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CELLULOSIC FIBERS FROM *MISCANTHUS*

BY

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Abstract. *Miscanthus* is an important biomass crop and has an important role in the sustainable production of renewable fuels and chemicals. It is currently used as a source of heat and electricity, or converted into biofuel products. There are many possibilities to valorize *Miscanthus* crops: combustion, gasification and pyrolysis for energy; liquefaction and hydrolysis for chemicals; delignification for cellulosic fibers. Chemical composition of *Miscanthus* is encouraging regarding its cellulose and lignin content, making them suitable for delignification. *Miscanthus* pulp can be obtained by usual delignification processes, among them soda cooking being very attractive. Pulps having different yields and lignin content were obtained by suitable choosing of the cooking parameters: NaOH addition, time and temperature. The strength properties of pulp mainly depended on its lignin content. Lignin-rich pulps show high compressive strength, being appropriate in paper manufacturing for corrugated board production.

Keywords: *Miscanthus* crop; soda cooking; chemical pulp; pulp properties; paper.

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1. Introduction

Due to the global rise in demand for energy and concerns about the growing greenhouse gas emissions, lignocellulosic biomass stands out with large potential for biofuels and biomaterials production based on the biorefinery processes. The genus *Miscanthus* comprises 17 species of perennial non-wood rhizomatous tall grasses native to subtropical and tropical regions originating from Asia, among them *Miscanthus tinctorius*, *Miscanthus sinensis* and *Miscanthus sacchrisforus* are of primary interest for biomass production (Greef and Deuter, 1993). In order to expand the genetic base of *Miscanthus* maximize the productivity and adaptive range of the crop, the genotype *Miscanthus giganteus* has attracted attention and now it is widely used in Europe. Harvestable *Miscanthus* yields (dry matter) have been estimated to be in the range of 12 to 40 t/ha, but yields of 27 to 40 t/ha have been reported (Lewandowski *et al*., 2000).

Fig. 1 – *Miscanthus Giganteus*.

Miscanthus as a key biomass energy crop with relatively low maintenance and high yield/energy content, has an important role in the sustainable production of renewable fuels and chemicals via thermo-chemical conversion. It is currently used in the European Union as a commercial energy crop, as a source of heat and electricity, or converted into biofuel products such as ethanol. Chemical composition of *Miscanthus x giganteus* (average values) is: cellulose 51.0%; hemicelluloses 30%, lignin 20.5%, ash, 3.0% (Hodgson *et al*., 2011).

There are many possibilities to valorize *Miscanthus* crops: combustion, gasification and pyrolysis for energy, liquefaction and hydrolysis for chemicals, delignification for cellulosic fibers (Table 1).

Among other raw materials, *Miscanthus* offers one of the highest productions of biomass and, consequently, of cellulosic fibers, as is presented in Table 2. This finding represents a good motivation for using *Miscanthus* as a raw material for paper industry.

Table 1
Processes to Valorize Biomass Feedstock (Like Mischantus)

Process	Parameters/reagents	Products	Literature
Combustion	> 1000ºC, oxygen	Heat	Brodeur *et al.*, 2011
Gasification	700-1000ºC, deficit of oxygen	Liquid and gaseous biofuels	Brodeur *et al.*, 2011; Larson *et al.*, 2009
Pyrolysis	200-1000ºC, no oxygen	Char, liquid and gaseous biofuels	Scurlock, 1998; Hodgson *et al.*, 2011
Liquefaction	200-400ºC, hydrogen 20-200 bars	Bio-oils, biofuels, chemicals	Scurlock, 1998; Hodgson *et al.*, 2011
Hydrolysis	Acids, enzymes	Sugars and lignin	Taherdazeh and Karimi, 2007; Yang, 2009; Sun and Cheng, 2002
Delignification	140-180ºC, NaOH, formic acid, methanol	Cellulosic fibers, lignin, chemicals	Brosse *et al.*, 2009; Ligero *et al.*, 2010; Villaverde *et al.*, 2010

Table 2
Yield of Cellulosic Fibers Resulted from Different Raw Materials

Raw material	Production [t/ha·year]	Cellulosic fibers [t/ha·year]
Wheat straw	4	1.9
Rice straw	3	1.2
Reed	20	8
Hemp	15	6.7
Miscanthus	20	8
Sugar cane	9	4.2
Kenaf	15	6.5
Softwood	3.5	1.8
Hardwood	12	6

Papermaking pulps are classified according to their average fiber length as is presented in Table 3. So-called long-fiber pulps are most appreciated by the paper manufacturer due to the fact that the resulting paper shows high strength properties. Short-fiber pulps are mainly used to obtain paper grades for printing. As Table 3 shows, *Miscanthus* pulp is included in the short-fiber pulp category, along with pulps made from wheat straw, rice, reed.

The goal of this paper is to obtain and characterize cellulosic fibers (chemical pulp) from *Miscanthus* by a delignification process (soda cooking) and to establish the opportunity of using the pulp as a raw material for paper industry.

Table 3
Papermaking Pulps Classification According to their Fiber Length (Adapted from Sixta, 2006 and Ververis et al., 2004)

Papermaking pulp category	Raw material	Fibers length [mm]
Long-fiber pulps	– wood (spruce, pine) – non-wood plants (kenaf, flax, hemp)	2.80 – 4.0 2.30 – 33.0
Short-fiber pulps	– wood (eucalyptus, poplar) – non-wood plants (wheat straw, rice, reed, *miscanthus*)	1.10 – 1.70 1.0 – 1.4

2. Experimental

2.1. Raw Materials

Stems of *Mischantus x Gigantheus* was obtained from a farm in Moldova region in eastern Romania. After harvesting, the vegetal material (without leaves) was dried in laboratory to a moisture content of about 10% and then chipped at 2-4 cm length.

2.2. Methods

The pulping trials (soda pulping) were performed using a 10 L stainless steel electric heated rotary laboratory digester. Cooking liquor was prepared in laboratory by dissolving sodium hydroxide (NaOH) in tap water. The ratio of solids to liquor was 1:5. The heating time to cooking temperature was 30 min in all cooking experiments. The pulping factors were alkali charge (12-18% NaOH), temperature (140-170ºC) and cooking time (30-60 min).

After pulping and digester degassing, the brown stock was defibered and washed with water until no color in the resulting liquor was observed. Washed pulp was screened using a vibratory screen with 0.25 mm slots, Fig. 2. The screened yield of pulps was determined and the screened pulps were analyzed for their lignin content expressed as Kappa number (TAPPI T 236 om-06, 2006). Furthermore, the pulps were subjected to beating in the Jokro mill (ISO 5264-3, 1979) and transformed in sheets in accordance with the Rapid Köthen method (ISO 5269-3, 1998). The following strength properties were determined: tensile strength (ISO 1924:2008); burst strength (ISO 2758:2001); crush resistance of flutes (also called CMT - ISO 7263:2011) and short span compression resistance (SCT - ISO 9895:2008).

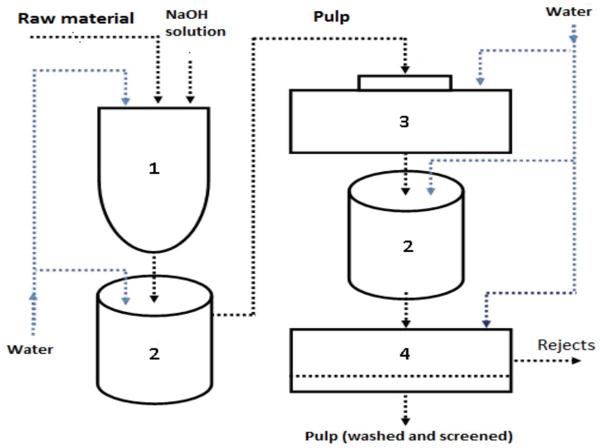

Fig. 2 – Laboratory equipment used for *Mischantus* delignification:
1-digester; 2-pulp pits; 3-pulp disintegrator; 4-vibratory screen.

3. Results and Discussions

Experimental conditions and results are presented in Table 4. The cooking parameters were selected to obtain a wide range of pulps regarding yield and lignin content. The screened pulps were analyzed according to three groups of properties: pulp yield and pulp lignin content (expressed as kappa number); strength properties: tensile and burst indexes; compressive properties: crush resistance of flutes (CMT index) and short span compression resistance (SCT index). All pulps were beaten in the Jokro mill at a refining degree of 20ºSR.

Common properties are represented by pulp yield and pulp lignin content. This study aims to obtain pulps having a large domain of residual lignin content, kappa number of pulp being in the range of 17-47 units. The pulp yield ranged from 44 to 58%. The pulp yield correlates with lignin content as is presented in Fig. 3. A linear correlation was found which is described by the ecuation $y = 0.468x + 37.422$. The value of coefficient of determination $R^2 = 0.8726$ shows that 87% of the variance in the dependent variable (pulp yield) is predictable from the independent variable (pulp lignin content).

Table 4
Experimental Conditions and Results

Exp. no	Cooking conditions (NaOH addition/temperature /time at temperature)	Kappa number of pulp	Pulp yield [%]	Tensile index [N·m/g]	Burst index [kPa·m^2/g]	CMT index [N·m^2/g]	SCT index [N·m/g]
1	12 % / 140°C / 30 min	37.0	52.3	60.9	3.72	2.52	31.8
2	15 % / 140°C / 30 min	29.6	51.1	71.1	3.94	2.72	33.6
3	18 % / 140°C / 30 min	31.0	51.2	65.3	3.92	2.61	31.8
4	12 % / 155°C / 30 min	39.6	50.9	66.4	3.78	2.82	35.0
5	15 % / 155°C / 30 min	28.9	51.5	65.1	3.44	2.40	29.6
6	18 % / 155°C / 30 min	26.7	51.9	71.0	3.21	2.97	33.0
7	12 % / 170°C / 30 min	47.4	58.5	40.9	2.12	2.22	28.8
8	15 % / 170°C / 30 min	30.8	50.0	50.4	2.48	2.36	28.7
9	18 % / 170°C / 30 min	22.4	47.6	41.7	2.33	2.11	26.8
10	12 % / 140°C / 60 min	40.5	56.5	51.9	2.93	2.53	27.8
11	15 % / 140°C / 60 min	35.2	55.7	55.9	3.13	2.91	29.2
12	18 % / 140°C / 60 min	32.3	55.0	60.1	3.26	2.52	27.8
13	12 % / 155°C / 60 min	42.3	58.0	51.2	3.14	2.60	27.7
14	15 % / 155°C / 60 min	30.5	50.5	56.8	2.95	2.68	27.4
15	18 % / 155°C / 60 min	27.2	49.0	60.7	2.82	2.55	27.7
16	12 % / 170°C / 60 min	43.9	56.3	53.2	3.28	2.38	30.6
17	15 % / 170°C / 60 min	28.8	53.5	51.8	2.74	2.52	31.6
18	18 % / 170°C / 60 min	17.5	44.3	53.0	2.80	2.55	32.1
19	15 % / 155°C / 60 min	29.2	51.1	61.4	3.35	2.76	31.8
20	15 % / 155°C / 60 min	29.5	52.0	56.7	3.40	2.79	31.6

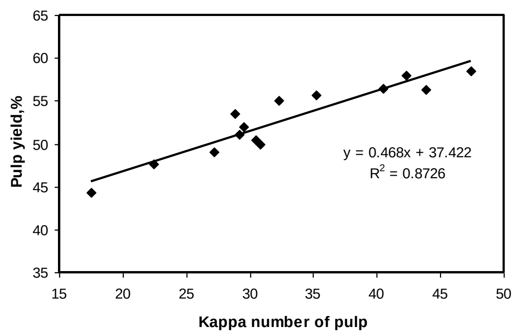

Fig. 3 – Correlation between pulp yield (y) and pulp lignin content (x- kappa number of pulp).

The most important parameter of a fibrous material (chemical pulp) is its lignin content, as the lignin content determines the pulp yield and pulp strength properties as well. *Miscanthus* soda pulp (unbleached pulp) can be obtained both with low lignin content (kappa number 17 – 30 units) or with high lignin content (kappa number up to 47 units). At higher lignin content, the pulp cannot be separated into individual fibers by defibering.

The study showed that lignin content of *Miscanthus* pulp can be fixed by the selection of the cooking parameters. The pulp lignin content is influenced by the NaOH addition, cooking temperature and cooking duration, but in different manner.

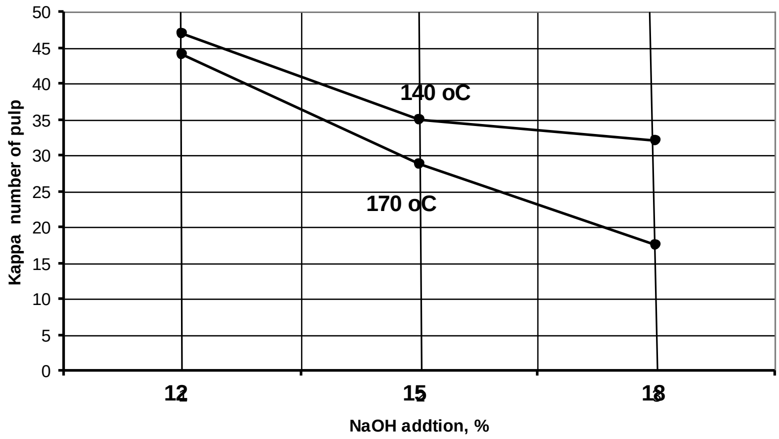

Fig. 4 – Influence of NaOH addition and cooking temperature on pulp lignin content (60 min time at cooking temperature).

Fig. 4 shows the influence of NaOH addition and cooking temperature on pulp lignin content. Time at cooking temperature was kept constant at 60 min. It is obvious if the NaOH addition increases, the lignin content of pulp decreases (regardless of the temperature value) as a result of extended delignification. If the cooking temperature is low (140ºC), pulp with high lignin content is obtained, even at high NaOH addition (18%). At higher temperatures (170ºC), the lignin content significantly reduces as a result of more delignification. The results conclude that the pulp lignin content can be fixed by the choice of the values of NaOH addition and cooking temperature.

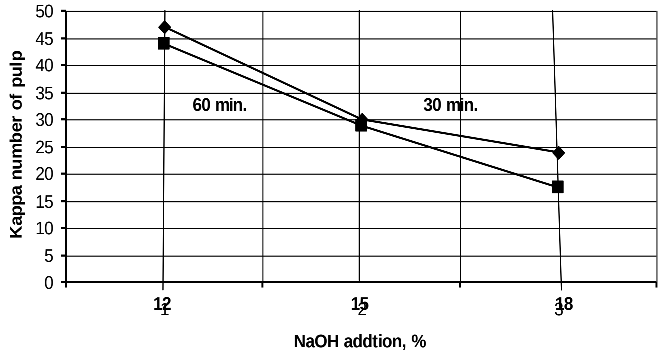

Fig. 5 – Influence of NaOH addition and cooking time on pulp lignin content (cooking temperature 170°C).

The influence of NaOH addition and cooking time on pulp lignin content is presented in Fig. 5. At constant temperature (170ºC), an increase of cooking time determines a relatively low reduction of pulp lignin content, irrespective of the value of the alkali addition. Figs. 4 and 5 show that the most important parameters influencing pulp lignin content are NaOH addition and cooking temperature.

Strength properties of *Miscanthus* pulp were investigated by testing of tensile and burst indexes. Fig. 6 shows the influence of pulp lignin content of tensile index of Miscanthus pulp and it is obvious that lignin content influence the tensile index of pulp. The tensile index shows better values for the pulp having lignin content expressed as kappa number around 30 units. By an increase of lignin content, tensile index reduces especially if pulp kappa number exceeds 40 units. The same conclusion can be drawn from the Fig. 7 regarding the influence of lignin content on pulp burst index. Burst index of *Miscanthus* pulp shows better values at pulp kappa number of 30-40 units, after which burst index decreases.

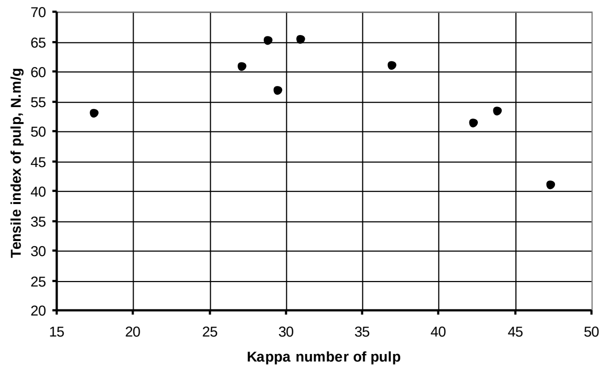

Fig. 6 – Influence of pulp lignin content of tensile index of *Miscanthus* pulp.

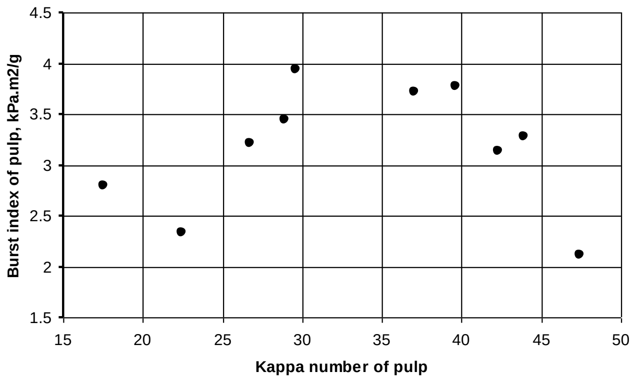

Fig. 7 – Influence of pulp lignin content of burst index of *Miscanthus* pulp.

Compressive properties of pulp are crush resistance of flutes (CMT index) and short span compression resistance (SCT index). The compressive properties of common papers are not tested in the usual way. These properties are tested for the papers for corrugated board. The corrugated board and package resistances strongly depend on the compressive properties of component papers. In this respect, corrugated board manufacturers use papers having values of compressive properties over certain limits.

Fig. 8 shows the influence of pulp lignin content on CMT index of *Miscanthus* pulp. It is clearly that CMT index attain its highest values for the pulps having lignin content in the range of 30-40 units. An important conclusion drawn from the Figs. 6, 7 and 8 is lignin content of pulp influences in the same manner both strength properties (tensile and burst indexes) and CMT index. These properties reach their maximum values at a pulp kappa number of 30-40 units.

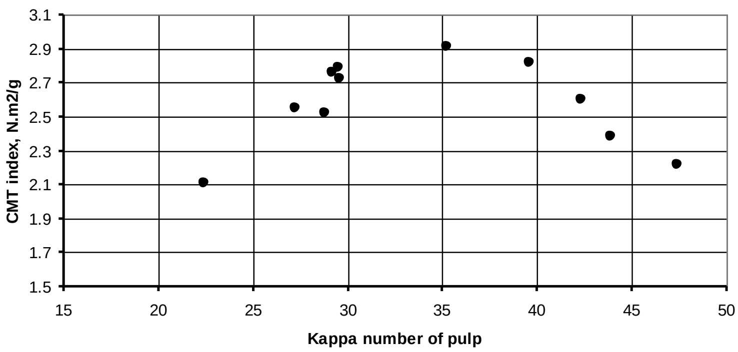

Fig. 8 – Influence of pulp lignin content of CMT index of *Miscanthus* pulp.

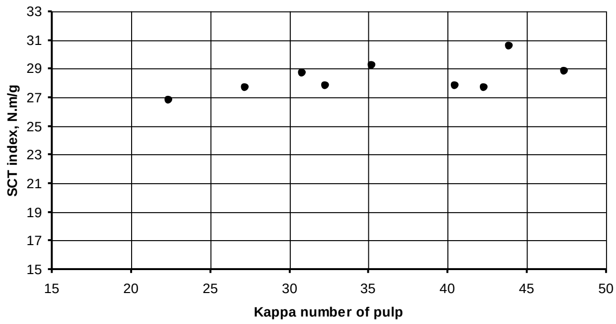

Fig. 9 – Influence of pulp lignin content of SCT index of *Miscanthus* pulp.

The evolution of SCT index with lignin content of pulp is completely different. Fig. 9 shows SCT index slowly enhances as pulp lignin content increases and no maximum value is recorded. Therefore, SCT index evolution substantially differs from the evolution of tensile, burst and CMT indexes. The explanation refers to the fact that higher lignin content enhances the fibers rigidity and as a result, the capacity of the fibrous structure to retrieve the forces parallel acting to surface of the sheet increases.

The present study showed that *Miscanthus* represents a valuable source of cellulosic fibers for paper industry, being one of the most promising biomass crops in Romania. *Miscanthus* is distinguished from other biomass crops by its high yields, particularly at cool temperatures, which can be more than double those typical of switchgrass. Chemical composition of *Miscanthus* is encouraging regarding its cellulose and lignin content, making them suitable for delignification. *Miscanthus* pulp can be obtained by usual delignification processes, among them soda cooking is very attractive. Pulps having different yields and lignin content can be obtained by suitable choosing of the cooking parameters: NaOH addition, time and temperature. The strength properties of pulp mainly depend on its lignin content. Lignin-rich pulps show high compressive strength, being suitable for obtaining of papers for corrugated board.

4. Conclusions

Miscanthus is an important biomass crop having an increasingly role in the sustainable production of renewable fuels and chemicals via thermo-chemical conversion. *Miscanthus* crop can be valorizing through: combustion, gasification and pyrolysis for energy, liquefaction and hydrolysis for chemicals and delignification for cellulosic fibers. *Miscanthus* can become a serious candidate as a raw material for obtaining of cellulosic fibers for paper industry. *Miscanthus* yields (dry matter) have been estimated at 12-40 t/ha·year.

Delignification of *Miscanthus* allows to obtain short-fiber pulps similar to other non-wood plants: wheat and rice straws and reed.

Delignification of *Mischantus* by soda (NaOH) process, allows to obtain pulps having a large domain of residual lignin content, kappa number of pulp being in the range of 17-47 units. Depending on the lignin content, the pulp yield ranged from 44% to 58%. The pulp lignin content can be fixed by the choice of alkali addition and cooking temperature.

Lignin content of pulp influences in the same manner both strength properties (tensile and burst indexes) and CMT index. These properties reach their maximum values at a pulp kappa number of 30-40 units. The evolution of SCT index with lignin content of pulp is completely different. SCT index enhances as pulp lignin content increases and no maximum value is recorded.

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FIBRE CELULOZICE DIN *MISCANTHUS*

(Rezumat)

Miscanthus este o cultură importantă și are un rol esențial pentru producerea durabilă a combustibililor și chimicalelor din resurse regenerabile. El este folosit în prezent ca sursă de căldură și electricitate sau este transformat în biocombustibili. Există multe posibilități de valorificare a culturilor de *Miscanthus*: prin ardere, gazeificare și piroliză pentru obținere de energie; prin lichefiere și hidroliză pentru obținere de chimicale; prin delignificare pentru obținerea fibrelor celulozice. Compoziția chimică a *Miscanthus* este încurajatoare în privința conținutului de celuloză și de lignină, făcându-l potrivit pentru delignificare. Celuloza din *Miscanthus* se poate obține prin procedee obișnuite de dezincrustare, dintre care procedeul natron este foarte atractiv. Se pot obține celuloze cu randamente și conținuturi de lignină diferite prin alegerea adecvată a parametrilor dezincrustării: adaosul de NaOH, durata și temperatura. Proprietățile de rezistență ale celulozei depind în principal de conținutul de lignină. Celulozele cu multă lignină au rezistență la compresiune ridicată, fiind potrivite pentru obținerea hârtiilor pentru cartonul ondulat.