SUSCEPTIBILITY OF SELECTED NON-WOOD FIBROUS RAW MATERIALS FOR PROCESSING INTO UNBLEACHED AND BLEACHED KRAFT PULPS

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The susceptibility of *Miscanthus* × *giganteus*, wheat straw and industrial hemp to delignification during the kraft pulping process was compared with that of birch and pine. The obtained pulps were then used to evaluate the susceptibility of their residual lignin to degradation during oxygen delignification and hydrogen peroxide bleaching processes. In the final part of work, the content of chromophoric moieties in the selected raw materials and in the produced unbleached pulps, as well as their susceptibility to degradation during oxygen delignification and hydrogen peroxide bleaching, was indirectly compared. The research shows that native lignin of *Miscanthus* × *giganteus* and wheat straw, as well as residual lignin and chromophoric groups in the pulps from these raw materials, is easier to degrade in typical delignification processes in comparison to those of hemp, birch and pine.

Keywords: *Miscanthus* × *giganteus*, wheat straw, hemp, birch, pine, delignification, susceptibility

INTRODUCTION

The earth's human population is constantly growing and, therefore, the demand for paper and board is also expected to grow in the near future.[1] One of the major problems that the pulp and paper industry may face soon is lack of wood because of the growing demand for this material and the limited quantities of wood that can be obtained from forests, considering the need to protect them.[2-4]

One of the solutions to this problem could be the wider use of non-wood plant fibrous raw materials (NPFRMs) for papermaking. A quite large number of NPFRMs can be used for this purpose.[5-32] These NPFRMs are also intensively researched due to their usefulness for the production of energy[33-38] and chemicals.[39,40] However, it should be pointed out that combustion of NPFRMs cannot be regarded as the most rational way of their utilization. Instead, NPFRMs could be possibly first processed into higher-value added products, such as paper, paperboard or products from their chemical constituents (*e.g.* viscose textiles), used once or several times and burned only when their functional properties are lost.

It is commonly known that plant fibrous raw materials (PFRMs) can be transformed into pulps for papermaking. This is done through chemical pulping and/or mechanical defibering processes. Pulps for certain applications have to be further refined through other chemical purification processes, aiming at delignification and degradation of chromophoric groups.

This paper presents a comparison of the susceptibility of wheat straw, *Miscanthus* × *giganteus*, hemp, birch and pine to delignification during the kraft pulping process, as well as the susceptibility of residual lignin (RL) and chromophoric moieties (CM) of the kraft pulps from these fibrous raw materials to degradation through selected bleaching processes.

These non-wood plants were selected for the study considering their possible cultivation under the climatic conditions prevailing in Poland.

EXPERIMENTAL

Characteristics of fibrous raw materials

In the study, air-dried stalks of *Miscanthus* × *giganteus* were obtained from a plantation in the Majdan Sieniawski village in Podkarpackie province; wheat straw – from a farm in the Kraszew village in Lodz province; industrial hemp stalks were obtained from the Institute of Natural Fibres and Herb Plants in Poznan; while industrial birch and pine chips were obtained from the International Paper Kwidzyn. Stems of *Miscanthus*, wheat straw and hemp were cut before pulping to lengths of 3-10 mm.

Kraft pulping process

The kraft pulping of PFRMs was performed in a sectional, rotary Santasalo-Sohlberg digester, in autoclaves with a capacity of 300 cm^3. Pulping liquors of 25% sulfidity and different active alkali amounts for each raw material were used. Active alkali (AA) was dosed in an amount proportional to the lignin content in the raw materials. The amount of active alkali for each fibrous raw material was calculated by multiplying the content of lignin in each raw material by a coefficient of 0.9. Therefore, for *Miscanthus*, wheat straw, hemp, birch and pine, the dosed quantity of AA amounted to 18.5, 20.0, 16.4, 18.4 and 25.7% on oven-dry (o.d.) fibrous raw material, respectively. The liquid-to-raw material ratio, heating-up time, pulping time and temperature of pulping were the same for all the PFRMs, as follows: 5, 90 min, 90 min and 165 °C, respectively. The pulped PFRMs were pre-washed with water on a sieve, washed by diffusion for 24 hours, disintegrated in a laboratory disintegrator and finally screened in a Weverk sorter equipped with 0.2 mm slot screen. The obtained pulps were dried, their kappa numbers were determined and then the percentage content of residual lignin (RL) was calculated by multiplying kappa number by a factor of 0.15. These percentage contents of lignin in the pulps were then used for calculating the amount of NaOH and H_2O_2 needed for pulp delignification in oxygen delignification and peroxide bleaching processes. Pulping was performed in duplicate for each PFRM.

Removal of metal ions (stage A)

Before oxygen delignification and bleaching with hydrogen peroxide, the transition metal ions were removed from the pulps through their treatment with dilute sulphuric acid at pH 3. The treatment was performed using 10% consistency of the fibre suspension, at 75 °C for 1 hour. After the process of metal ions removal from the pulps, the pulp slurry was filtered through a Büchner funnel, washed with 1000 cm^3 of distilled water, again filtered and stored for further treatment.

Oxygen delignification (stage O)

For the experiments, 20 g (o.d.) samples of pulps were used. These samples were initially disintegrated in water, then filtered and dewatered by pressing. Next, the amount of distilled water to obtain 8% consistency of pulp suspension was calculated. An amount of 0.2% sodium hydroxide and magnesium sulphate on o.d. pulp was mixed with water and added to the pulp. The amount of sodium hydroxide was calculated proportionally to the residual lignin content in each pulp. This amount was determined by multiplying the percentage content of lignin in each pulp by a coefficient of 0.5.

Oxygen delignification was conducted in a Jayme reactor. The autoclave was closed and filled with oxygen to a pressure of 0.5 MPa, the mixing mechanism was switched on and the pulps were heated to 100 °C for 30 minutes. This temperature was then maintained for 60 minutes. After the pre-set time elapsed, the oxygen reactor was degassed and emptied. Then, the pulps were washed in the same way as described in stage A.

Hydrogen peroxide bleaching (P)

For the experiments, unbleached kraft pulps were used. The peroxide bleaching stage was done using a bleaching liquor containing 0.2% $MgSO_4$, 0.4% EDTA (calculated on o.d. pulp), and amounts of NaOH and H_2O_2 proportional to the RL (residual lignin) content in each pulp. The percentage quantities of NaOH and H_2O_2 used in bleaching the pulps were determined by multiplying the percentage content of lignin in the pulp by a coefficient of 1.0. Each bleaching experiment was performed with 10% consistency of the pulp suspension, at 80 °C for three hours, with mixing at 30 minute intervals. Then, the pulps were washed in the same way as described in stage A.

Processing of PFRM into RMP

In order to determine the brightness of the selected plant raw materials, they were converted to refiner mechanical pulps (RMP). In the first stage of the processing, birch and pine chips, hemp and *Miscanthus* stalks were cut into sticks, while the straw stalks were cut into fragments of 0.5-10 mm. PFRMs prepared in this way were then flooded with water and disintegrated mechanically in a Bauer laboratory refiner, at first into the form of splinters, using thick-grooved discs, and then into fibrous pulps, using narrow-grooved discs. The obtained pulps were then used to form handsheets, which were dried and used to determine the brightness of the PFRMs.

Determination of chemical and physical properties of PFRMs and pulps

- Lignin content in PFRMs, according to TAPPI T 222 om-11 standard.[41]
- Kappa number, using standard ISO 302.[42]
- Brightness of the pulps, according to ISO 2470, using the Spectrocolor 01 apparatus.[43]
- Solubility of PFRMs in 1% NaOH, according to TAPPI T 212 om-02 standard.[44]

UV measurements of filtrates

Five cm^3 of each filtrate from the determination of PFRM sawdust solubility in 1% NaOH were diluted with distilled water in the proportion of 1:50. The spectra of these filtrates in UV light were obtained using a T70 UV-VIS spectrophotometer, PG Instruments (United Kingdom).

Determination of delignification susceptibility indices of fibrous raw materials, pulps and kraft pulps

The index of susceptibility of PFRMs to delignification in the kraft pulping process, S_1, was calculated by dividing the amount of lignin removed during kraft pulping (difference between the lignin content in the raw materials and the content of residual lignin in the kraft pulps, both expressed in grams), by the amount of active alkali used for pulping the PFRM sample (also expressed in grams). When calculating the lignin content in the pulps, its yield in the pulping process was taken into account. The lignin content in the pulp was determined by multiplying the kappa number by 0.15.

The index of susceptibility of PFRM to delignification in the oxygen delignification and peroxide bleaching processes, S_2 and S_3, respectively, was calculated by dividing the amount of lignin removed during these processes from the pulps (the difference between the residual lignin content in unbleached kraft pulps and the content of residual lignin in oxygen delignified and peroxide bleached pulps, expressed in grams) by the amount of NaOH used in these processes, also expressed in grams.

The susceptibility indices of the pulps' chromophoric moieties (CM) to degradation during oxygen delignification and peroxide processes, S_4 and S_5, were calculated by dividing the pulp brightness change during these processes by the amount of NaOH used in the process, expressed in percentages.

Elaboration of results

The results of all the determinations performed in this work, *i.e.* the lignin content in PFRMs, solubility of PFRMs in 1% NaOH, kappa number of the pulps after kraft pulping, oxygen delignification and hydrogen peroxide bleaching, and pulp brightness, are expressed as the arithmetic mean of two procedures carried out under identical conditions.

RESULTS AND DISCUSSION

Figure 1 shows the content of lignin in the PFRMs selected for the study, the kappa number of the kraft pulps from these PFRMs and the index of PFRM susceptibility to delignification in the kraft pulping process, S_1.

The data presented in Figure 1 show that *Miscanthus* and wheat straw are similarly susceptible to delignification in the kraft pulping process and more susceptible to delignification than hemp stalks. The values of S_1 index also indicate that *Miscanthus* and wheat straw are more prone to delignification in the kraft pulping process than birch and pine, but the difference is not high.

The higher susceptibility to delignification of *Miscanthus* and wheat straw, in comparison with birch and pine, can probably result from the presence of the so-called hemi-lignin or non-core lignin in these PFRMs, as well as from the presence of easily degradable p-coumaric acid residues in their lignin, linked *via* ester bonds (more susceptible to alkaline hydrolysis than the ether bonds occurring in lignin), and the smaller content of dimers and trimers in their lignin structure (such as phenylcoumarin, resinol and spirodienone in *Miscanthus*) than in wood, as reported in the literature.[45-48]

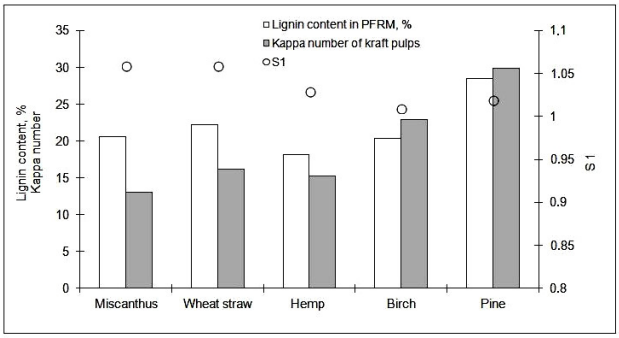

Figure 1: Lignin content in fibrous raw materials, kappa number and delignification susceptibility index of PFRMs, S_1

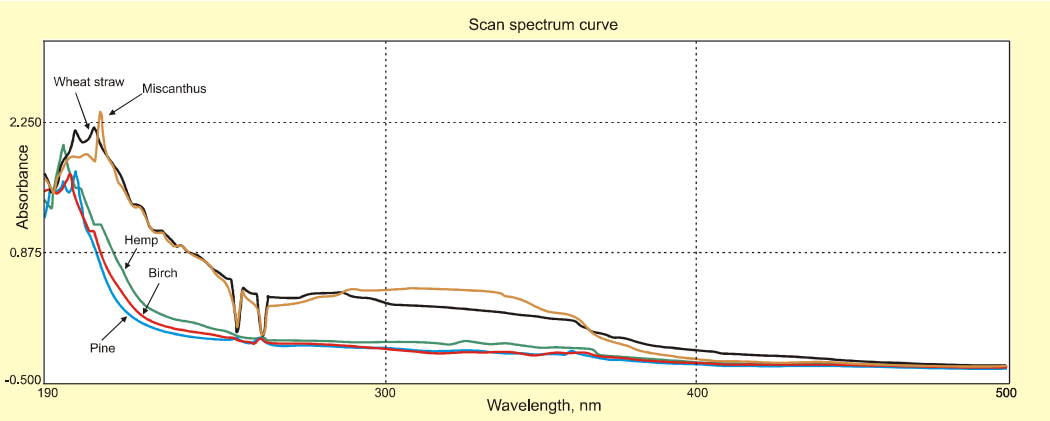

Figure 2: Spectra of 1% NaOH filtrates from *Miscanthus*, wheat straw, hemp, pine and birch

Table 1
Solubility of PFRMs in 1% aqueous solution of NaOH

PFRMs	Solubility, %
Wheat straw	38.2
Miscanthus	35.4
Hemp	29.2
Birch	16.9
Pine	14.4

A higher susceptibility of the lignin present in *Miscanthus* and wheat straw to alkali action, compared to wood, is also confirmed by the spectra of the filtrates from PFRM extraction with 1% NaOH (Fig. 2).

It is well known that the UV light absorption peaks characteristic of lignin appear at 205, 230 and 280 nm wavelengths.[49] The filtrates from the extraction of wheat straw and *Miscanthus* using 1% NaOH exhibited higher absorption at these wavelengths than the filtrates from the extractions of hemp, birch and pine wood. This result indicates that probably more lignin was extracted using 1% NaOH from wheat straw and *Miscanthus* than from other PFRMs.

The higher amount of p-coumaric and ferulic acid residues in the lignin from *Miscanthus* and wheat straw may also be confirmed by the higher absorption peaks at 315 nm. According to some authors, the maxima of absorption in this region of the UV spectra should correspond to the removal of these acids from PFRMs.[50,51] This also confirms the lower absorption of the hemp filtrate in this spectral region, because of the low content of p-coumaric acid residues in this raw material.[52]

However, it cannot be excluded that part of the absorption of the *Miscanthus* and wheat straw filtrates presented in Figure 2 may be caused by the presence of degradation products of chemical components other than lignin, including, above all, hemicelluloses.

The presence of components other than lignin in the filtrates indicates a large amount of material extracted from NPFRMs using hot 1 % NaOH, which was significantly higher than those extracted from birch and pine (Table 1).

Probably, the high solubility of some chemical components of wheat straw and *Miscanthus* in alkali can partly explain the small difference in the susceptibility of these NPFRMs to delignification in kraft pulping, in comparison with birch and pine (Fig. 1), because this feature of wheat straw and *Miscanthus* could significantly reduce the amount of active alkali in their bulk delignification phase of kraft pulping, in comparison with wood.

The data presented in Figure 1 and Table 1 also show that the susceptibility of industrial hemp to alkaline delignification or extraction was somewhat lower in comparison with that of other tested NPFRMs. This may result from the similarities between the chemical structure of lignin of woody-core hemp and that of hardwoods, and, in particular, from the low content of p-coumaric and ferulic acid residues in hemp.[52] The structure similarity between hemp woody-core and birch lignin was highlighted by the results of the course of delignification of

hemp woody-core in kraft pulping, which turned out to be similar to the delignification of birch.[53]

Further purification of pulps demands their bleaching, *i.e.* the removal of residual lignin (RL) and the degradation of chromophoric moieties (CM). The susceptibility of RL in the pulps obtained from the studied PFRMs to degradation through oxygen delignification and hydrogen peroxide bleaching processes is shown in Figure 3.

The data presented in Figure 3 show that the RLs contained in the *Miscanthus* and wheat straw kraft pulps are more susceptible to degradation by oxygen in alkaline medium than the RLs of birch and pine kraft pulps. The delignification susceptibility of the hemp pulp was lower than that of the pulps from *Miscanthus* and wheat straw.

As regards the degradation susceptibility of pulp RL in the hydrogen peroxide bleaching process, the results showed that it was again higher for the pulps from *Miscanthus* and wheat straw than for the birch and pine kraft pulps. Comparing the pine and birch pulps, the lignin in the pine pulp exhibited higher susceptibility to degradation by hydrogen peroxide in the alkaline solution, unlike the case of the oxygen delignification process. Thus, the results indicate that the chemical structure of the RLs contained in the *Miscanthus* and wheat straw pulps has a positive influence on the efficiency of their degradation in the processes of oxygen delignification and hydrogen peroxide bleaching.

An important issue, in addition to the susceptibility of native lignin and residual lignin (RL) to degradation in the kraft pulping process, is also the susceptibility of native lignin to chemical transformation leading to the formation of chromophoric moieties (CM), as well as susceptibility of those moieties to degradation in oxygen-alkali delignification or bleaching processes.

The data in Figure 4 show that the *Miscanthus*, wheat straw and birch selected for investigation have a significantly higher content of CM groups in the raw material (their corresponding RMP brightness was approx. 32, 28 and 36%, respectively) than hemp and pine (with RMP brightness of about 47% and 49.5%, respectively).

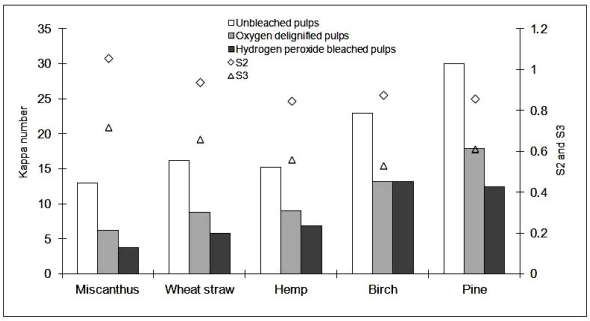

Figure 3: Kappa number of pulps and susceptibility indices of these pulps to delignification in oxygen delignification and hydrogen peroxide bleaching processes, S_2 and S_3

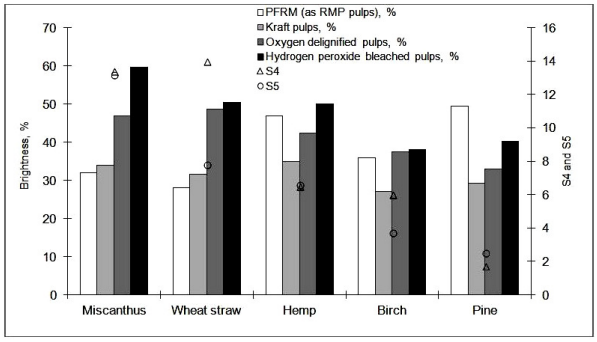

Figure 4: Brightness of PFRMs and kraft pulps before and after their processing, as well as susceptibility indices of chromophores to degradation in oxygen delignification and hydrogen peroxide bleaching processes, S_4 and S_5, respectively

However, the content of CM in *Miscanthus* and wheat straw kraft pulps is lower than in birch and pine kraft pulps, as results from comparing the brightness of these pulps. This indicates that the delignification of *Miscanthus* and wheat straw creates a lower amount of CM groups in the kraft pulping process than the delignification of birch and pine. Having found no data in the literature about the differences in the content of phenylcoumarin and pinoresinol dimmers of lignin in the studied PFRMs, precursors of such CM in kraft residual lignin like stilbene and butadiene54, the lower content of CM in the kraft pulps from *Miscanthus* and wheat straw may be partly explained by the lower content of guaiacyl (G) and syringyl type monomers (S) in these NPFRMs, resulting from the higher content of p-hydroxyphenyl monomer (H) units in their lignin than in wood lignin45,49, which are not converted to the quinone-type chromophoric structures during kraft pulping.[55-57]

Figure 4 also shows the S_4 and S_5 indices, which allows comparing indirectly the susceptibility of the CM of kraft pulp RL to degradation in oxygen delignification or bleaching with hydrogen peroxide. The obtained results indicate that the CM contained in *Miscanthus* kraft pulp were equally susceptible to degradation in oxygen delignification to those in wheat straw kraft pulp, but the CM of the latter were less susceptible to degradation by hydrogen peroxide in comparison to the former. The susceptibility to degradation of the CM in hemp kraft pulp represented almost half of that observed for *Miscanthus* pulp. The lowest susceptibility to degradation in oxygen delignification and peroxide bleaching processes was observed for the CM of pine pulp.

The observed trend of better degradability of the CM contained in *Miscanthus* and wheat straw pulps, in comparison with those of wood pulps, can be explained by the lower content of quinone structures, which can be cleaved effectively with stronger bleaching agents, such as chlorine dioxide and ozone.[58,59]

CONCLUSION

Miscanthus and wheat straw show greater susceptibility to delignification and lower tendency to form chromophoric groups in the kraft pulping process, in comparison with birch and pine wood. However, under the experimental conditions in this study, the relative difference in susceptibility to delignification between these NPFRMs and birch and pine in kraft pulping process is not high.

The susceptibility of the residual lignin and chromophoric moieties of *Miscanthus* and wheat straw kraft pulps to degradation in oxygen delignification and hydrogen peroxide bleaching processes was also better compared to that of birch and pine kraft pulps.

The susceptibility of native lignin, residual kraft lignin and chromophoric moieties of hemp to degradation in delignification processes was lower than in the case of *Miscanthus* and wheat straw.

ABBREVIATIONS

AA: active alkali
CM: chromophoric moieties
PFRMs: plant fibrous raw materials
NPFRMs: non-wood plant fibrous raw materials
RL: residual lignin
RMP: refiner mechanical pulp

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