

Selected Grass Plants as Biomass Fuels and Raw Materials for Papermaking, Part II. Pulp and Paper Properties

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The yield and kappa number of kraft pulps from tall wheatgrass, tall fescue, tall oatgrass, and *Miscanthus* were determined after pulping with 0.9% active alkali per 1% lignin content in raw materials. Fibre properties and test papers were also studied to evaluate the usefulness of these plants for papermaking. These results were compared with pulps prepared from birch and pine wood. Kraft pulps from the straws of grasses had yields similar to that of pulp from pine wood and lower kappa numbers than pulps from birch and pine wood. The tested pulps exhibited a favourable number of fibres in 1 g of pulp, and they resulted in papers with clearly differentiated properties from very resistant to rupture dense papers with very low air permeability, to less resistant to breaking more bulky papers.

Keywords: Tall wheatgrass; Tall fescue; Tall oatgrass; *Miscanthus × giganteus*; Kraft pulping; Properties of pulps

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INTRODUCTION

The cultivation of tall wheatgrass (*Elytrigia elongate*), tall fescue (*Festuca arundinacea*), tall oatgrass (*Arrhenatherum elatius*), and *Miscanthus × giganteus* has recently begun in Poland. In Part I of this study, the yields of these crops per hectare of cultivation, their calorific value, and their chemical compositions were presented (Danielewicz *et al.* in press). The suitability of the straws of these grass crops for papermaking from the point of view of their chemical composition was also discussed.

The literature regarding the processing of different grass straws into fibrous pulps is relatively rich, and it concerns mainly the application of wheat straw and rice straw in papermaking. Apart from this type of work, one can also cite the following works in this field (Kordsachia *et al.* 1991, 1993; Wisur *et al.* 1993; Madakadze *et al.* 1999; Thykenson *et al.* 1998; Law *et al.* 2001; Ghatak 2002; Obi Reddy *et al.* 2014).

Relatively few studies have dealt with the delignification of grass plants in the kraft pulping process and the papermaking properties of kraft pulps. For example, Pahkala *et al.* (1997) studied the effects of processing of tall fescue into kraft pulp but did not determine the properties of test papers from the obtained pulp. The properties of thermomechanical pulp (TMP) and bleached chemi-thermomechanical pulp (CTMP) from *Miscanthus* have been determined (Cappelletto *et al.* 2000). Marín *et al.* (2009) studied the effects of semi-mechanical pulping of *Miscanthus* on pulp properties. Janson and Jousimaa (1996)

investigated the papermaking potential of tall fescue using the autocausticizable salt trisodium phosphate as cooking alkali and anthraquinone as catalyst. The totally chlorine free (TCF) bleaching of acetosolv and organosolv pulps has been reported (Villaverde *et al.* 2009a,b, 2011), and the general suitability of *Miscanthus* for paper production has been examined (Ververis *et al.* 2004).

The properties of kraft pulps from tall wheatgrass, tall fescue, tall oatgrass, and *Miscanthus × giganteus* have not been examined sufficiently. A comparison between these kraft pulps with the papermaking properties of birch and pine wood is needed, particularly from the point of view of the pulp and paper industry.

Thus, in Part II of this work, tall wheatgrass, tall fescue, and tall oatgrass straws and the stalks and leaves of *Miscanthus × giganteus* were assessed for their suitability in paper production. The yield of unbleached kraft pulps from these raw materials and their kappa numbers were determined. The delignification process was investigated, and morphological characteristics of fibres and the properties of test papers were evaluated in comparison with pulps from birch and pine wood.

EXPERIMENTAL

Fibrous Raw Materials

Straws from the following grass species were used: tall wheatgrass [*Elytrigia elongata* (Host) Nevski], tall fescue (*Festuca arundinacea* Schreb.), and tall oatgrass (*Arrhenatherum elatius* J. et C. Presl). *Miscanthus* (*Miscanthus × giganteus* Gref et Deu.) stalks and leaves were also used.

Typical wood materials used in the Polish papermaking industry, *i.e.*, wood chips from birch (*Betula pendula*) and pine (*Pinus sylvestris*), were obtained from the International Paper Mill in Kwidzyn, Poland.

Pulping of Fibrous Raw Materials

Pulping of fibrous raw materials was performed in an autoclave with a capacity of 300 mL and placed in a rotating sectional digester (Santasalo-Sohlberg). White liquor of 25% sulfidity was used in the experiments. The active alkali to be dosed (on the basis of oven dry amount of the specific plant material) was calculated by multiplication of the lignin content in the plant material by the factor 0.9. Thus, for tall wheatgrass, tall fescue, tall oatgrass, stems and leaves of *Miscanthus*, birch, and pine, the amounts of active alkali dosed per oven-dried raw material was 16.5%, 14.3%, 13.9%, 18.6%, 16.2%, 18.4%, and 25.7%, respectively.

The ratio of liquid to raw material, heating-up time, and time of pulping at the pulping temperature were the same in all experiments and were as follows: 5:1, 90 min, and 90 min at 165 °C. Pulped raw materials were pre-washed with water on a sieve from black liquor, washed by diffusion for 24 h, and finally disintegrated in a laboratory propeller disintegrator.

Determination of Fines Content in Pulps

Ten grams of oven-dried pulp was disintegrated in 2 L of water in a laboratory disintegrator for 3 min and then transferred to a laboratory distributor and diluted with water up to a total volume of 5 L. Next, the suspension was poured into a Büchner funnel

with a wire net bottom with openings of $75 \times 75 \mu\text{m}$. Then, the fraction of fibres was collected, diluted with water again up to a total volume of 5 L, and filtered again through the sieve. The procedure was repeated for each pulp until the water passing through the metal net was clear. Six such cycles were carried out for each pulp. After the last cycle, the fraction of fibres was collected from the wire net of the Büchner funnel, dried, and weighed. The amount of fines was calculated from the difference between the mass of the dry pulp sample subjected to analysis, and the dry mass of the fibre fraction collected from the metal net at the end of the analysis.

Extraction of Grass Straws with 1% Solution and UV Measurements of Filtrates

Extraction of raw materials from sawdust with a 1% NaOH solution was performed according to TAPPI test method T 212 om-02 (2002). After the extraction, 5 mL of filtrates were collected and diluted with water at a ratio of 1:50 in a 250-ml glass flask. UV spectra were obtained with a T70 UV-VIS spectrophotometer (PG Instruments, United Kingdom).

Microscopic studies of pulps

Small amounts of pulp were stained on microscope glass using standard Herzberg solution. Photographs were taken with a Biolar DT optical microscope (PZO, Poland).

Determination of Properties of Pulps and Papers

Characterization of pulps involved the following parameters: kappa number (PN-85/P50095/02 1985), number of fibres in 1 g of pulps and their dimensions (MORFI LB-01 apparatus, Techpap, France), brightness (ISO 2470 1999), and Shopper-Riegler (SR) number (PN-EN ISO 5267-1, 2002). Pulps were beaten in a Jokro beater according to the PN-EN 25264-3 standard (1999), while the paper handsheets were prepared according to the PN-EN ISO 5269-1 standard (2005). From paper sheets, bulk (PN-EN ISO 534, 2012), tensile strength, stretch, and TEA (PN-EN ISO 1924-2, 2010), tear index (PN-EN ISO 1974, 2012), and air resistance (TAPPI T 460 om-02, 1998) were determined for handsheets of $75 \pm 3 \text{ g/m}^2$.

Elaboration of Results

Kraft pulping experiments of fibrous raw materials were repeated twice. Therefore, the yields of pulps and their kappa numbers were the arithmetic average of two procedures performed in parallel. The standard deviations in the number of fibres in a weight unit of pulps, the content of fines, weighted average length and width of fibre, coarseness, brightness, SR freeness, tensile index, stretch, TEA, tear index, bulk, and air permeability are shown under the tables containing these results.

RESULTS AND DISCUSSION

Figure 1 shows the yield and kappa number of pulps obtained from the kraft pulping of straws from tall wheatgrass, tall fescue, tall oatgrass, and *Miscanthus*. In the process, the amount of active alkali (AA) dosed per oven-dried material was proportional to the lignin content of these straws (0.9% AA per 1% lignin content).

The unbleached pulps produced from tall wheatgrass, tall oatgrass, and stems and leaves of *Miscanthus* were characterized by yields in the range of 42% to 44%. These

results are similar to the yield of unbleached regular kraft pulps from softwood (paper grade pulp) (Sjöström 1981; Danielewicz and Surma-Ślusarska 2011b). The kappa numbers of pulps from tall wheatgrass, tall fescue, tall oatgrass, and *Miscanthus*, however, were similar to that of the unbleached pulps, or even oxygen-delignified birch pulp (Valchev *et al.* 1999; Danielewicz and Surma-Ślusarska 2007a). It is in fact included in the range of 12 to 17 kappa number units. Comparison of the kappa numbers of pulps from straws indicates that the lignin of tall oatgrass and *Miscanthus* stems was more susceptible to degradation by kraft pulping than the lignin of tall wheatgrass and *Miscanthus* leaves, and the lignin in *Miscanthus* leaves was less susceptible to degradation than the lignin in the stems of this plant.

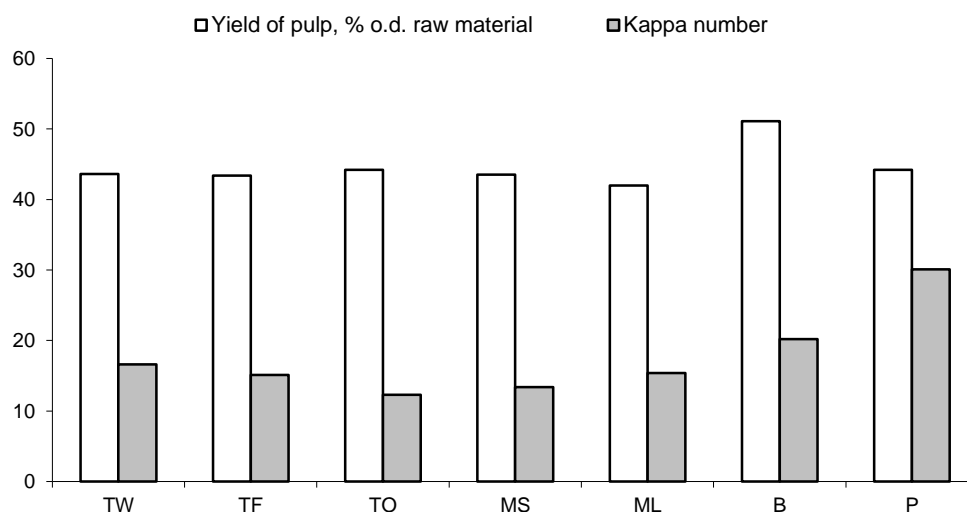


Fig. 1. The yield of pulps of fibrous raw materials and their kappa number (TW- tall wheatgrass; TF – tall fescue; TO – tall oatgrass; MS – *Miscanthus* stems; ML – *Miscanthus* leaves; B – birch; P – pine)

The increased susceptibility of lignin in grass plants to delignification in alkaline pulping results from hemilignin (lignin with a lower degree of polymerization) in these plants, as well as the different nature of the biopolymer and carbohydrate bonds, in particular, the greater proportion of ester bonds that form the residues of ferulic and p-coumaric acids of lignin with xylan (Buranow and Mazza 2008; Vogel 2008). During the alkaline treatment of biomass, saponification of these intermolecular bonds is the first reaction to take place (Wang and Liu 2012).

Figure 2 shows the absorption spectra of filtrates from the extraction of raw material sawdust with 1% NaOH, per a standard test method (TAPPI T212 om-02, 2002). The absorption peaks of lignin appear at the 205, 230, and 280 nm frequencies, with the proviso that absorbance at 280 nm declines continuously (Schmidt 2010). Extraction filtrates from tall wheatgrass, tall fescue, tall oatgrass, and, above all, *Miscanthus* stalks and leaves exhibited higher absorption at these frequencies than birch or pine wood. This result indicates that more lignin was extracted by 1% NaOH from grass plants than wood.

The higher amount of ester bonds in lignin from the grass plants may affect the rate of its delignification; this hypothesis is confirmed by the stronger absorption at 315 nm in grass plant filtrates. Maxima in this region correspond to the conjugated phenolic units in

ferulic and p-coumaric acid and hydroxycinnamic acid-type structures in lignin (Pan and Sano 1999; Oliveira *et al.* 2009; Zhao and Liu 2010). The intensity of light absorption in this region was in relatively good agreement with the degree of delignification of grass raw materials during kraft pulping. Tall wheatgrass and tall fescue straws underwent less pulping than the stalks of *Miscanthus* and tall oatgrass (Fig. 1).

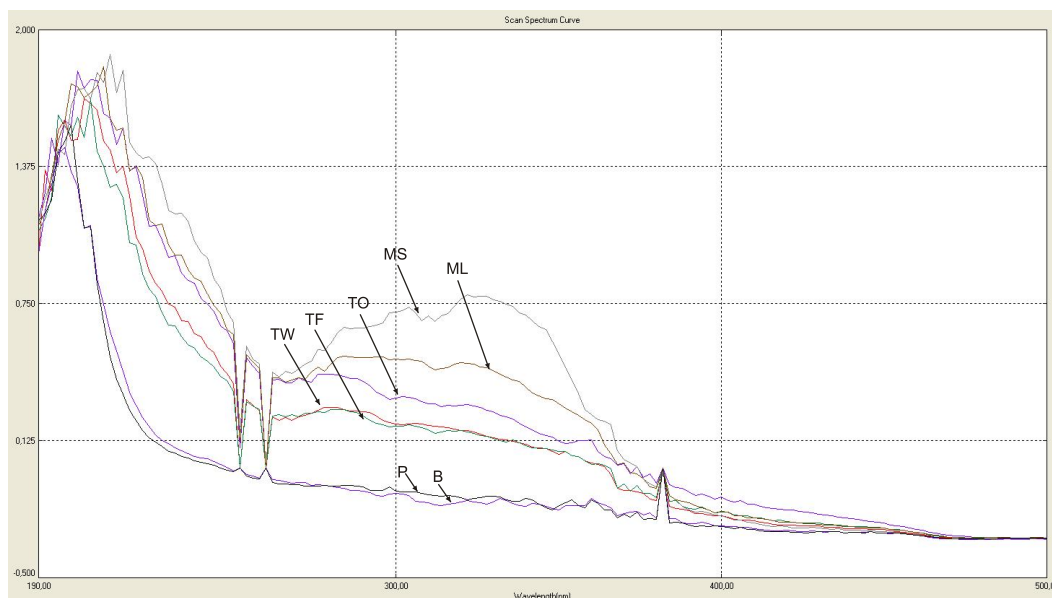


Fig. 2. Spectra of the filtrates of 1% NaOH treatment of sawdust of tall wheatgrass (TW), tall fescue (TF), tall oatgrass (TO), *Miscanthus* stalks (MS) and leaves (ML), as well as sawdust of pine (P) and birch (B) water (dilution 50 \times , Y axis - absorbance)

The reduction of paper basis weight, increased yield in pulping, increased proportion of mineral fillers, and recovery of fibres from waste paper are ways to use less primary fibres from forest materials during paper production (reviewed in Hubbe 2014). However, non-wood fibres could also partially replace forest material fibres. The combined pulping of wood with non-wood raw material is an attractive alternative in this respect, but the higher susceptibility of grass straws to pulping is a problem for cellulosic mills because the draining sieves of digesters can become clogged with pulped pieces of straw. Additionally, these straws contain too much silica and other mineral substances, but this problem may be partially solved in the near future (Paavilainen 1996; Tutus and Eroglu 2004; Kumar 2012).

In the evaluation of grass plants for papermaking, fibre content and morphology, the fines fractions in pulps are important factors (Heikkurinen *et al.* 1991; Mohlin 1999; Tiikkaja 1999; Seth 2003). These characteristics are shown in Tables 1 and 2. The criterion that decides whether cells in the pulps are fibre or fines is length; fibres have a length greater than 0.2 mm (Seth 2003).

While the fibre contents of pulps from tall wheatgrass, tall fescue, and *Miscanthus* stems and leaves were similar (8.8 to 13.2 million), the number of fibres in pulp from tall oatgrass was noticeably different (20.0 million) (Table 1). In the case of tall wheatgrass, tall fescue, and *Miscanthus* pulps, the fibre content was similar to that of beech, hornbeam, and birch pulp; tall oatgrass fibre content was twice as high but close to the fibre content of acacia wood pulp, which is also used for the preparation of fibrous papermaking pulps

(Paavilainen 2000; Mohlin and Hornatowska 2006; Danielewicz and Surma-Ślusarska 2007b, 2010).

Table 1. Fibres and Fines Content in Pulps of Various Fibrous Raw Materials

Raw Material	Number of Fibres in 1 g of Pulp (10 ⁶ /g)	Fines Content (wt.%)
TW	9.5	7.9
TF	13.2	12.4
TO	20.0	10.0
MS	8.8	16.3
ML	10.7	19.2
B	11.1	2.7
P	3.8	2.2

Standard deviations in the number of fibres and the fines contents ranged from 0.3 to 1.1 × 10⁶ and from 0.3 to 0.6%, respectively.

In the straws of grasses, a fibre content of up to 20 million per g of pulp is considered beneficial, as a higher fibre content is associated with a reduction in fibre length. For example, hemp core pulp has a fibre content above 20 million and a mean fibre length of 0.5 mm (Kovacs *et al.* 1992; Danielewicz and Surma-Ślusarska 2011a). Shorter fibre length results in a very low resistance to tearing in test papers (Zomers *et al.* 1995; de Groot *et al.* 1999; Danielewicz 2013).

The pulps from grass straws was also distinguished from wood pulps by the high content of primary fines (Table 1). The fines content was 3 to 8 times higher in grass pulps than in birch or pine. In tall wheatgrass pulp, the fines content was comparable to that of beech and acacia pulps (Danielewicz and Surma-Ślusarska 2010), while in tall fescue, tall oatgrass, and *Miscanthus* pulps, it was 1.2 to 2.2 times higher than in these wood species. Pulps from *Miscanthus* stems and leaves contained especially large amounts of fines (16.3 and 19.2 wt. %, respectively).

Table 2. Properties of Fibres of Kraft Pulps

Raw Material	Fibre Length (weighed; mm)	Fibre Width (µm)	Coarseness (mg/m)
TW	0.920	28.5	0.165
TF	0.914	24.9	0.121
TO	0.825	21.6	0.081
MS	1.068	23.7	0.169
ML	0.896	23.3	0.143
B	0.920	21.3	0.117
P	2.342	30.9	0.192

Standard deviations in fibre length, width, and coarseness ranged from 0.021 to 0.043 mm, 1.1 to 2.4 µm, and 0.005 to 0.012 mg/m, respectively.

Pulps from straw of grasses showed reasonably good results in terms of average fibre length. As shown in Table 2, these values were similar to the average fibre length of birch pulp and greater than the average fibre length of pulps from *Acacia* species (0.66 to 0.73 mm), eucalyptus, and hemp core (Kovacs *et al.* 1992; Paavilainen 2000; Mohlin and Hornatowska 2006; Danielewicz and Surma-Ślusarska 2010; Danielewicz 2013).

Fibres from tall fescue and *Miscanthus* stems and leaves had greater widths than birch pulp fibres. Tall wheatgrass fibres had a width similar to pine pulp fibre (Danielewicz and Surma-Ślusarska 2010). The coarseness of tall wheatgrass and *Miscanthus* fibres was higher than for fibres of tall fescue and *Miscanthus* leaves pulps and especially tall oatgrass. The similar widths of tall oatgrass and birch pulps fibres suggest that this grass has much lower cell wall thickness. Similarly, *Miscanthus* stalks pulp fibres would have much greater cell wall thickness than the other grasses.

Table 3. Properties of Unbeaten Pulps and Unbleached Kraft Pulps from Grass Straws and Birch and Pine after a 12-min Beating in the Jokro Mill

Pulp	Brightness (%)	SR Freeness Before Beating (°SR)	SR Freeness After Beating (°SR)	Tensile Index (N·m/g)	Stretch (%)	TEA (J/m ²)	Tear Index (mN·m ² /g)	Bulk (cm ³ /g)	Air Resistance (sec)
TW	34.2	17	50	73.2	2.5	84.8	8.0	1.624	477.3
TF	33.7	20	55	99.8	3.2	124.8	8.0	1.315	1032.8
TO	31.9	26	41	91.4	3.2	117.6	7.8	1.229	>30 min
MS	33.3	14	32	58.9	2.1	57.1	10.8	1.441	78
ML	32.4	17	46	56.5	2.3	64.3	5.4	1.354	>30 min
B	27.5	16	19	92.3	2.0	81.6	8.4	1.454	19.4
P	27.0	12	15	76.1	2.0	68.5	11.2	1.662	12.6

Standard deviations in brightness, SR freeness, tensile index, stretch, TEA, tear index, bulk, and air permeability ranged from 0.5 to 1.9%, 1 to 2 °SR, 1.1 to 4.4 N·m/g, 0.1 to 0.3%, 2.3 to 5.6 J/m², 0.2 to 0.5 mN·m²/g, 0.030 to 0.052 cm³/g, and 2.3 to 16.4 sec, respectively.

Table 3 shows the properties of the pulps obtained from grass straws and wood after the same beating time (12 min) in a Jokro mill. Brightness of kraft pulps from tall wheatgrass, tall fescue, tall oatgrass, and *Miscanthus* stems and leaves was 4.6% to 6.9% higher than the brightness of kraft pulps from pine and birch. The Schopper-Riegler (SR) freeness of grass straw pulps after beating was higher than the SR freeness of pulps from birch and pine. Thus, these pulps show a much higher susceptibility to beating, which is beneficial for paper pulps. On the other hand, higher SR values of grass pulps are unfavourable because this number determines the drainage rate of pulp on the wire (Seth 2003). After the equally intense beating of pulps from grass straws, they were characterized by varying tensile strength. The highest value of tensile index (comparable to pulp birch) was that of pulps from tall fescue and tall oatgrass. Pulp from tall wheatgrass had far lower values, and pulps from the stems and leaves of *Miscanthus* had the lowest. Compared with the tensile index determined for kraft pulps from hardwood species (Fišerová and Gigac 2011), the test paper sheets from grass pulps generally had good tensile strength. Tall fescue and tall oatgrass kraft pulps were as strong as birch, hornbeam, and poplar pulps. Tall wheatgrass pulp has tensile strength similar to beech and oak pulp, while pulps from *Miscanthus* were characterised by tensile index similar to black locust pulp. Test papers from non-wood pulps are usually characterized by higher elongation values than birch and pine pulps, even when their tensile strength is lower. Because of the higher elongation, test papers from grass straw pulps can absorb similar amounts of energy during tensile tests (TEA values, Table 3) as birch pulp (pulp of tall wheatgrass) or pine pulps (pulp from *Miscanthus* leaves) or more than birch pulp (tall fescue and tall oatgrass pulps).

Non-wood pulps fared better in terms of resistance to tearing. The tear index of tall wheatgrass, tall fescue, and tall oatgrass pulps was similar to the tear index of birch pulp, whereas *Miscanthus* stems pulp was similar to pine pulp. The lowest resistance to tearing was shown by the test papers from *Miscanthus* leaves ($5.4 \text{ mN}\cdot\text{m}^2/\text{g}$). The bulk of test papers from grass straws pulps, which is important for printing papers and paperboard (Engman 1989; Koran 1989), also varied. For tall fescue, tall oatgrass, and *Miscanthus* leaves pulps, bulk was lower than for test papers from birch pulp; *Miscanthus* stem pulp paper was comparable to birch pulp test paper. For test papers made from tall wheatgrass pulp, bulk was comparable that of pine pulp handsheets. The high bulk of test papers made from the tall wheatgrass pulp is interesting and can be explained by the larger width and coarseness of its fibres.

The air resistance of test papers from non-wood pulps was also examined using the Gurley method, which is a measure of porosity. Test papers made from grass pulps had a much smaller porosity than test papers prepared from birch and pine pulps. Sheets from *Miscanthus* stems pulp had the most porous structures, while test papers from tall wheatgrass and tall fescue pulps had structures with low porosity. Test papers made from tall oatgrass and *Miscanthus* leaves were almost impermeable to air under the pressure used in the Gurley apparatus. In the case of *Miscanthus* leaves pulp, this effect resulted from the high fines content, but in the case of tall oatgrass pulp, it could also be due to the distinctly lower coarseness of the pulp fibres. The low air permeability was achieved without intensively beating the pulps or damaging the fibres, which could be beneficial in some grades of paper (tracing paper, papers for packaging of food products, *etc.*). On the other hand, reduced porosity retards moisture escape from paper sheets and slows drying. Both factors contribute to decreased pulp or paper machine productivity (Seth 2003).

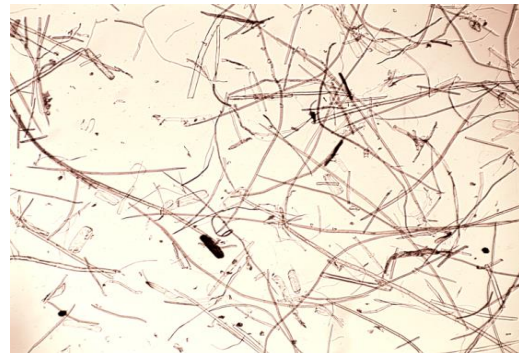
The properties of grass pulps can be explained by fibre morphology. For example, the high tensile index for tall fescue and tall oatgrass pulps can be explained based on good suitability for collapse of fibres of these pulps, resulting from their low coarseness, as well as with higher number of load-bearing layers in the sheet. Good resistance to tearing in tall wheatgrass and tall fescue test papers may be due to long fibres near the short fibres in these pulps (Fig. 3). These phenomena were also observed in hemp stalk pulp. While it contains relatively low numbers of very long bast fibres, these fibres significantly boost tear resistance in the hemp stalk test papers (Danielewicz 2013).

The high tear strength of *Miscanthus* stalks pulp could result from the higher average length fibres and higher coarseness than in tall wheatgrass and tall fescue pulps. The latter parameter influences fibre stiffness, which increases tearing work by increasing the energy consumed in the bending and interaction of the edges of paper being torn (Paavilainen 1993).

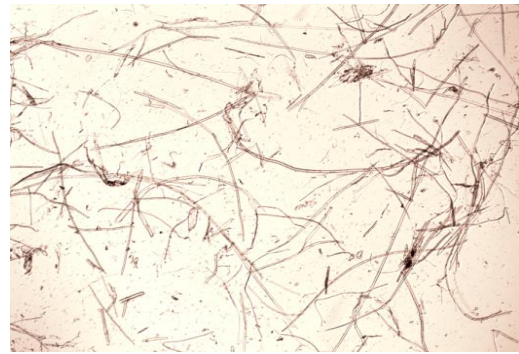
The high bulk of tall wheatgrass pulp is due to the greater width of its fibres (Table 2), as is visible in Fig. 3, and also from high coarseness. High air impermeability of paper from tall oatgrass pulp can be explained by the low coarseness of its fibres. This allows the fibres to create a very dense paper mat, which is additionally sealed by the fines (10% of this pulp). According to Paavilainen (1993), fibre coarseness explains 70% and 80 %, respectively, of the variation in air density and tear in handsheets from the cellulosic pulps of European and American softwood species.



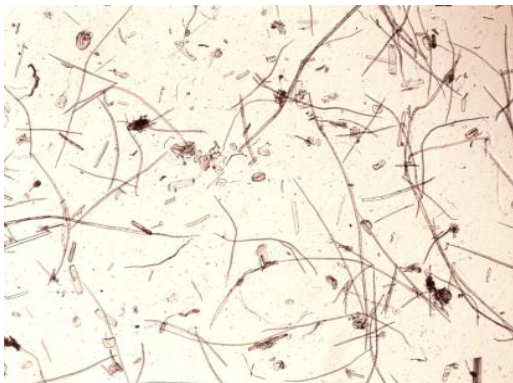
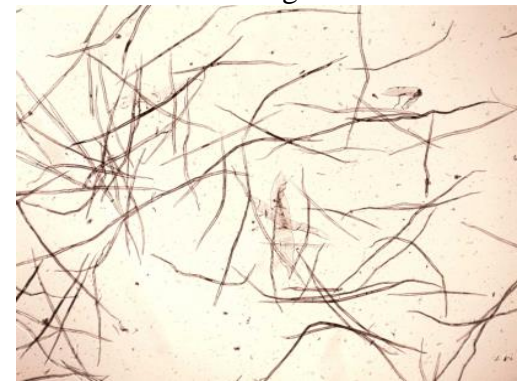
Tall wheatgrass



Tall fescue

*Miscanthus* stalks

Tall oatgrass

*Miscanthus* leaves

Birch



Pine

Fig. 3. Pulps from grass straws and birch and pine wood under an optical microscope (40× magnification)

CONCLUSIONS

1. The straws of tall wheatgrass, tall fescue, tall oatgrass, and *Miscanthus* stems and leaves were characterized by higher susceptibility to delignification during kraft pulping than birch and pine.
2. Unbleached kraft pulps from grasses were similar in fibre content to pulps from European hardwood species, such as beech, hornbeam, birch, and acacia.
3. The fibre length of pulps from straw grasses was similar to the fibre length of pulps from hardwood, while the fibre width ranged between those of birch and pine pulps.
4. Unbleached pulps from tall wheatgrass, tall fescue, tall oatgrass, and *Miscanthus* stems and leaves have varying properties. Tall fescue and tall oatgrass pulps can be used in paper products that require good tensile strength, elongation, TEA, and low air permeability, while less attention is paid to good drainage performance and high bulk. *Miscanthus* stem pulp can be used where a low Shopper-Riegler freeness, high tear resistance, and higher air permeability are needed. With regard to the pulps from tall wheatgrass and *Miscanthus* leaves, it is worth underlining the high bulk of the former and the low tensile strength and tear resistance of the latter which limit its use.
5. Pulps from tall wheatgrass, tall fescue, tall oatgrass, and *Miscanthus* stalks can be regarded as alternative sources of fibers that can be used to produce definite kinds of paper and cardboard products. *Miscanthus* leaves pulp was the worst material in this regard.

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