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NSSC PULPING OF *MISCANTHUS GIGANTEUS* AND BIRCH WOOD PART 2: A COMPARISON OF PAPERMAKING POTENTIAL AND STRENGTH PROPERTIES

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ABSTRACT

In this paper we compare the anatomical features (number of fibrous elements in the pulp unit, fiber length, diameter of fibers and lumens, coefficients of slenderness, flexibility and the Runkel coefficient) of both *Miscanthus giganteus* and birch wood. The raw materials were tested prior to pulping, after pulping and after refining. Comparisons of defibering ability and selected strength properties (CMT, SCT, tear resistance, burst) allowed evaluating the papermaking potential of neutral sulfite pulps obtained from the above-mentioned raw materials.

KEYWORDS: *Miscanthus giganteus*, papermaking indices, NSSC pulping.

INTRODUCTION

Research on the preparation of pulp from *Miscanthus giganteus* as published so far has mainly concerned sulfate, sodium and high yield pulps such as TMP (thermomechanical pulp) and CTMP (chemi-thermomechanical pulp) (Iglesias et al. 1996, Thykesson et al. 1998, Cappelletto et al. 2000, Marin et al. 2009, García et al. 2014, Danielewicz et al. 2015). The production of neutral sulfite pulps has been the subject of an even smaller number of studies (Kordsachia et al. 1993, Ahmadi et al. 2010). The pulps mentioned above differ in their method of preparation, chemical composition and strength properties, which determines their use for the production of various types of paper and various other purposes. Diversification of pulp use results in the need for analyses of their mechanical strength, and various indices are assessed, e.g. in printing and typewriting paper, strength properties such as breaking strength, tear resistance and bulk structural properties are important. In fluting grades, obtained from neutral sulfite pulp and designed for corrugated medium, stiffness measured with SCTs or CMT is more important. The values of all of the strength and mechanical factors depend on many factors, such as fiber morphology, the beating degree and the method of pulp production, which determines their chemical composition. The dynamic strength properties (tear index, burst) are influenced by the number and surface of fiber binding points, which mainly depend on the level of external fibrillation. Stiffness is also influenced by chemical composition, e.g. lignin content and morphological characteristics (fiber dimensions, curl).

The aim of this paper is to compare the strength properties of neutral sulfite pulps obtained from Miscanthus stems with reference to a typical European raw material, namely birch wood that is used to produce this type of fibrous material.

MATERIAL 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. Birch wood (*Betula verrucosa* L.) sawdust obtained from industrial chips (a fraction collected over screens > ϕ7 and > ϕ3 according to SCAN–CM–40: 94) 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 as a two-stage process (impregnation and digestion), with preliminary impregnation of sawdust by vapors of neutral sulfite liquor. The impregnation time (the resulting temperature was 140 °C) was ca. 15 min. The comparative conditions of digestions were controlled by the H factor (cooking time ca. 15 and 30 min.). The maximum temperature during cooking was ca. 177 °C. Two industrial cooking liquors were used for all of the digestions, as they allowed differentiating the charges of alkali on o.d. wood. Neutral sulfite liquors with two various chemical charges, namely 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 and 12.94, respectively, were used for impregnation and the cooking stage. For the proper cooking stage representing simulation of real industrial conditions, the "red" liquor (spent liquor obtained after NSSC pulping) with a density of 1.035 g·cm^{-3} was added to the portion of liquor introduced before the impregnation stage. For all of the neutral sulfite cooking, hydro modules (the liquor-to-wood ratio was l/w) from 2 to 5 were used. Alkaline Na_2SO_3-to-wood (o.d.) ratio ranged from one that was similar to industrial conditions, 7.4 % on o.d. wood, to excessive charges of 49% on o.d. wood, thus making possible the appropriate refining of pulp and paper sheet forming for the strength tests. For each experiment, two to four independent cooking procedures were carried out. After digestion, the pulps were washed with running water until neutral pH was obtained.

Test methods

The morphological characteristics were measured on the macerated material. The maceration process was carried out using a 30% mixture of acetic acid and hydrogen peroxide at 60°C for 20 hours. Microscopic preparations were made from the obtained macerates on which the length of the fibers, fiber diameters and lumen diameters were measured. A total amount of 50 fibers was measured from each raw material; the diameters were determined in the widest parts of the tested fibers. Measurements of morphological characteristics were carried out using the Primo Star Light Microscope (Carl Zeiss Microscopy, Germany) in combination with a computer image analyzer using the Motic Images Plus 4.0 program. Based on the measurements made, fiber wall thickness, slenderness ratio (as the fiber length/fiber diameter), flexibility coefficient [(as the fiber lumen diameter/fiber diameter) × 100] and the Runkel coefficient [(2 × fiber cell wall thickness)/lumen diameter] were calculated (Saikia et al. 1997, Ogbonnaya et al. 1997).

Measurement of the number of fibers per pulp unit of raw material was made on two equal samples (0.001 g) which were cut along the fibers of the birch wood and *Miscanthus* stems. The samples were macerated before weighing according to the procedure as outlined above. The calculations were made by analyzing the image on the measurement stand as described above.

An analysis of the fractional composition of the birch and *Miscanthus* pulps was made by computer image analysis in the PulpExpert PEFBA 14 apparatus. The prepared sample, which was in the form of a fibrous suspension, was introduced into the capillary and then into the device. The analyzer created several photos which were then analyzed. The apparatus grouped the fibers into classes with narrow fiber length ranges and the results were presented in graphic form. Each fiber class was expressed as a percentage of all the marked fibers.

For each of the studied technological parameter, the obtained pulp samples were refined in a PFI laboratory mill to reach the appropriate Schopper-Riegler degrees. Hand sheets were made after Schopper-Riegler freeness tests (PN-EN ISO 5267-1, 2002) had been conducted for all of the pulp samples.

The defibrability index was calculated by dividing the number of rotations during the refining time by the degree of freeness. Four strength properties were examined in the dried and conditioned paper sample, namely the SCT – Short Crush Test (EN/ISO 9895), CMT – Concora Medium Test (EN/ISO 7263), Tear strength (EN 21974) and Burst strength (EN/ISO 2758).

Statistical analysis

The normality of distribution of the studied traits was tested using Shapiro-Wilk's normality test. One-way analysis of variance (ANOVA) was carried out to determine the effects of both *Miscanthus giganteus* and birch wood on CMT, SCT, Tear and Burst strength development. Mean values, least significant differences (LSDs) and p-values were calculated, which allowed to create homogeneous groups for the observed traits. Data analysis was performed using the GenStat 18.2 edition.

RESULTS AND DISCUSSION

The quantitative measurements of fibrous elements in a unit of pulp of birch wood and *Miscanthus* stems, taken in the raw material before digestion, indicate a higher share of fibers in the birch wood (Tab. 1).

Tab. 1: Number of anatomical fibrous elements in 0.001 g of raw material (before digestion).

Miscanthus stems	Birch wood
4100	8447

Such a comparison of results suggests the birch fibers' greater papermaking potential than that of the *Miscanthus* stems. It should be noted that the *Miscanthus* stems contained a large number of non-fibrous elements, parenchyma and other cells (primary fine fraction), which were not taken into account in the above analysis (Fig. 1).

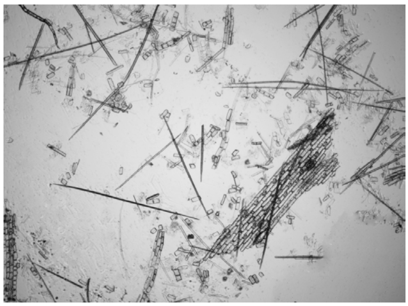

Fig. 1: Non-fibrous elements, parenchyma and other cells – primary fine fraction in Miscanthus pulp.

The high content of the non-fibrous fine fraction is also unfavorable from the point of view of the web formation process, as it limits dewatering capacity without compensation for the paper's structural features (volume weight, density of the paper structure).

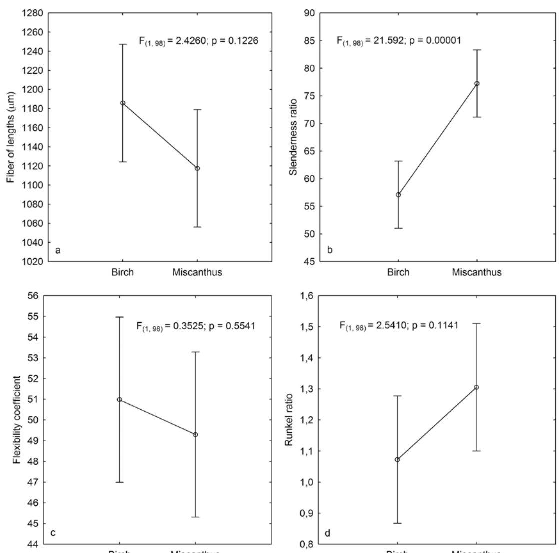

Fig. 2: Comparison of fiber length, slenderness, flexibility and Runkel coefficient for birch wood and Miscanthus stems (before digestion). The vertical bars stand for 0.95 confidence intervals.

The microscopic analysis of morphological features of the fibrous elements started with a comparison of the raw materials before digestion. The fiber length analysis indicated no significant differences between the tested raw materials (Fig. 2), but the fiber and lumen diameters of the *Miscanthus* fibers were smaller than those of the birch fibers, which also meant that the thickness of the cell walls was reduced.

The thinner cell walls of the *Miscanthus* fibrous anatomical elements significantly accelerated delignification reactions during cooking. The average length of the *Miscanthus* fibers (1117.5 µm), fiber diameter (16.2 µm) and lumen (8.64 µm) were similar to the literature data (Ververis et al. 2004, Danielewicz et al. 2015). Also, data similar to the results of the work of Ververis et al. (2004) were obtained for measurements of mean values of the slenderness coefficient (77.2), flexibility (49.3) and Runkel coefficient (1.3). Comparative statistical analyses showed that the *Miscanthus* fibers were characterized by both a similar flexibility and Runkel coefficient but a higher slenderness index than the birch fibers. The average value of the Runkel coefficient was higher for *Miscanthus*, but differentiation of cell wall thickness meant that the measured values were characterized by a high coefficient of variation (Fig. 2).

It is known that digestion in an even slightly alkaline environment causes changes in the morphological characteristics of pulp, and the dimensions of the fibers and the share of the fine fraction also change. The fibers in the refining process are subjected to even greater changes, where particularly the proportion of the secondary fine fraction increases. All of these processes ultimately affect the properties of the paper. After digestion, again no significant difference in the length of birch and *Miscanthus* fibers was found, but changes in the cross-sections caused statistically significant differentiation of the slenderness, flexibility and Runkel coefficients. Although the coefficient of slenderness was more favorable for *Miscanthus*, the remaining coefficients (flexibility and Runkel) were worse than for the birch fibers (Fig. 3).

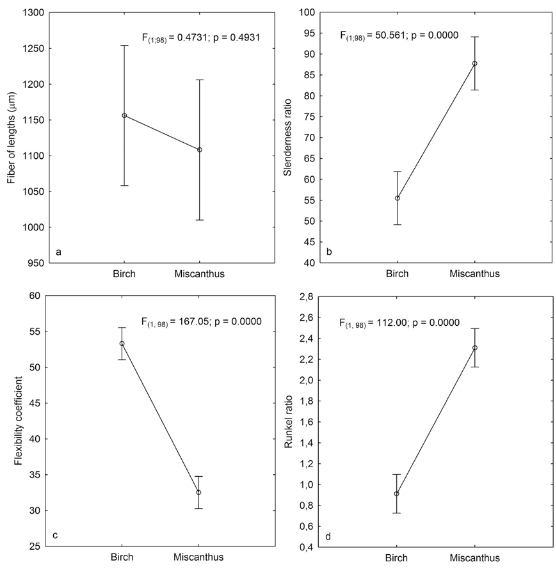

Fig. 3: Comparison of fiber length, slenderness, flexibility and Runkel coefficient for birch wood and Miscanthus stems after digestion (41% Na_2SO_3 on o.d. wood, 15 min, H 187). The vertical bars stand for 0.95 confidence intervals.

The results of the microscopic analysis of fibers taken from the hand sheets formed after refining (not shown) were similar. The slenderness coefficient was still significantly higher than that for the birch fibers. The size of differentiation on the cross-section of both the birch and *Miscanthus* fibers decreased – this was visualized by a decrease in the differences (but still statistically significant) of the flexibility coefficient and a reduction of the Runkel coefficient for the *Miscanthus* fibers, which are quite long and very slender, and slimmer than other grasses (Kamoga et al. 2016). This feature may affect the increase in physical strength of paper, particularly breaking strength (Kamoga et al. 2016). Fiber length also positively influences the tear resistance of paper (Paavilainen 1991, Ververis et al. 2004). This effect takes place particularly when long fibers are accompanied by an appropriate fraction of short fibers (Danielewicz 2013). However, the indices of the flexibility of *Miscanthus* fibers were worse than those for birch and other hardwood (Ververis et al. 2004), which may deteriorate the strength of the paper, particularly breaking strength, but also burst (Ogbonnaya et al. 1997). The smaller fiber diameters of *Miscanthus* and the smaller thickness of cell walls also degrade the value of the bulk index of papers and the stiffness of fibers. The least favorable indicator for *Miscanthus* fibers is the Runkel coefficient. Its high value as compared to birch wood, and particularly the average values for hardwood species (Ververis et al. 2004); indicates poorer felting power and is correlated with the weaker flexibility of *Miscanthus* fibers. A Runkel coefficient above "1" will affect the deterioration of the paper's mechanical properties.

The results of the microscopic analyses supplement the analyses of the fractional composition of the birch and *Miscanthus* pulps studied here. The analyses were made by means of computer image analysis in the PulpExpert apparatus (Tab. 2).

Tab. 2: Analysis of the fractional composition of neutral sulfite pulps from Miscanthus and birch (with and without preliminary impregnation, 41% Na_2SO_3 on o.d. wood, cooking time 15 min, H 187).

Pulp	Digestion	Curl (%)	Fines <0.2 mm	Short fibers 0.2-0.8	Middle fibers 0.8-1.8	Long fibers 1.8-5.0
Miscanthus	Initial impregnation	**10.80**	**16.00**	**47.83**	**25.53**	**10.67**
	Std. dev.	0.173	0.200	0.252	0.252	0.451
	Without impregnation	**11.40**	**15.73**	**47.27**	**25.27**	**11.73**
	Std. dev.	0.173	0.379	0.208	0.451	0.115
Birch	Initial impregnation	**9.00**	**4.10**	**42.60**	**44.60**	**8.73**
	Std. dev.	0.000	0.100	0.200	0.100	0.289
	Without impregnation	**9.00**	**4.17**	**42.77**	**44.83**	**8.73**
	Std. dev.	0.100	0.289	0.462	0.379	0.416

The tests were carried out on pulps obtained after pulping with and without preliminary impregnation. For birch pulp, the pulping process (preliminary impregnation) did not affect the quantitative differentiation of individual fiber fractions. The *Miscanthus* pulps obtained after pulping with impregnation differed slightly (a statistically significant, higher content of both long and short fibers, less curl). They also contained nearly four times more fine fractions than those of birch wood. Such a high content of fine fibers explains, to a certain extent, the differences between the defibrability index and the speed at which the assumed freeness was achieved during the refining of *Miscanthus* and birch pulp (Tab. 3). The high content of the fine fraction, which swells easily in a water environment, will deteriorate the dewatering process during formation on the wire and increase the paper's structural integrity but also limit the dynamic strength properties of the paper. If the content of short fibers (0.2-0.8 mm) is only ca. 5% higher in *Miscanthus* pulps, then the share of medium fibers (0.8-1.8 mm) is nearly two times lower than in birch pulps. The lower share of medium fibers after refining does not compensate for the higher content of long fibers, by ca. 3%, in pulps from *Miscanthus*.

Tab. 3: Defibrability index of neutral sulfite pulps from Miscanthus stems and birch wood; (preliminary impregnation, then cooking, 31% Na_2SO_3 on o.d. wood, time 15 min, H 187).

Defibrability index (quantity of PFI refiner revolutions /°SR)	
Miscanthus stems	Birch wood
32.9	69.4

Such proportions would explain lower resistance to tearing and worse remaining strength indices, including the stiffness of *Miscanthus* fibers in comparison with birch pulp. The poorer strength properties of fibers from *Miscanthus* were also reflected in an increased share of the "curl" ratio in comparison to birch. It should be added that an important element affecting the tear index and burst may also be the low content of hemicellulose passing through to the solution during cooking, which reduces the possibility of bonding between the surfaces of neighboring fibers.

Differences in the digestion chemistry of *Miscanthus* stems and birch wood as well as differences in morphological characteristics (fiber length, fine fraction content) are also reflected by the defibrability index, calculated by dividing the number of laboratory PFI refiner and pulp freeness. Stems of *Miscanthus* that were more easily subjected to delignification than the wood of birch (Joachimiak et al. Part I of the paper), with a low proportion of fibrous elements and large proportion of fine fraction and short fibers, were also subjected to fiberization more quickly than birch wood (Tab. 3). The number of rotations of the refining rotor that were necessary to defibrate pulp from *Miscanthus* stems which did not have significant woody areas in the fibers, such as cannabis (Cierpucha et al. 2004), kenaf (Nieschlag et al. 1960) or grasses other than *Miscanthus giganteus* (Pažitný et al. 2013), was much smaller than that for birch wood.

Strength properties tests were performed for two different experimental variants due to the large differences in the defibrability index. The first option included an analysis of pulps under the same conditions but with different freeness (Fig. 4).

The results of ANOVA indicate that the differences between the studied properties (CMT, SCT, Tear and Burst) of both *Miscanthus* and birch wood pulps were significant for all four traits. It was difficult to achieve a pulp with comparable freeness to birch pulps during refining due to the high content of primary and secondary fine fractions in the *Miscanthus* pulps. More intensive shortening of the fibers had an influence on the reduction of dynamic strength indices (tear resistance, burst) and stiffness (SCT, CMT) of *Miscanthus* fibers.

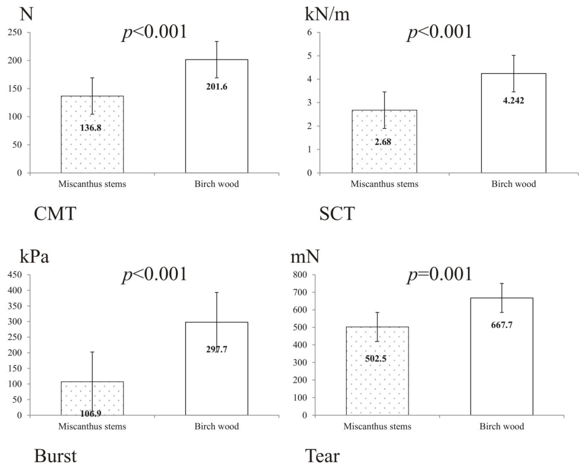

Fig. 4: Comparison of strength properties of neutral sulfite pulps from Miscanthus stems and birch wood. Experimental conditions: impregnation, digestion H 116.6, 7.4% Na_2SO_3 on o.d. mass. Freeness: Miscanthus 40°SR, birch 36°SR.

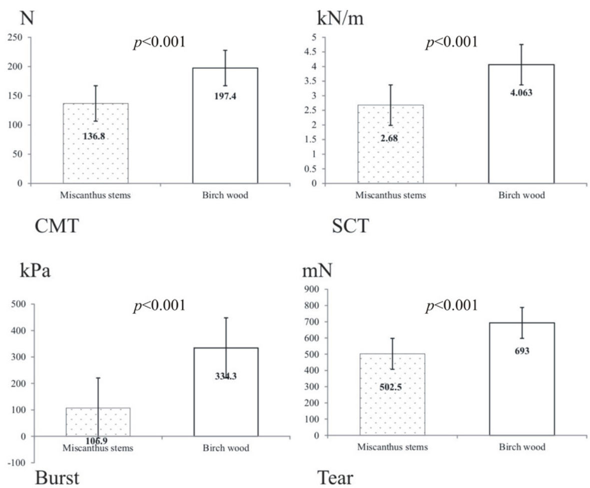

Fig. 5: Comparison of strength properties of neutral sulfite pulps from Miscanthus stems and birch wood. Experimental conditions: Miscanthus – impregnation, H 116, 7.4% Na_2SO_3 on o.d. mass, 40°SR; birch – impregnation, H 187, 49% Na_2SO_3 on o.d. mass, 40°SR.

In subsequent tests (Fig. 5), hand sheets obtained from *Miscanthus* fibers and from the sawdust of birch wood that had been cooked in the same way were tested. Digesting included initial impregnation, different conditions of the cooking time and the alkali charge. The difference was in the use of a longer cooking time and a larger alkali charge in the case of birch wood, which improved this raw material refining conditions in the laboratory mill. The indices of the tested pulp (CMT, SCT, Burst, Tear) refined to similar degrees of the Schopper-Riegler clearly confirmed that the stems of *Miscanthus* were a poorer raw material for the production of neutral sulfite pulps than birch wood. Similar results were obtained for different series of comparisons (Figs. 4 and 5), differing in the pulp's freeness, with the greatest differences for the burst strength indices in all tests. Although the neutral sulfite pulps from *Miscanthus* stems are not characterized by such high strength properties as birch pulps, the relatively small differences in the SCT index and tear resistance suggest that this raw material can be used as a supplementary raw material for birch wood.

Tab. 4: Strength properties of neutral sulfite pulps from birch wood (90%) with the addition of Miscanthus stems (10%). Experimental conditions: impregnation, digestion H 131, 41% Na_2SO_3 on o.d. mass, l/w 3. Freeness: 30°SR.

Tear (mN)	Burst (kPa)	CMT (N)	SCT ($kN{\cdot}m^{-1}$)
757.5	377.7	220	4.3

This observation is supported by data in Tab. 4, which show the relatively good strength properties of birch pulp obtained with the addition of 10% of *Miscanthus* stems.

CONCLUSIONS

1. The stems of *Miscanthus* are a raw material with poorer papermaking properties as compared to birch wood due to the smaller share of fibrous anatomical elements in the mass unit, the high content of fine fraction (parenchyma and other cells), and a high (above 1) Runkel coefficient. Despite the relatively high coefficient of slenderness, the above-mentioned factors will adversely affect the dynamic strength properties of the paper (e.g. tear resistance) and the stiffness indices of the pulp.

2. The thin walls of the fibrous anatomical elements and the lack of significant areas in woody stems of *Miscanthus* facilitate the rapid delignification and improvement of defibrability of the stems. The *Miscanthus* fibers, after the neutral sulfite pulping process, were characterized by a higher slenderness coefficient than the birch fibers, but the flexibility coefficients (lower value) and Runkel coefficient (higher value) were worse than in the case of the birch fibers.

3. Statistical analysis of the results of the strength determinations clearly indicates that the stems of *Miscanthus* are a poorer raw material for the production of neutral sulfite pulps than birch wood. Although the neutral sulfite pulps from *Miscanthus* stems are not characterized by high strength properties such as birch pulps, the relatively small differences in the SCT index and tear resistance suggest that this raw material can be used as a supplementary raw material for birch wood.

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