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COMPARISON OF *MISCANTHUS GIGANTEUS* AND BIRCH WOOD NSSC PULPING PART I: THE EFFECTS OF TECHNOLOGICAL CONDITIONS ON CERTAIN PULP PROPERTIES

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ABSTRACT

Chemical composition and susceptibility to delignification by neutral sulfate liquor of *Miscanthus giganteus* stems and of birch wood were compared. The yield of pulping as well as degree and selectivity of delignification were tested in various technological conditions (cooking time, hydro module, alkali charge). Having a similar chemical composition, *Miscanthus giganteus* stems are subjected to quicker and deeper delignification with neutral sulfite liquor than birch wood. This phenomenon is probably associated with differences in the qualitative composition of lignin, distribution of lignin in the cell walls and in the morphological features of both raw materials.

KEYWORDS: *Miscanthus giganteus*, birch wood, NSSC pulping.

INTRODUCTION

Paper packaging producers manufacture cardboard mainly from paper obtained from recycled fibers. Increasing sanitary requirements, particularly in the case of paper used in contact with food, result in constant interest in maintaining the production of fluting and liners from virgin fibers. Traditionally, fluting is obtained from hardwood, mainly using the neutral sulfite semi-chemical process (NSSC). Satisfactory strength properties and high yield have resulted in continued use of NSSC methods and in their importance among the "traditional" pulping processes. Simultaneously, limited resources of hardwood have induced a search for new raw materials that would be useful in the production of semi-chemical pulps.

According to the literature, 10% of world paper production is made from non-wood raw materials (Bullard 2001). In Europe, however, this is less than 1% of pulp from grass raw materials (Visser et al. 2001). Fast-growing plants whose properties and morphological structure are close to wood constitute a perfect raw material reservoir that can be used as an addition to current processes of pulp production, without significant quality deviation of the pulps or ready-made paper products (Pahkala et al. 1997, Pažitný et al. 2013). Their use may affect the rationalization of forest resources management (Fowler et al. 2003). In addition, increasing the cultivation of grassy plants also constitutes an opportunity for the reduction of CO and CO2 emissions that arise from the burning of agricultural waste. The use of annual plants to obtain NSSC pulps is known from studies on the use of hemp and its waste (Danielewicz 2013). Different varieties of *Miscanthus* are very promising among the fast-growing plants when the economical aspect of pulp production is considered. So far, relatively few research papers have been dedicated to obtaining pulp from *Miscanthus*, including mainly sulfate, sodium and high-yield TMP or CTMP processes (Cappelletto et al. 2000, Danielewicz et al. 2015, García et al. 2014, Iglesias et al. 1996, Marin et al. 2009, Thykesson et al. 1998). The production of neutral sulfite pulps has been the subject of an even smaller number of studies (Ahmadi et al. 2010, Kordsachia et al. 1993).

This paper presents the results of comparative studies on the production of neutral sulfite pulps from *Miscanthus* stems in reference to a typical European raw material, namely birch wood that is used in the production of such pulp type.

MATERIALS AND METHODS

Raw materials

During the research, *Miscanthus giganteus* straw hybrids GM-4 (of stalks) with a fraction of 0.1 – 1.0 - 3.5 cm (thickness-width-length) were applied. Industrial birch wood (*Betula verrucosa* L.) sawdust (a fraction collected over screens > ϕ7 and > ϕ3 according to SCAN–CM–40:01) was used for all of the digestions.

Digestion

The digestion process (with the exception of the preheating period) was carried out under isocratic conditions in Hägglund's laboratory autoclaves, immersed in a glycerin bath. The experiments were performed with initial impregnation of sawdust by vapors of neutral sulfite liquor. The impregnation time (the resulting temperature was 140°C) was ca. 15 min; the cooking time was 15, 30, 90 and 120 min. The comparative conditions of the digestions were controlled by the H factor. The maximum temperature during cooking was ca. 177°C. Two industrial cooking liquors were used for all of the digestions. For the impregnation and cooking stage, neutral sulfite liquors with two various chemical charges (Na_2SO_3 97.97 g·dm^{-3} and 165.06 g·dm^{-3}; Na_2CO_3 66.04 g·dm^{-3} and 72.08 g·dm^{-3}, pH 12.08 - 12.94, respectively) were applied. For the proper cooking stage representing simulation of real industrial conditions, "red" liquor (spent liquor obtained after NSSC pulping) with a density of 1.035 g·dm^{-3} was added to the portion of liquor introduced before the impregnation stage. For all of the neutral sulfite cooking, hydro modules (l/w) from 2 to 5 were used. The alkaline Na_2SO_3-to-wood (o.d.) ratio ranged from similar to industrial conditions: 7.4% on o.d. wood to excessive charges from 19 up to 31 and 41% on o.d. wood, thus making possible appropriate refining of pulp and forming of paper sheet for further strength tests (published in another paper). For each experiment, two to four independent cooking processes were carried out. After digestion, the pulps were washed with running water until neutral pH was obtained. The data in Tab. 1 show that using preliminary impregnation in conditions of a 7.4% alkali charge on o.d. wood on a laboratory scale leads to similar cooking liquor consumption in comparison to industrial conditions.

Tab. 1: Comparison of spent liquor indices obtained during laboratory and industrial digestion of Miscanthus giganteus stems and birch sawdust using the same alkali charge (cooking time – 15 minutes).

Digestion	Na_2SO_3 (%) on o.d. wood	Raw material	pH of spent liquor	Residual alkali	Dry mass content (%)
Laboratory conditions	7.4	*Miscanthus*	7.97 - 8.87	14.5 - 18.3	17.7
		Birch	6.73	10.8	14.3
Industrial cooking		Birch	6.8 - 7.56	16.4 - 22.0	14.5

After washing, the pulps were stored and conditioned in room temperature for one week. After that, the moisture content and yield were determined.

Test methods

Before and after cooking, the following chemical compositions were determined in the *Miscanthus* stalks and in birch sawdust: Klason lignin (PN-74 / P50092), Seifert's cellulose (PN-62 / P-50099), holocellulose (PN-74 / P50092), pentosans (Tollens' method), extractives with hot water (TAPPI T-207 om-88), extractives with cold water (TAPPI T-207 om-88), extractives with benzene-methanol (TAPPI T-204 cm-97), extractives with 1% NaOH (TAPPI T-212 om-02) and 10% NaOH (Prosiński 1984), and ash content (TAPPI T-211 om-07).

In pulps obtained after cooking, the yield, the degree ($\Delta L/L$) and selectivity of delignification (L/Hc - ratio of lignin to holocellulose; $\Delta L/\Delta Y$ - ratio of the degree of delignification to yield loss) were determined.

In the spent liquors, pH (PN-EN ISO 5267-1 2002), residual alkali as Na_2SO_3 by titration with 0.1 M $Na_2S_2O_3$ and dry solids (drying to constant mass) were determined. Extraction of *Miscanthus* stalks with a 1% NaOH solution was carried out according to test method (TAPPI T-212 om-02). After extraction, 5 ml samples of filtrates were collected and diluted with distilled water at a ratio of 1:50 in 250 ml glass flasks. UV-spectra were obtained on a Varian Cary UV-Vis spectrophotometer.

Elaboration of results and statistical analysis

The pulping experiments for each of the tested parameters were repeated twice. The results of determining the chemical composition of the pulps are the arithmetic average of three or four determinations performed in parallel. Standard deviations were calculated. It was assumed that the results are differing over 1% for yield and 0.4% for chemical composition.

RESULTS

Miscanthus giganteus stems were selected for digestion as they constitute the main part of the plant's mass (ca. 76% of the raw material) and ensure homogeneity of the plant's fibrous fraction in pulps (leaf fibers differ morphologically from the stem) (Cappelletto et al. 2000). Although the tested varieties of both *Miscanthus* and birch did not differ in terms of content of the main structural components (lignin, cellulose), some quantitative differences exist in the case of holocellulose, pentosans, extractives and minerals (Tab. 2).

Tab. 2: Chemical composition of Miscanthus giganteus stems and birch wood.

Content (%)	*Miscanthus*	Birch	Content (%)	*Miscanthus*	Birch
Lignin	21.6	21.1	10% NaOH	50.9	35.6
Cellulose	44.6	43.7	Cold H_2O	4.4	0.7
Holocellulose	72.1	68.7	Hot H_2O	6.0	1.6
Pentosans	29.1	23.5	Extractives	4.5	2.4
1% NaOH	35.3	15.8	Ash	1.6	0.4

The relatively high cellulose (44.6%) and relatively low lignin content (21.6%) in *Miscanthus* is noteworthy. According to Nieschlag et al. (1960), fibrous plants containing more than 34% of α-cellulose are suitable for papermaking. A high content of holocellulose (72.1%) also predestines this raw material for the production of semi-chemical pulps (Marin et al. 2009). Chemical composition of analyzes presented here are more or less similar to those in the literature data (Cappelletto et al. 2000, Iglesias et al. 1996, Papatheofanous et al. 1995, Scurlock 1999, Vega et al. 1997, Ververis et al. 2004, Ye et al. 2005). Ash content, although higher than for birch, is relatively low for grassy plants (Finell 2003). Similar data have been reported in the literature (Cappelletto et al. 2000, Iglesias 1996, Ververis et al. 2004). The relatively low content of mineral substances should not be an obstacle in the neutral sulfite pulping process, as it does not use the recovery of spent liquors in the same range as the sulfate method.

The varied qualitative composition of lignin and hemicellulose components of holocellulose in both of the studied raw materials (birch and *Miscanthus*) probably had an influence on the significant differences in the content of soluble substances in 1% and 10% NaOH. The high content of the latter substances in *Miscanthus* stems (35.3%) determined the behavior of this raw material during cooking (Iglesias et al. 1996). The spectra of the alkaline extract (1% NaOH), particularly high absorption at ca. 280 nm, confirmed the higher solubility of lignin substances in *Miscanthus* than in birch. The point of inflection observed at ca. 320 nm may be related to the presence of conjugated phenol unit in ferulic acid, p-coumaric acid and hydroxycinnamic lignin structures (Oliveira et al. 2009, Pan and Sano 1999, Zhao and Liu 2010). These compounds are formed as the result of saponification of the ester bonds of lignins and xylans of grass plants (Danielewicz et al. 2015). The spectrum of the birch wood extract showed no absorption in the above-mentioned wavelength ranges. Although the differences in solubility in alkali suggest the possibility of using milder cooking conditions for *Miscanthus* (alkali charge, temperature), comparable lignin content between both raw materials prompted us to commence experiments under the same cooking conditions for *Miscanthus* tems and for birch wood (Fig. 1).

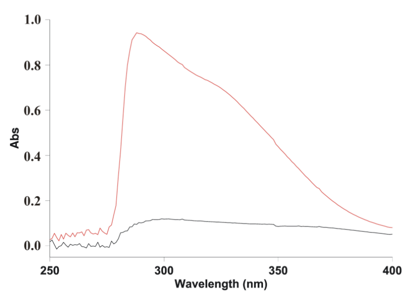

Fig. 1: UV spectra of filtrates of 1% NaOH solution treatment for birch wood (black) and Miscanthus giganteus stems (red).

Cooking in an alkaline environment, although with a lower pH than, for example, the sulfate method (Joachimiak et al. 2016), affected a lower yield of the *Miscanthus* pulps as compared to pulps obtained from birch wood (Fig. 2). The excessive dose of alkali used in the experiment (in comparison to industrial conditions, pH 9.8 of the spent liquor is ca. 1.5 units higher) allowed to observe differences in the reactivity of both *Miscanthus* stems and birch wood.

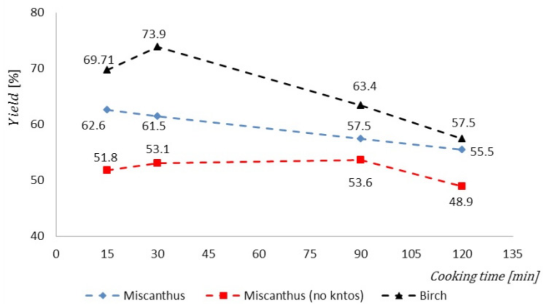

Fig. 2: Influence of cooking time on NSSC pulp yield obtained from Miscanthus giganteus stems and birch wood (31% Na_2SO_3 on o.d. wood; both birch and Miscanthus giganteus with impregnation stage).

The significant loss of mass from the *Miscanthus* stems took place already after 15 minutes of cooking, which corresponds to the industrial conditions for the production of NSSC pulps in Bauer digesters. These types of pulps, after blowing from the digester, occur in the form of softened chips and require defibration. As the processing time increased, differences in the yields of birch and *Miscanthus* pulps were reduced. Fig. 2 also shows the yield of *Miscanthus* pulps but without knots – in this case the differences in yield (despite large variation in the cooking times) are small, which suggests that the loss of mass resulted mainly from processes of delignification and dissolution of hemicelluloses and extractives.

The differentiated reactions of the chemical components of both *Miscanthus* and birch to neutral sulfite liquor are presented in Fig. 3.

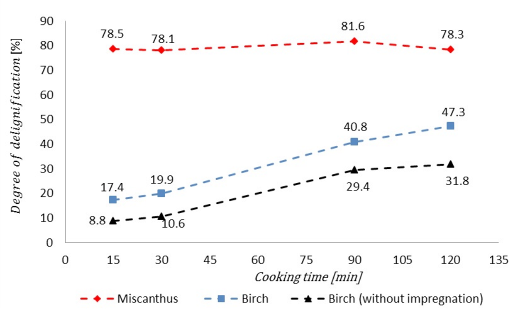

Fig. 3: Degree of delignification of birch wood and Miscanthus giganteus stems (31% Na_2SO_3 on o.d. wood; birch with and without the impregnation stage).

The degree of delignification of birch wood increases along with a longer cooking time, with a significant increase in delignification after 30 minutes of cooking. Faster delignification took place for wood after preliminary impregnation than for wood processed without this stage.

The *Miscanthus* stems were rapidly and deeply delignified in the first 15 minutes of digesting. Prolonging the process did not improve the effectiveness of residual lignin removal.

Contrary to birch wood, prolonging the cooking time of *Miscanthus* stems did not affect changes in the level of delignification or in the selectivity of this process that is presented as a change in the proportion of lignin to holocellulose (Fig. 4).

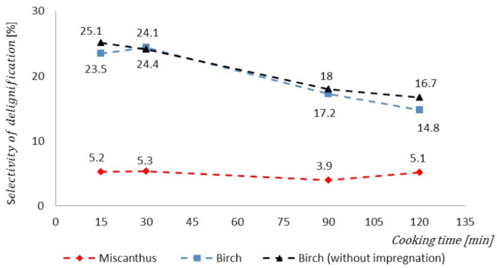

Fig. 4: Selectivity of delignification (L /Hc) of birch wood and Miscanthus giganteus stems (31% Na_2SO_3 on o.d. wood; birch with (blue) and without the impregnation stage (black), Miscanthus with impregnation (red)).

The selectivity of delignification of birch wood deteriorated along with an increase in the cooking time. In the cooking of birch, there were no differences in selectivity between digestion with or without impregnation.

The graphs of changes in indices of selectivity show difference in terms of changes in lignin content and pulp yield (Fig. 5). Selectivity of *Miscanthus* delignification worsens along with the cooking time. The losses of lignin in relation to the loss of yield are reduced.

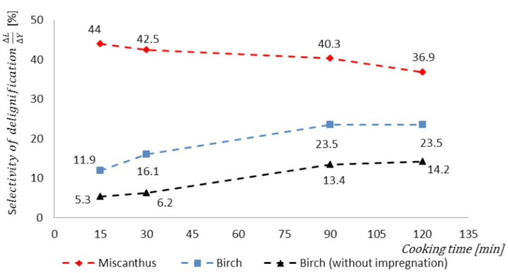

Fig. 5: Selectivity of delignification (ΔL/ ΔY) of birch wood and Miscanthus giganteus stems (31% Na_2SO_3 on o.d. pulp, birch with (blue) and without the impregnation stage (black), (red) – Miscanthus with impregnation).

The indices for birch wood are to some extent correlated with the graphs of the yield changes (Fig. 2) and the level of delignification (Fig. 3). The lignin losses increase with respect to the yield losses.

An analysis of the data presented in Figs. 1 - 4 indicates that *Miscanthus* is a raw material that quickly undergoes fairly deep delignification, and yield losses during the digestion time are associated not only with a loss of the carbohydrate components of holocellulose but also of other alkaline soluble compounds. The results of the experiments confirmed the data indicating high susceptibility of grass plant lignin to delignification in an alkaline environment, which was associated with the presence of the so-called hemilignin, a compound with a lower degree of polymerization than wood lignin (Buranov and Mazza 2008, Danielewicz et al. 2015). The differences in the morphology of the anatomical elements and the distribution of lignin also had an impact on the diversity of grassy and woody plant delignification.

The above results pertain to cooking periods carried out with a large excess of alkali (31% to o.d. mass) in order to more easily conduct pulp sample refining and then to determine the strength properties (part II – published in a separate paper). The test results for lower dosages of Na_2SO_3 are more reliable, including conditions similar to those that are currently used in the industrial practice of neutral sulfite cooking of hardwood (initial impregnation, 7.4% Na_2SO_3 to o.d. mass, 15 min cooking time). In the laboratory conditions used in this study, it was possible to achieve a yield of birch neutral sulfite pulp similar to that obtained in industrial practice (84.1%). The yield of *Miscanthus* stems was lower, although the differences between this raw material and birch were reduced along with increasing alkali doses for a shorter cooking time that was similar to industrial practice (15 min) (Fig. 6).

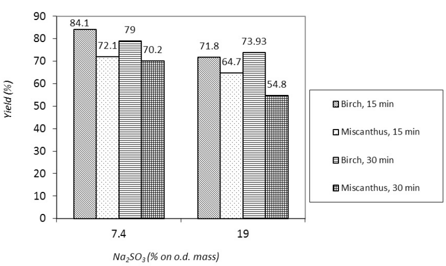

Fig. 6: Influence of Na_2SO_3 dose on birch and Miscanthus giganteus pulp yield (initial impregnation, 15 min cooking time (H 187) and 30 min cooking time (H 278)).

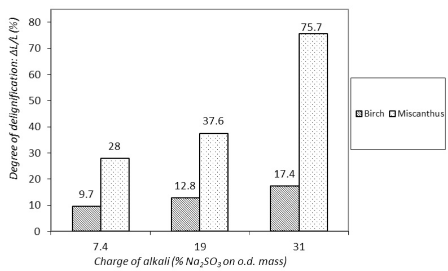

Fig. 7: Influence of Na_2SO_3 dose on the delignification level (ΔL/L) of birch and Miscanthus giganteus pulps (initial impregnation, 15 min cooking time (H 187))

The effect of the alkali dose on the level of delignification is shown in Fig. 7. A clear tendency is visible that there is an increase in the difference between the degree of delignification of birch and *Miscanthus* as the alkali concentration increases. The degree of delignification in *Miscanthus* stems is already high and reaches 28% for a low dose of 7.4% Na_2SO_3 as used in industrial practice.

The comparison of delignification selectivity indicators for both *Miscanthus* and birch calculated in relation to carbohydrate losses (L/Hc) is more favorable for birch wood. The reason for the reduction in selectivity indices for *Miscanthus*, regardless of the alkali dose used is a significant loss of yield associated with the dissolution of carbohydrate components.

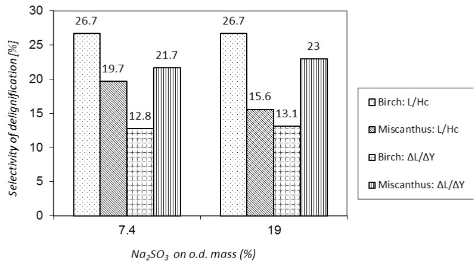

Fig. 8: Influence of Na_2SO_3 dose on delignification selectivity of birch and Miscanthus giganteus pulps (initial impregnation, 15 min cooking time (H 187)).

The fact that productivity losses in the case of *Miscanthus* are mainly related to carbohydrates and extraction substances and not to lignin is confirmed by the results of the determinations of lignin losses with respect to yield losses (ΔL/ΔY). Losses of lignin in relation to losses of pulp yield from *Miscanthus* stems were relatively smaller than for birch wood. This dependence was repeated irrespective of the level of alkali doses during cooking (Fig. 8).

CONCLUSIONS

1. Despite a similar chemical composition, *Miscanthus giganteus* stems are subjected to quicker and deeper delignification with neutral sulfite liquor than birch wood. This phenomenon is most probably associated with differences in the qualitative composition of lignin, distribution of lignin in cell walls and the morphological features of both raw materials.
2. Delignification selectivity of *Miscanthus giganteus* stems is worse than that of birch wood, which is associated with significantly higher losses of carbohydrate components and extractive compounds during digestion.
3. *Miscanthus giganteus* stems are not a competitive material in relation to birch wood in traditional variants of neutral sulfite pulping methods. The high content of holocellulose and the relatively low ash content indicate that *Miscanthus giganteus* stems may be a complementary material to hardwood.

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