking so a thorough knowledge of all physic-chemical processes of semichemical cooking allows to obtain fibers with properties expected by papermakers.

The aim of this study was to evaluate the influence of selected semichemical cooking variables on the strength and chemical properties of the received pulp. To assess the level of delignification, confocal microscopy was applied.

MATERIAL AND PULPING METHODOLOGY

Cooking was preceded by sawdust preimpragnation with cooking liquor vapors. Preimpragnation was conducted in Hagglund's autoclaves. Sawdust was placed on a metal net over the cooking liquor mirror. Preimpregnation conditions were as follows: autoclaves heating up to the temperature of 140ºC. Sawdust (fraction collected over the screens >O7 and >O3 (according to SCAN–CM–40:94)) of birch wood (Betula verucosa L.) were digested. The digesting process was carried on in a glycerin bath in Hagglund's autoclaves. Variable cooking time (15, 30, 90, 120 minutes; hydromodule 5) and hydromodule (2, 3, 4, 5; cooking time – 90 minutes) were tested. The targeted cooking temperature was 178ºC. In all trials the industrial cooking liquor with characteristics of Na_2SO_3 – 165g/dm^3 and Na_2CO_3 - 50g/dm^3 was used.

STUDIED PARAMETERS

Pulp samples were collected after digesting at different parameters. For each of the tested parameters the chemical components such as lignin (PN-74/P-50092), holocellulose (PN-74/P50092) and α-cellulose (PN-62/P-50099) were determined. All the pulp samples were refined in PFI laboratory mill to gain 30ºSR (Shopper-Riegler) pulp freeness, then paper sheets were obtained (Rapid_koeten) for the strength test. Four strength properties were examined: SCT (EN/ISO 9895), CMT (EN/ISO 7263), Tear strength (EN 21974) and Burst strength (EN/ISO 2758).

CONFOCAL MICROSCOPY

From each pulp sample thin section was prepared for confocal microscopy measurements. Fibers were washed in distilled water for 1 minute and then were placed in a water-soluble, not fluorescing, Aqua/Polymount mounting medium. The natural fluorescence effect of lignin was applied to control the lignin content in the examined fibers. As reference of lignin content, the cross-section of birch wood stained with 0.1% acriflavin (aqua solution) was used. Thin wood section was soaked in a stain solution for 3 minutes and then washed in distilled water. All samples were analyzed with the use of C1 (Nikon) Confocal microscope after laser excitation with wave length 488 nm (filter before detector 515/30) for green color channel (Ch2), and wave length 543 nm (filter before detector 650 LP) for red color channel (Ch3). The brightness of the taken pictures was analyzed with the use of Region of Interests (ROI) methodology available in EZ-C1 (Nikon) software.

RESULTS

Preliminary studies on the effect of the digesting hydromodule on the change of the chemical composition of the NSSC pulp was carried out under industrial conditions (Joachimiak et al. 2011). Due to a small variation of hydromodules (1.4 - 2.0), which could be controlled, there was no relationship found between the changes in the strength properties and the chemical composition of the NSSC pulp. Slightly more information was provided as a result of the research on the effects of temperature and hydromodulus on the strength properties of NSSC pulp carried out under the same conditions (Bocianowski et al. 2012 Bocianowski et al. 2013). With the use of statistical methods there was the possibility demonstrated of obtaining NSSC pulp with satisfactory strength properties during relatively short cooking time (13 min) and low hydromodulus (1.5). In this paper the results of neutral sulfite liquor digesting in laboratory conditions was analyzed. The results were far different from the industrial NSSC process conditions. In the stationary Hagglund autoclaves it is not possible to repeat the typical conditions of industrial NSSC cooking in the steam-liquid phase. However, it is possible to make an approximate assessment of the impact of various technological factors (temperature, time, hydromodule) on the delignification process.

The results presented in the Table 1 indicate a clear trend of the lowering of the strength properties of neutral sulfite pulp (CMT, SCT and Burst) with cooking duration over 15 minutes. Different trends were observed for the Tear factor - increase in value together with the duration of cooking. It is important that the highest strength properties (CMT, SCT, Burst) were obtained for the pulp least lignified, with the lignin content similar to that of the industrial NSSC pulp. In case of the tests in which the sawdust was preimpregnated before cooking and in which variable hydromodules were used, no explicit dependency was obtained, but the lack of significant differences in strength properties of CMT and SCT for extreme hydromodules 4 and 2 is worth noticing. To some extent, these results indicate similar trends as the above mentioned study in industrial conditions – which means the possibility to receive a pulp with good strength properties at relatively low hydromodules (Bocianowski et al. 2012). Retaking of the experiments on the hydromodule effect on the strength properties, but without the preimpregnation before sawdust cooking, showed a clear trend of the CMT and SCT decreasing together with the reduction of the hydromodule in the range from 5 to 3. However, the conditions of this experiment differ significantly from the industrial practice (no preimpregnation, long digesting - 90 minutes), which does not allow to draw clear conclusions - these strength properties have dropped despite increasing lignin content in the fibrous pulp.

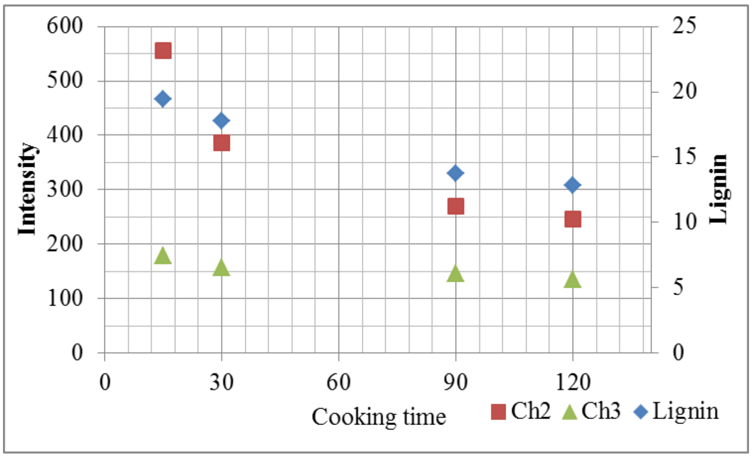
Table 1 Content of strength properties (CMT, SCT, Burts, Tear) and chemical composition (lignin, holocellulose, alfacellulose) of pulp samples in various cooking conditions.

Conditions	Strength properties				Chemical composition		
	CMT [N]	SCT [kN/m]	Burst [kPa]	Tear [mN]	Lignin [%]	Holocellulose [%]	Alfacellulose [%]
Cooking time influence							
15min	197,4	4,061	334,34	693	19,4	78,75	80,01
30min	178,9	3,716	267,57	659	17,8	79,83	81,85
90min	187	3,994	321,4	708	13,7	84,94	83,98
120min	167	3,714	255,73	741	12,8	85,44	84,01
Influence of hydromodule with preimpregnation							
1:4	157	3,376	218,49	606	16,5	82,50	82,80
1:3	178	3,653	288,87	786,5	13,4	84,82	83,36
1:2	154	3,262	198,82	502,5	20,5	78,22	81,52
Influence of hydromodule without preimpregnation							
1:5	177,5	3,726	293,38	730,5	14,9	83,20	83,57
1:4	168	3,513	240,92	628	16,4	81,42	82,81
1:3	162	3,455	240,09	667	18,6	80,77	82,32

The results of the chemical composition determination of the neutral sulfite pulp (Table 1) indicate the possibility of receiving a pulp of similar composition to the industrial NSSC pulp in the tested experimental conditions (digestion in Hagglund autoclaves). This observation refers to the pulps obtained after cooking with shorter time of 15 - 30 minutes and lower hydromodule (2 - 3). Despite the differences in laboratory and industrial digesting technique (Hagglund autoclaves vs. Bauer digester), laboratory cooking results can be, to some extent, related to industrial conditions results. This applies particularly to the delignification process.

Confocal microscopy can be a useful tool for the delignification analysis [Joachimiak et al. 2012]. While analyzing the laboratory pulps with confocal microscopy, the highest values of Ch2 (555), and Ch3 (179) were obtained for the pulp with the highest lignin content, i.e. 19.4% (Table 1) (Figure 1). The results of the microscopic examination showed a high correlation with the results of the gravimetric determinations in the research on the influence of cooking time on the delignification process. In the case of the research on the influence of the hydromodule on the effects of digestion, these results showed a correlation with the preimpregnation digestions (not shown). In the case of the pulp obtained without preimpregnation, some discrepancies were observed between the microscopic results and the gravimetric analyses (not shown), which suggests uneven delignification of the pulps (differences in the content of lignin on the surface of samples).

A



B

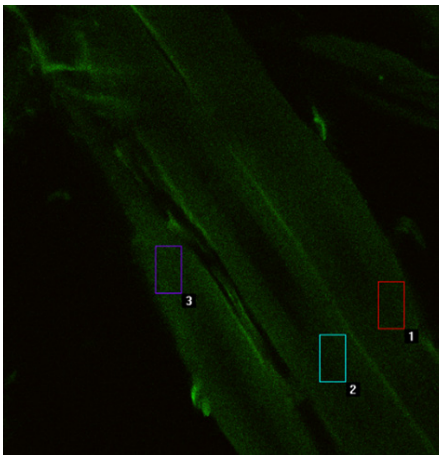


Figure 1 (A) Dependency of lignin content [%] determined with gravimetric method vs. intensity of lignin determined with use of confocal microscope. (B) Regions of Interest in lignin determination of pulp sample obtained in conditions: 15 minutes cooking and 1:5 hydromodule.

CONCLUSION

Cooking time longer than 15 minutes together with hydromodule 1:5 influenced the neutral sulfite pulp decreasing their SCT, CMT and Burst. Preimpregnation of sawdust during cooking in Hagglund's autoclaves simulates industrial conditions and should be applied during further trials. The confocal microscopy results indicate that preimpregnation of sawdust may have the equalization effect on the lignin distribution through the pulp fibers.

REFERENCES

1. JOACHIMIAK K., BOCIANOWSKI J., WÓJCIAK A., 2011: The effect of liquor to wood ratio on NSSC pulp chemical composition and fibers distribution characteristic, Annals of Warsaw University of Life Sciences – SGGW Forestry and Wood Technology 74, 161-165
2. BOCIANOWSKI J., JOACHIMIAK K., WÓJCIAK A., 2012: The influence of process variables on the strength properties of NSSC birch pulp. Towards the limits of optimization: part one – the effect of liquor ratio, Drewno. Prace Naukowe, Doniesienia, Komunikaty vol. 55 no 188, 17 - 32
3. BOCIANOWSKI J., JOACHIMIAK K., WÓJCIAK A., 2013: The influence of process variables on the strength properties of NSSC birch pulp. Towards the limits of optimization: part two - the effect of temperature and time of cooking, Drewno. Prace Naukowe, Doniesienia, Komunikaty vol. 56, no 189, 71 - 87
4. JOACHIMIAK K., BOCIANOWSKI J., WÓJCIAK A., 2012: The effect of process variables on the NSSC pulp chemical composition - the use of confocal microscopy, Annals of Warsaw University of Life Sciences – SGGW Forestry and Wood Technology 78, 130-134

Streszczenie: *Właściwości fizyko-chemiczne mas obojętnosiarczynowych z trocin brzozowych.* Celem pracy było określenie różnic w składzie chemicznym oraz parametrów wytrzymałościowych mas włóknistych otrzymanych w wyniku roztwarzania trocin brzozowych w różnych warunkach. Mikroskopia konfokalna została zastosowana jako metoda określania zmienności procesu delignifikacji. Uzyskane wyniki z badań z użyciem mikroskopu konfokalnego wskazują, iż próbki trocin bez użycia impregnacji wstępnej mogą wykazywać nierównomierną dystrybucję ligniny pozostałej po procesie roztwarzania.

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