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# Studies on Isolation of Cellulose Fibres from Waste Plant Biomass

**Abstract**
*This paper presents the results of cellulose fibre extraction from plant biomass including: rape, hemp and flax straws. The selected materials were treated by thermal/mechanical/chemical method in order to remove non-cellulosic components such as lignin, hemicellulose, and pectin as well as to obtain cellulosic material with suitable structure and properties necessary for the manufacture of nanofibres. It has been shown that the use of multistage chemical treatment using oxygen and peroxide compounds as delignifing agents for hemp and flax straws, allowed the safe removal of lignin and other non-cellulosic components without degradation of the cellulose fibres obtained, while maintaining a polymerization degree above 1000 units. It was found out that it is possible to obtain cellulose fibres from hemp and flax straws (type retted flax straw-fibre variety, flax straw-oil variety), which can be processed into cellulose micro-and nanofibres.*

**Key words:** *waste plant biomass, cellulose fibres, cleaning, delignification.*

## Introduction

In recent years there has been a growing interest of various industries in renewable plant materials. The most commonly proposed method of biomass waste management is just its incineration as a renewable energy source. Yet, the biomass is also a valuable source of cellulose and, more precisely, cellulose fibres. At present, over half of the 70 million tons of different fibres manufactured every year is derived from oil based materials [1]. Therefore, many efforts are made to replace at least a portion of synthetic fibres with cellulose fibres obtained from plant waste materials. Consumer awareness is still increasing, and more and more people want to buy environmentally friendly products. Experts estimate that the commodities partially made from renewable resources will contribute to 10 percent of the total U.S. production in 2020 and up to 50 percent in 2050 [2]. The increased use of agricultural fibres is one of the ways to give consumers a wider choice of environmentally friendly products. Growing awareness of global deforestation also increases the demand for alternative raw materials for the manufacture of wood pulp and paper.

So far, various plants are used for the production of cellulose pulp, such as cotton, hemp, agave, flax, kapok, sisal, ramie and hemp. Current studies indicate the possibility to obtain natural cellulose fibres from wheat straw [3], rice straw [4], corn stalks and husk [5, 6] leaves and stalks of sorghum [7] pineapple leaves [8], banana leaves [9], sugar cane [10] and even nettle [11]. The obtained natural cellulose fibres may be used for textile, pulp and paper, as a component of composites and other industrial applications. Annual world production of major cereals such as rice, wheat, soybeans, and corn amounts to 696, 653, 265 and 840 million tonnes, respectively (FAO, 2010) [12]. Even assuming that only about 10% of all products which are suitable for the production of fibres, and the yield of the process of about 20%, possibly up to 50 million tons per year of technical cellulose fibres can be obtained from only four of the above cereals. The use of biomass waste would also increase the value of crops [13].

The cellulose fibres obtained from waste materials can be used in the preparation of micro-and nano- cellulose fibres, micro-and nanoparticles and nanowhiskers. The first reports on the isolation of cellulose microfibrils date back to 1983 [14, 15] with the starting material being wood cellulose (softwood). Current research relates, to a large extent, to non-wood raw materials, including: the preparation of nanofibres from wheat straw and soybean hulls [16], the impact of delignification on isolation of bamboo nanofibre [17], the use of steam explosion to obtain banana nanofibres [18] and the extraction of nanowhiskers from bagasse [19].

From the economic point of view it is preferable to produce fibres from locally available biomass waste in order to avoid high transport costs of the raw materials. Therefore, studies conducted in Poland should focus on the use of wheat, rye, barley, rape, flax and hemp straws. The straw harvest in Poland exceeds demand arising from animal production (fodder, bedding). The average cereal straw production amounts to 29.3 million tonnes per year, with an average surplus of 11.5 million tonnes [20]. All of the rape

## List of acronyms

$T_P$ – *sodium-peroxide method*
$T_A$ – *sodium hydroxide method*
*AO* – *active oxygen*
*RS* – *rape straw*
*WS* – *wheat straw*
*CS* – *corn straw*
*Rys* – *rye straw*
*HS* – *hemp straw*
*FSR* – *retted flax straw-fibre variety*
*FOS* – *flax straw-oil variety*
*CL* – *carrot leaves*
*SS* – *sunflower straw*
*BS* – *bean straw*
*O* – *oxygen delignification*
*D* – *sodium chlorite delignification*
*Pa* – *peracetic acid delignification*

straw can be processed as well as hay and the residues from the production of vegetables.

**The aim of this study** was to determine the chemical composition of biomass plant materials in terms of their suitability to extract cellulose fibres. An attempt was made to develop methods of thermal/mechanical/chemical processing, namely: fragmentation, swelling and digesting, in order to separate the biomass components like lignin, hemicellulose, and pectin as well as to prepare a purified material for the production of cellulosic micro- and nanofibre structures.

## Materials and methods

Different types of biomass material such as: rape straw, wheat straw, rye straw, corn straw, bean straw, sunflower straw, hemp straw, retted flax straw-fibre variety, flax straw-oil variety and carrot leaves were used in the study. The hemp straw and flax straw are the raw material after separation from bast fibres of hemp and flax plants. These materials were obtained from the Agricultural University in Cracow under the Innovative Economy Operational Programme Project No. 01.01.02.-10-123/09 BIOMASS.

**Purification of the non-cellulosic components of biomass**

***Pre-steaming of the biomass material*** - the thermal treatment was performed in a revolving digester with a capacity of 15 $dm^3$. The sample (from 0.5 to 1 kg) of biomass was placed in a digester and steam-treated at 0.2 MPa. for 10 minutes.

***Refining of the biomass material*** – presteamed pulp was placed in a container with hot water and subjected to refining in a Sprout-Waldron mill at a gap width of 0.5, 0.2 and 0.1 mm.

**Chemical treatment of the biomass material:**

- sodium hydroxide/hydrogen peroxide digestion method ($T_P$) was performed in a revolving digester with a capacity of 15 $dm^3$. The pulp sample after pre-steaming (from 0.5 to 0.7 kg as bone dry weight) was placed in a container and a cooking liquor was added at about 60 °C containing: 5% NaOH, 5.5% $H_2O_2$, 0.3% EDTA, 0.5% $MgSO_4$ and 5% liquid glass, per sample bone dry weight. The pulp was stirred for about 10 minutes. The liquor ratio was 4:1 (amount of liquor ratio to biomass material). Next the pulp was placed in the digester and cooked at 110 °C for 60 minutes.
- sodium hydroxide digestion method ($T_A$) was performed in a revolving digester with a capacity of 15 $dm^3$. The pulp sample after pre-steaming (from 0.5 to 0.7 kg as bone dry weight) was placed in a container and a cooking liquor was added at about 60 °C containing 7% NaOH, per sample bone dry weight. The pulp was stirred for about 10 minutes. The liquor ratio was 4:1 (amount of liquor ratio to biomass material). Next the pulp was placed in the digester and cooked at 110 °C for 60 minutes.

***Oxygen delignification of the biomass material (O)*** - after pre-steaming, digesting and centrifugation, the pulp sample (from 0.5 to 0.7 kg as bone dry weight) was added to distilled water at about 60 °C (8% pulp consistency); next 2.5% NaOH and 0.5% $MgSO_4$ (per sample bone dry weight) was added, mixed and placed in a revolving digester. Oxygen was added to obtain the initial pressure of 0.28 MPa. The pulp was digested at 120 °C for 60 minutes.

**Sodium chlorite delignification of the biomass material (D)** - after pre-steaming, digesting and oxygen delignification the pulp sample was centrifuged; next the pulp sample (from 0.2 to 0.5 kg, as bone dry weight) was added to distilled water at about 60 °C, and 14 g/$dm^3$ $NaClO_2$ and 3 g/$dm^3$ $H_2SO_4$ was added, and the pulp was placed in a laboratory thermostat at a temperature of 70 °C for 120 min. After sodium chlorite delignification an alkaline extraction step followed. Centrifuged and weighed pulp was added to distilled water at about 60 °C, and 1% NaOH, per sample bone dry weight, was added; next pulp was placed in a laboratory thermostat at a temperature of 70 °C for 120 min.

***Peracetic acid delignification of the biomass material (Pa)*** - after pre-steaming, digesting and oxygen and sodium chlorite delignification the pulp sample (from 0.2 to 0.5 kg, as bone dry weight) was added to distilled water at about 60 °C; next 2% $CH_3COOOH$ based on active oxygen (AO) and 0.5% $MgSO_4$ (per sample bone dry weight) was added and placed in a laboratory thermostat at a temperature of 80 °C for 120 min.

After each step of chemical treatment the pulp sample was subjected to determining pH, residual $H_2O_2$, $ClO_2$ and AO (according to the method of chemical treatment). The sample was washed until neutral pH and weighed.

The test methods described above are based on descriptions in the cited literature [21 - 23].

**Evaluation of biomass samples before and after chemical treatment**

The properties of initial biomass and biomass after chemical treatment were evaluated according to the following standards:

- The contents of cellulose, holocellulose, lignin, substances soluble in ethanol and mineral substances were determined according to Polish Standard PN-92/P-50092 - Raw materials for the paper industry. Wood. Chemical analysis.
- Alpha-cellulose content was determined according to Polish Standard PN-62/P-50099 -Paper Industry Products - Technical Tests - Determination of Alpha-cellulose in Special Pulps.
- The limiting viscosity number and the degree of polymerisation DP were determined in accordance with ISO 5351:2010 Pulps - Determination of limiting viscosity number in cuprietylenediamine (CED) solution.
- Pectin content was determined according to the IFU MA26:1996 method - Determination of Pectin.

## Results and discussion

**Study of the chemical composition of different types of plant biomass**

The analysis of chemical composition of plant biomass was performed in order to determine its usefulness for the isolation of cellulose fibres intended for conversion into cellulose nanofibres (***Table 1***).

Among all studied materials, hemp straw, and flax straw were characterized by a favourable qualitative and quantitative composition. The alpha-cellulose content was 60.09% for hemp straw and 51.56% for retted flax straw-fibre variety. The lignin content in these two materials was 12.36 and 18.16%, respectively (***Table 1***).

The organic substances content in hemp straw was 3.88% and in the retted flax straw-fibre variety it was 3.58%, which

***Table 1.*** *The chemical composition of different types of biomass.*

| Starting raw material | Holocellulose, % | Alfa-cellulose, % | Lignin content, % | Soluble substances in ethanol, % | Mineral substances, % | Pectin, % |
|---|---|---|---|---|---|---|
| **Rape straw** (RS) | 73.06 | 40.85 | 19.00 | 3.28 | 4.49 | 6.32 |
| **Wheat straw** (WS) | 79.12 | 40.80 | 22.45 | 4.15 | 5.65 | 0.53 |
| **Corn straw** (CS) | 70.88 | 38.83 | 19.95 | 5.70 | 5.84 | 1.42 |
| **Rye straw** (RyS) | 78.14 | 45.07 | 21.65 | 4.79 | 4.94 | 0.83 |
| **Hemp** straw (HS) | 83.92 | 60.09 | 12.36 | 3.88 | 3.16 | 0.50 |
| **Retted flax straw-fibre variety** (FSR) | 77.61 | 51.56 | 18.16 | 3.58 | 2.55 | 2.56 |
| **Flax straw-oil variety** (FOS) | 70.12 | 41.86 | 22.90 | 7.00 | 4.50 | 4.47 |
| **Carrot leaves** (CL) | 52.89 | 31.58 | 18.51 | 10.34 | 15.21 | 1.88 |
| **Sunflower straw** (SS) | 71.85 | 40.41 | 19.45 | 8.80 | 7.31 | 4.75 |
| **Bean** straw (BS) | 59.52 | 40.20 | 18.13 | 7.93 | 10.57 | 11.02 |

was lower in comparison with the other raw materials by 2 to 6 percentage units, on average (***Table 1***).

The chemical composition of hemp straw and retted flax straw-fibre variety was similar to wood since the average cellulose content in wood is 40 - 44%, lignin in softwood is 28% and in hardwood 21%, organic matter is 2 - 5%, and minerals matter is 0.5% [24]. High cellulose content and low content of lignin as well as organic and inorganic substances indicate the suitability of biomass for the production of cellulose fibres.

The other raw materials studied had worse properties in comparison with hemp and flax straws. Significant amount of organic substances soluble in ethyl alcohol, exceeding 7% in the case of, for example, bean straw, sunflower stems and carrot leaves, hinders thorough purification of biomass using sodium hydroxide or sodium hydroxide/hydrogen peroxide methods. The presence of these substances inhibits the delignification process as a result of their condensation reactions with lignin, which make lignin difficult to remove from plant biomass.

The analysed straws also contained significant amounts of mineral substances (mainly silica), at least six times higher than wood, which is typical for all annual plants. Such raw material is less susceptible to loosening and swelling of the fibre structure.

### Study on purification and isolation of cellulose fibres from biomass samples

Purification of plant biomass from non-cellulosic components was performed using two methods: with sodium hydroxide/hydrogen peroxide ($T_P$) and sodium hydroxide ($T_A$). Initially, plant biomass was subjected to steaming and grinding. Mechanical treatment and the steaming process modifies the structure of fibres, by an internal fibrillation (loosening of fibre structure by breaking the hydrogen bonds between the hydroxyl groups of cellulose chains) and also by external fibrillation, i.e. loosening the bonds between fibrils located on the fibre surface. Through the grinding process, cellulose fibres of a uniform structure can be obtained. Using chemical treatment of plant materials we attempted to compare the purifying and delignification effects with selected methods like sodium hydroxide/hydrogen peroxide and sodium hydroxide methods.

The aim of chemical treatment was to obtain pulp containing only the so-called residual lignin, while maintaining the alpha-cellulose content above 60%. For chemically treated hemp straw (HS), retted flax straw-fibre variety (FSR) and

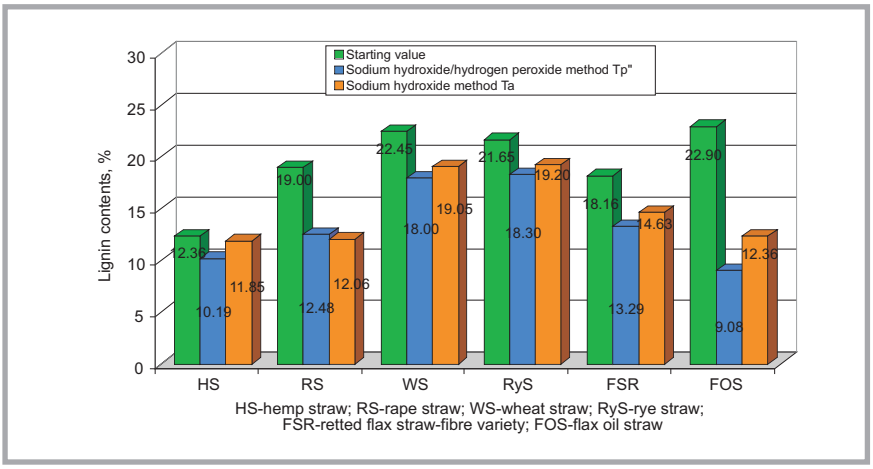


***Figure 1.*** *Evaluation of lignin content after chemical treatment by sodium hydroxide/hydrogen peroxide ($T_P$) and sodium hydroxide ($T_A$) methods.*

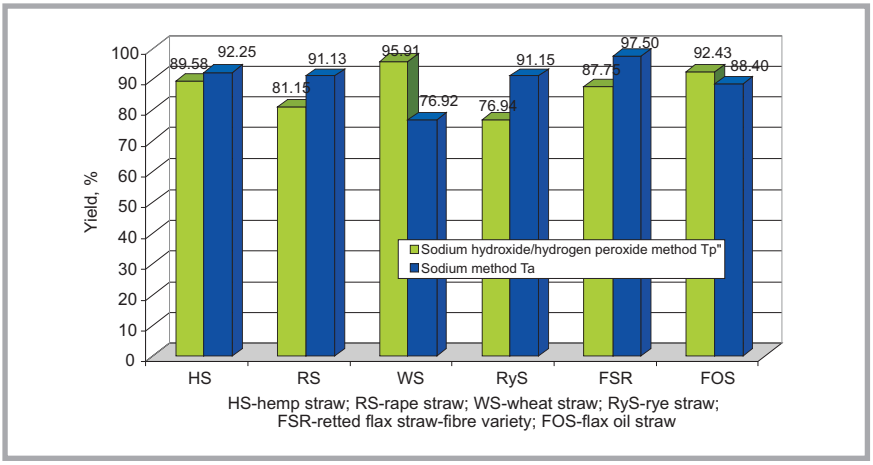


***Figure 2.*** *Yield of purification process after chemical treatment by sodium hydroxide/hydrogen peroxide ($T_P$) and sodium hydroxide($T_A$) methods.*

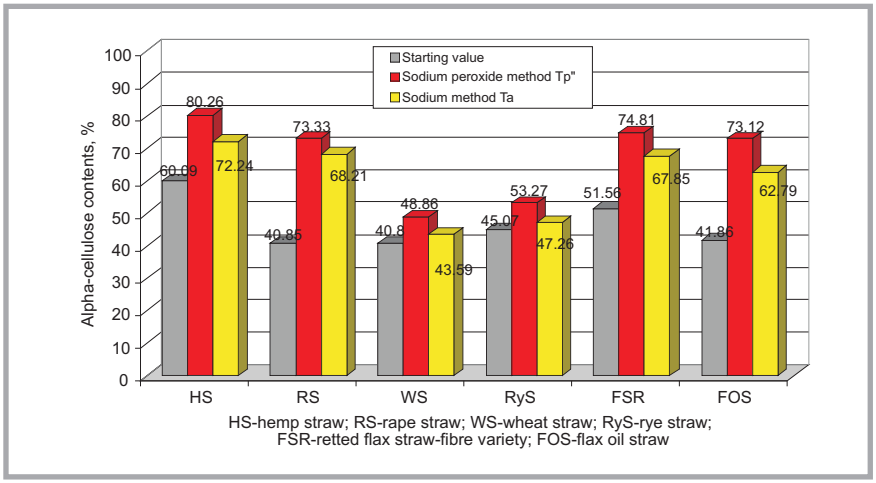


***Figure 3.*** *Evaluation of alpha-cellulose content after chemical treatment by sodium hydroxide/hydrogen peroxide ($T_P$) and sodium hydroxide ($T_A$) methods.*

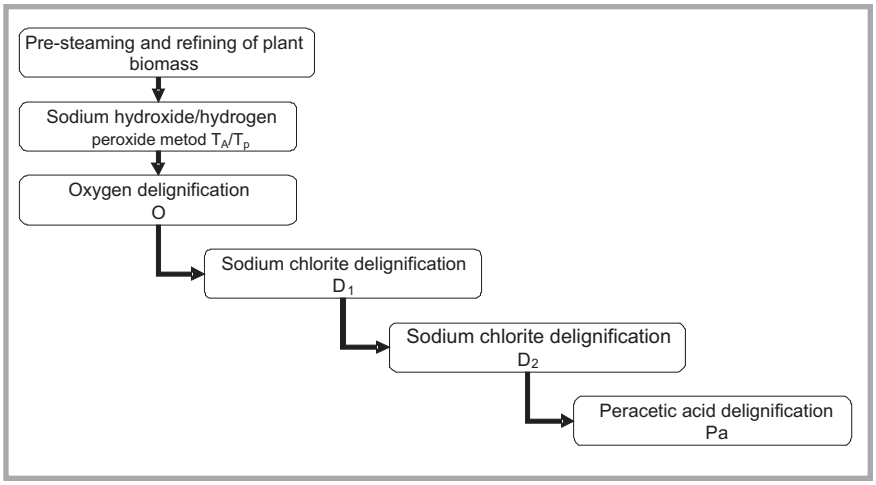


***Figure 4.*** *Scheme of multi-stage treatment of plant biomass.*

flax straw-oil variety (FOS), the alpha-cellulose and lignin contents obtained and yield show that the $T_P$ method was a more effective treatment method of biomass than the $T_A$ method. In the case of the sodium hydroxide/hydrogen peroxide method a decrease in lignin content was observed: from 12.36% to 10.19% for HS sample, from 22.9% to 9.08% for FOS sample and from 18.16% to 13.29% for FSR sample, without much loss of yield (compared with the initial biomass) (***Figure 1, 2*** see page 169). The pulps obtained from flax, hemp and rape straws were also characterized by high alpha-cellulose content, which increased above 70% (***Figure 3***). However, it should be noted that the purification degree depends mainly on the biomass type.

The pulps obtained from rye straw (RyS) and wheat straw (WS) were only apparently delignified, since the lignin content in the pulp decreased only 3 percentage points, without major changes in the alpha-cellulose content. The amount of alpha-cellulose in WS and RyS samples did not exceed the level of 50% and increased about 6 percentage units on average compared to the initial value (***Figure 1***). The low level of alpha-cellulose content indicates that the cellulose structure might be damaged. Additionally, it was observed that those pulps contained significant amounts of non-digested straw material.

The $T_P$ method treatment was more effective compared to the $T_A$ method because in the $T_P$ method a higher degree of lignin removal, and an increase in alpha-cellulose content was obtained. In the case of biomass treated using the sodium hydroxide method a tendency of fibre fibrillation was not observed, probably because the process of removing lignin and other non-cellulosic components was not very effective. In the pulps obtained from hemp straw or flax straw only a slow fibre fibrillation process was observed. Incomplete removal of lignin from the biomass implies the necessity for further purification stages, which should be carried out in such a way that the cellulose structure is not damaged.

### Study of the multi-stage chemical treatment

The use of different types of chemical treatment of biomass has shown that it is possible to partially remove the non-cellulosic components, such as lignin, pectin, organic and inorganic substances. Complete purification of plant biomass is possible by using the so-called "extended" multi-stage delignification. It is possible to remove lignin and other non-cellulosic components, through, for example, more extreme conditions of delignification; however, it may affect the cellulose structure and result in a decrease in the process yield. In order to prevent this, and to obtain "pure" cellulose fibres from plant biomass, a slow multi-stage treatment was applied, which protects cellulose fibre from excessive degradation.

The biomass types, which showed a tendency to the fibrillation of fibres, like hemp straw (HS), retted flax straw-fibre variety (FSR) and flax straw-oil variety (FOS) were used for the multi-stage delignification process (***Figure 4***). Taking advantage of the susceptibility of these materials to the action of oxidizing agents (hydrogen peroxide) under alkaline conditions, as the first step of treatment, the sodium hydroxide/hydrogen peroxide method $T_P$ was used. The second step was delignification with oxygen (O). Oxygen is an effective and selective delignification agent operating in a phenylpropane unit of lignin, which leads to the formation of organic and inorganic peroxy compounds. Lignin reacting with oxygen in alkaline medium is oxidized and degraded, and as a result, becomes soluble in alkaline solutions and is thus easily removable from the pulp. Sodium chlorite and peracetic acid have a similar effect on lignin, and therefore the next delignification steps were carried out with the use of two-step sodium chlorite treatment ($D_1$ and $D_2$) and peracetic acid (Pa) treatment.

As a result of multi-stage treatment, hemp straw (HS) and retted flax straw-fibre variety (FSR) pulps with a low amount of residual lignin, 0.45% and 0.86%, were obtained, while increasing alpha-cellulose content to 85.5% for the HS sample

and 79.78% for the FSR sample and final yield (compared with the starting material) above 50% (***Table 2***).

Hemp straw proved to be the most susceptible to multi-stage treatment with oxygen and peroxy delignification agents. The high polymerization degree, above 1200 units, (***Table 2***) is an indicator of the high quality of the pulp obtained, which also was characterized by the presence of long fibres with a tendency to form fibrils on their ends.

Multi-stage delignification also was applied to the retted flax straw-fibre variety (FSR). After chemical treatment with oxygen (O) and two-step treatment with sodium chlorite (D), the residue lignin was 2.72%, and the yield 62.87%. To improve the purification degree of pulp (to further remove lignin), an additional step - selective delignification with peracetic acid (Pa) - was introduced. As a result of this treatment, lignin content was reduced to 0.86% and the polymerization degree was 1106 units (***Table 2***). The cellulose obtained fibres showed a tendency for swelling with simultaneous loosening of their structure, and finally, for the progressive fibrillation.

Breaking lignin-carbohydrate binding proved to be more difficult for the flax straw-oil variety compared with the hemp straw or retted flax straw-fibre variety. After multi-stage purification of biomass, pulp with lignin content of 2.09% was obtained, while decreasing the yield to 52.15%. Multi-stage treatment can lead to degradation of the cellulose structure as alpha-cellulose content decreased to 65.22%, despite a relatively high polymerization degree of 1025 units (***Table 2***).

In the case of hemp and flax straws, the application of a multi-stage delignification process with oxygen and peroxide agents enabled effective purification from lignin and non-cellulose components without degradation of the cellulose fibres obtained, which showed a tendency for creating fibrils. This can significantly facilitate further processing of pulp into micro and nanofibres.

***Table 2.*** *The selected pulps after multi-stage chemical treatment.*

| Pulp sample | Delignification stages | Kappa number | Lignin content, % | Alpha-cellulose, % | DP | Total yield % |
|---|---|---|---|---|---|---|
| Hemp straw (HS) | $T_P$ | 67.9 | 10.19 | 80.15 | nt | 89.58 |
| | O | 65.3 | 9.80 | nt | nt | 81.11 |
| | $D_1$ | 18.7 | 2.81 | nt | nt | 71.63 |
| | $D_2$ | 3.0 | 0.45 | 85.50 | 1266 | 65.89 |
| Retted flax straw-fibre variety (FSR) | $T_P$ | 88.6 | 13.29 | 74.81 | nt | 87.75 |
| | O | 18.1 | 2.72 | nt | nt | 81.81 |
| | $D_1$ | 40.3 | 6.05 | nt | nt | 73.34 |
| | $D_2$ | 18.1 | 2.72 | nt | nt | 62.87 |
| | Pa | 5.7 | 0.86 | 79.78 | 1106 | 60.11 |
| Flax straw-oil variety (FOS) | $T_P$ | 60.5 | 9.08 | 70.25 | nt | 92.43 |
| | O | 54.6 | 8.19 | nt | nt | 81.92 |
| | $D_1$ | 47.2 | 7.08 | nt | nt | 75.20 |
| | $D_2$ | 16.0 | 2.40 | nt | nt | 64.02 |
| | Pa | 13.9 | 2.09 | 65.22 | 1025 | 52.15 |

## Summary and conclusions

1. The analyses of the chemical composition of plants biomass (in particular, lignin, mineral and organic content), have shown that the most suitable raw materials for obtaining cellulose fibres are: hemp straw, retted flax straw-fibre variety and flax straw-oil variety.

2. The application of the sodium hydroxide/hydrogen peroxide method $T_P$ (compared with the sodium hydroxide method $T_A$) facilitated the purification of plant materials from non-cellulosic components. The amount of lignin removed (method $T_P$) was 34%, on average, with an increase in alpha-cellulose above 70%. This was especially evident in the case of hemp and flax straws.

3. The use of multi-stage treatment of biomass plant with oxygen and peroxide delignification agents allowed to safely remove lignin and other non-cellulosic components, without degradation of the cellulose fibres obtained, as evidenced by a high polymerization degree exceeding 1000 units.

The study results justify the appropriate use of multi-stage chemical treatment which enables purification of selected types of biomass from non-cellulose substance and yields raw material suitable for the production of micro- and nano fibres.

## References


1. Huda S, Reddy N, Karst D, Xu W, Yang W, Yang Y. Non-traditional biofibers for a new textile industry. J. *Biobased Mater. Bioenergy* 2007; 1 (2): 177–190.
2. Fiber Futures is a Sustainable-Future; http://www.sustainable- future.org/ futurefibers/solutions.html
3. Reddy N, Yang Y. Preparation and characterization of long natural cellulose fibres from wheat straw. *J. Agric. Food Chem*. 2007; 55; 21: 8570-8575.
4. Chen X, Yu J, Zhang Z, Lu C. Study on structure and thermal stability properties of cellulose fibers from rice straw. *Carbohydrate Polymers* 2011; 85; 1: 245-250.
5. Reddy N, Yang Y. Structure and properties of high quality natural cellulose fibers from cornstalks; *Polymer* 2005; 46: 5494–5500.
6. Reddy N, Yang Y. Properties and potential applications of natural cellulose fibers from cornhusks; *Green Chem*. 2005; 7: 190–195.
7. Reddy N, Yang Y. Structure and properties of natural cellulose fibers obtained from sorghum leaves and stems; *J. Agric. Food Chem*. 2007; 55; 14: 5569–5574.
8. Sasa Sofyan Munawar Kenji Umemura Shuichi Kawai. Characterization of the morphological, physical, and mechanical properties of seven non-wood plant fiber bundles; *J. Wood Sci.* 2007; 53: 108–113.
9. Sinha MK. The use of banana-plant fibre as a substitute for jute. *J. Textile Inst.* 1974; 65; 27: 27–33.
10. Costa SM, Mazzola PG, Silva JCAR, Pahl R, Pessoa Jr. A, Costa SA. Use of sugar cane straw as a source of cellulose for textile fiber production. *Industrial Crops and Products* 2013; 42: 189–194.
11. Bodors E, Baley Ch. Study of the tensile properties of stinging nettle fibres. *Mat. Lett*. 2008; 62; 14: 2143-2145.
12. www.faostat.fao.org
13. Reddy N, Yang Y. Natural cellulose fibers from soybean straw. *Bioresource Technology* 2009; 100: 3593–3598.
14. Herrick FW, Casebier RL, Hamilton JK, Sandberg KR. Microfibrillated cellulose: Morphology and accessibility. *Journal of Applied Polymer Science, Applied Polymer Symposium* 1983; 37: 797–813.

15. Turbak AF, Snyder FW, Sandberg KR. Microfibrillated cellulose, a new cellulose product: Properties, uses, and commercial potential. *Journal of Applied Polymer Science, Applied Polymer Symposium* 1983; 37: 815–827.
16. Ayse Alemdar, Mohini Sain. Isolation and characterization of nanofibers from agricultural residues – Wheat straw and soy hulls. *Bioresource Technology* 2008; 99: 1664–1671.
17. Okahisa Y, Abe K, Nogi M, Nakagaito AN, Nakatani T, Yano H. Effects of delignification in the production of plant-based cellulose nanofibers for optically transparent nanocomposites. *Composites Science and Technology* 2011; 71: 1342–1347.
18. Deepa B, Eldho Abraham, Bibin Mathew Cherian, Bismarck A, Blaker JJ, Laly A. Pothan, Alcides Lopes Leao, Sivoney Ferreira de Souza, Kottaisamy M. Structure, morphology and thermal characteristics of banana nano fibers obtained by steam explosion. *Bioresource Technology* 2011; 102: 1988–1997.
19. Eliangela de Morais Teixeira, Thalita Jessika Bondancia, Kelcilene Bruna Ricardo Teodoro, Ana Carolina Correa, Jose Manoel Marconcini, Luiz Henrique Caparelli Mattoso. Sugarcane bagasse whiskers: Extraction and characterizations. *Industrial Crops and Products* 2011; 33: 63–66.
20. http://www.ecbartos.pl/biomasa.html
21. Stupińska H, Milczarek S, Madaj J, Palenik J. A producing method of bleached paper-pulp from stems of bast plants (in Polish). Pat appl. P. 198456, 2008.
22. Stupińaska H, Milczarek S, Madaj J, Palenik J, Wójcik M, Grządzielewski P. A producing method of a long-fibrous paper-pulp from fibres of bast plants (in Polish). Pat appl. P. 197602, 2008.
23. Kopania E. *Study of the ability of hydrogen peroxide and Rother organic and inorganic derivatives in non-chlorine bleaching of wood pulps*. PhD Thesis, Łódź, 2005.
24. Wandelt P. *Technologia celulozy i papieru Cz. 1. Technologia mas włóknistych*. WSiP Ed. II. Warszawa, 1996.

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