Article

Nitrate-alkaline pulp from non-wood plants

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Abstract: To investigate this suitability of Black Mustard (*Brassica Nigra* L.) and Camelina (*Camelina Sativa* L.) for pulp manufacturing the nitrate-alkaline method was used. The non-wood plants were characterized by chemical analysis, especially lignin, cellulose, ash, extractives and alpha-, beta-, gamma-cellulose. The pulp was cooked in 6% nitric acid and then underwent the extraction by 5% sodium-hydroxide and neutralized by 1% acetic acid. The cooked pulp was characterized by delignification degree – Kappa number. The laboratory sheets were made from this cooked pulp and they were characterized by tensile index, breaking length, smoothness and compared with commonly available papers.

Keywords: nitrate-alkaline pulp, Black Mustard, Camelina, tensile index, chemical analysis

1. Introduction

In addition to wood pulp the pulp and paper industry utilizes fibers and agricultural waste. The fibers of linen, hemp and cotton are being processed since the beginnings of paper making; These fibers are being utilized for special types of paper. Even the straw, sugar cane, cotton and other non-wood plants are utilized in cellulose and paper industry. In case of choosing the raw material, it is important to know the chemical, morphological composition and the structure of the plants. [1]

Wood is the main source for the paper industry nowadays. There is lack of the wood sources which suits growing inquiry in lot of countries. Besides wood pulp, this causes utilizing fibers and waste from agricultural industry for pulp and paper making. 70% of raw material in pulp and paper industry in China and India comes from non-wood plants.[2]

Hemp, sisal and linen belong among plants which are utilized in paper making. The other raw material which is being added to banknote paper production is the cotton. The field planting areas in the Czech Republic have significantly changed during past years according to planted crops. The largest planting areas are sown by cereals such as wheat, barley or rapeseed but there has been significant grow of the corn, poppy, mustard and camelina planting areas recently.

The non-wood plants have chemical and morphological characteristics different to the wood materials and that is the reason for other conditions for cooking and bleaching.[3]

The non-wood plants are being processed the chemical method. The amaranth, atriplex and Jerusalem artichoke[4] and rapeseed straw.[5,6] The peracetic acid chemical method was used for wheat, rapeseed, amaranth and lavatera.[7] The neutral sulfite semi-chemical pulp has been produced by rapeseed straw.[8,9]

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Other chemical method for pulp making can be the very nitrate-alkaline process where the cellulose-lignin bond is hydrolytically released during the cooking in the acid. The lignin is being nitrated and partly oxidizes into nitro-lignin which is soluble in alkalines.[10]

The aim of this paper was to cook the nitrate-alkaline pulp of the black mustard (*Brassica Nigra* L.) and camelina (Camelina Sativa L.) and to examine the mechanical properties of paper made from these pulps. This study is focused on examining the chemical pulping of agricultural crops and widens the results by the other potential raw materials for cellulose fibers.

2. Materials and Methods

The Black mustard (*Brassica Nigra* L.) was grown on fields in the highlands of Ore mountains whereas the camelina (*Camelina Sativa* L.) was harvested in the lowlands of Podyjí area. The raw materials consist of stems and fruits which made up to quarter of whole quantity. The chemical analysis according to the Tappi Test Methods was performed in advance of the cooking.[11] The inorganic part of the plant such as ash (TAPPI T 211 om-02) and silica (TAPPI T 245 cm-98) had been determined. The quantity of extractive substances had been determined together with inorganic part especially into acetone and binary mixture of ethanol-toluene (TAPPI T 280 pm-99), into 1% sodium hydroxide (TAPPI T 212 om-88) and also the extractives transitioning into the water (TAPPI T 207 om-93). The chemical analysis of organic part was only focused on determination of the quantity of lignin by Klason (TAPPI T 222 cm-02) and quantity of the Seifert cellulose.[12] The alpha-cellulose, beta-cellulose and even the gamma-cellulose were determined for samples of plants (TAPPI T 203 cm-99) to provide the insight into the quantity of cellulose solubility in hydroxide solutions.

The raw materials were disintegrated into chips of 1cm length in advance of nitrate-alkaline cooking. Nitrate-alkaline cooking was processed in lab conditions by utilizing 2l boiling flasks. Each flask consisted of 50g air dry material and the ratio of the chemical to raw material was 20:1. The cooking process started by 45 minutes of cooking under the reflux condenser in 6% nitric. Then the washing by water was followed by the extraction by 5% sodium hydroxide, the solution was heated and then cooked for 10 minutes. The heating was not performed under the reflux condenser. The additional washing by water was followed by another pulping and the neutralization by 1% acetic acid for 5 minutes.

The cooked pulp was thoroughly washed by water after cooking and the rejects were separated. The pulp was utilized for making of the sheets on lab sheet former RAPID-KOTHEN. The mechanical properties of lab sheets were tested to determine tensile properties on device by FRANK-PTI company according to ISO 1924-2 standard[13] such as breaking length, tensile index or tensile absorption index. The smoothness by Bekk was determined on the device by FRANK-PTI according to the ISO 5627 standard[14] so the results can be reproduced.

3. Results

3.1. Planting areas

Figure 1 shows that there is a significant increase in the share of technical crops together with rapeseed (canola) over the last twenty years. This is also true about area of crop land devoted to poppy seed. At the same time, a decline in barley, rye and potatoes means that less food crops are grown in the Czech Republic over the observed period of time.

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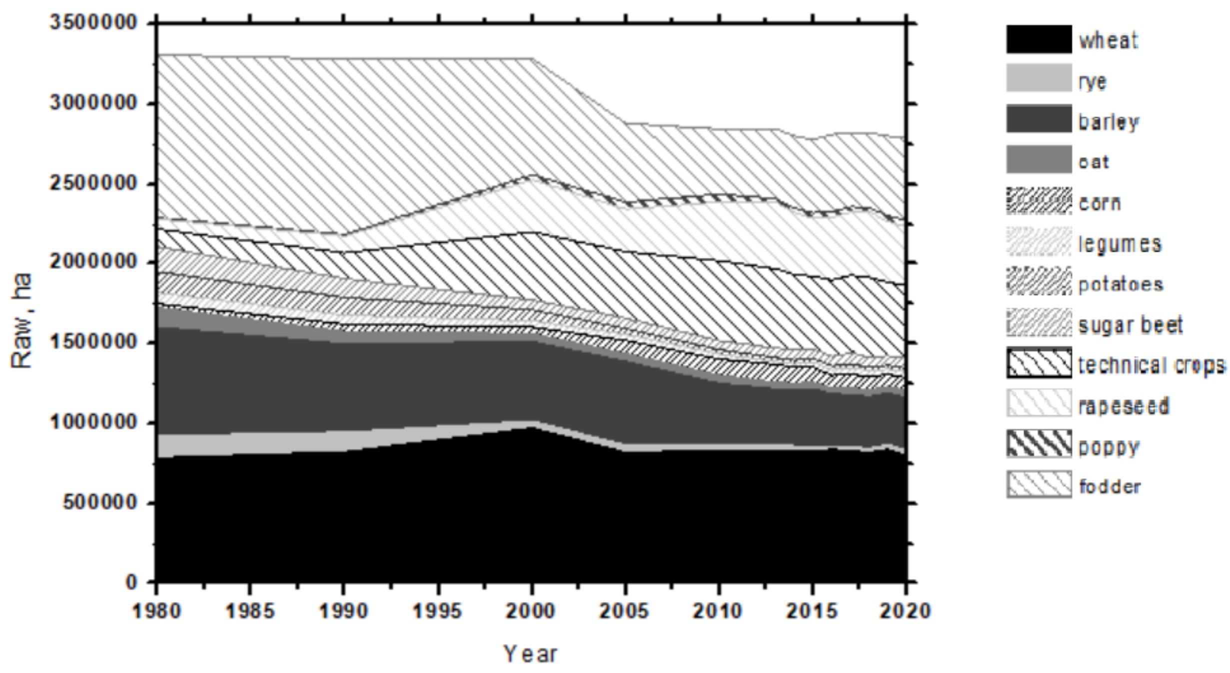

Figure 1. Planting areas in the Czech Republic.

3.2 Chemical analysis

The chemical composition of input material has significant impact on pulp yield and fiber characteristics. Cellulose is the main component of fibers. Non-cellulose components of cell walls are formed by hemicellulose, lignin, pectins and some mineral substances. Table 1 contains the chemical properties of the black mustard (*Brassica Negra* L.) and camelina (*Camelina Sativa* L.). As is generally known, the chemical composition of plants depends on the plant part, plant genotype, climate and even the growing locality. This may be the reason why some of the chemical composition values differs from similar studies.

Table 1. Chemical compounds (in mass %)

Raw	Cellulose	Lignin	Ash	Silica
Black Mustard (*Brassica Nigra* L.)	37.63	28.36	3.327	0.1058
Camelina (*Camelina Sativa* L.)	39.19	20.43	3.286	0.1945

Table 2 contains shows the solubility of cellulose. The properties are being assessed by solubility in alkali and alpha-cellulose from practical point of view. Alpha-cellulose is the part that is non-soluble in 17,5% sodium hydroxide. In contrary the beta-cellulose is not dissolved and is excluded after acidification. The gamma-cellulose remains in the solution even after the acidification. From the chemical point of view the alpha-cellulose is basically cellulose in the meaning of its chemical definition but consists of small quantity of hemicelluloses. The beta-cellulose originally not exists in wood and plants. It arises by the destruction of cellulose. The gamma-cellulose consists of hemicelluloses.[19]

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Table 2. Cellulose solubility (in mass %)

Raw	Alpha-cellulose	Beta-cellulose	Gamma-cellulose
Black Mustard (*Brassica Nigra* L.)	15.09	9.38	3.02
Camelina (*Camelina Sativa* L.)	20.89	14.24	4.13

In addition to chemical analysis of raw materials the determination of solutions, extractible to organic and polar solvents. The sample was disintegrated by ball mill at first. The acetone, binary mixture, ethanol-toluene, cool water, hot water and 1% solution of sodium hydroxide were used as solvents. Table 3 contains the concentrations of solutions which were extracted.

Table 3. Extractives (in mass %)

Raw	Cold water	Hot water	1% NaOH	Acetone	Ethanol-toluen
Black Mustard (*Brassica Nigra* L.)	10.49	8.33	29.75	2.23	10.15
Camelina (*Camelina Sativa* L.)	8.41	6.41	36.97	2.16	4.35

3.2. Nitrate-alkaline pulping

Nitrate-alkaline delignification was processed for the black mustard (*Brassica Negra* L.) and camelina (*Camelina Sativa* L.). The ration of chemicals was 20:1 both for cooking in 6% nitric acid and for extraction in 5% sodium hydroxide.

The delignification degree for the cooked pulp was determined by the Kappa number which was 20.2 for the pulp from black mustard and 23.1 for the camelina. It is obvious that according to these values the cooking with nitric acid and the extraction in sodium hydroxide reaches the Kappa number which is common for long cook pulps or the pulps which were not bleached. So, we can claim that significant delignification occurs during this process. Final yield was not as small as should be expected for long cook time or after the bleaching process. The pulp processed this way was used for paper sheets making for further analysis as tensile index. Table 4 consists data which closely describe general properties and value of smoothness by Bekk for possible reproduction of the pulps from these pulps.

Table 4. General properties of paper from the nitrate-alkaline pulp

Raw	Kappa number	Yield, %	Basis weight, g m^{-2}	Thickness, mm	Smoothness, s
Black Mustard (*Brassica Nigra* L.)	20.2	45.34	77.0	0.0955	21.25
Camelina (*Camelina Sativa* L.)	23.1	38.35	78.5	0.1265	15.53

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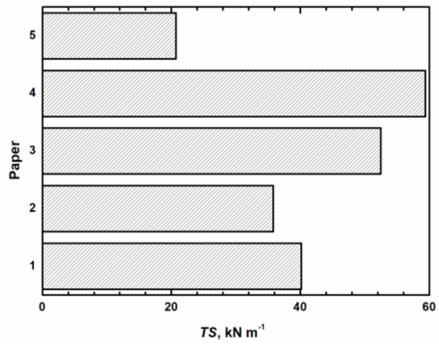
3.3. Pulp characteristics

Table 5 contains the mechanical properties of nitrate-alkaline pulp for plants measured by us compared with authors which examine other agricultural plants.

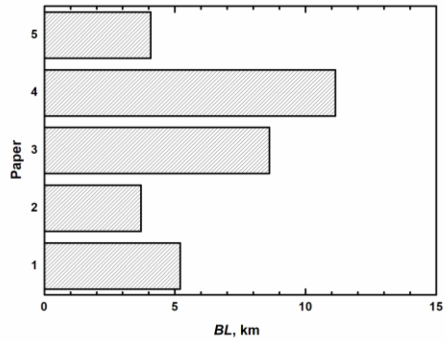
Table 5. Mechanical properties

Pulp	Raw	Breaking length, km	Relative elongation, %	Tensile index, N m g^{-1}	Tensile absorption index, J g^{-1}
Nitrate-alkaline pulp	Black Mustard	7.16	2.31	40.16	1.15
	Camelina	3.66	1.12	35.83	0.27
Soda pulp	Rapeseed stalks[6]	4.06	1.70	–	0.47
	Rice straw[5]	–	–	26.10	–
	Corn[5]	0.30	–	3.20	–
	Okra stalks[20]	–	–	71.94	–
Neutral sulphite semi-chemical pulp	Rapeseed residue[9]	3.63	–	35.60	–
Chemo-mechanical pulp	Rapeseed straw[21]	–	–	37.70	–
	Bagasse[21]	–	–	33.50	–
Peracetic acid pulp	Rapeseed straw[7]	4.20	–	–	–
	Wheat straw[7]	5.90	–	–	–
Ethanol-soda pulp	Sugarcane bagasse[17]	–	–	80.00	–

It is obvious from Table 5 that black mustard has better characteristics than camelina. The black mustard has also good characteristics according to the comparison of the nitrate-alkaline paper characteristics from black mustard with papers made from the other non-wood plants processed by other methods. Only the sugarcane ethanol-soda pulp has higher strength values than the black mustard paper. Same values were achieved for okra stalks soda pulp. In the case of non-wood papers, the measured values by us were compared with in industrially made papers, such as sulfite paper, craft paper and parchment paper. This comparison is described by the Figure 2.



(a)



(b)

Figure 2. Mechanical properties: (**a**) Tensile index in N m g^{-1}; (**b**) Breaking length in km (1 – Nitrate-alkaline paper from black mustard, 2- Nitrate-alkaline paper from camelina, 3- Parchment paper, 4 – Kraft paper, 5 – Sulfite paper).

4. Discussion

The Seifert method, which was chosen to determine the cellulose, provides almost identical content of cellulose as chromatography which is considered most accurate.[12] The content of cellulose by Seifert was similar for both plants. In comparison to rapeseed straw 33.9%,[6] to the corn 33.6%,[15] and to the amaranth 31.9%[4] the values were lower than the values measured by us. In contrary, the values of the sunflower 37.5%,[15] of the wheat 38.2%[5] were similar. The values were lower in comparison to the wood values. Barbash et al.[7] states the values for birch 41.0% and for pine even 47.0%.

The lignin was analyzed by Klason method and the quantity of lignin of camelina is very similar to deciduous in comparison to wood, for oak 21.4% and for beech 24.5% of lignin.[16]

In contrary, the Klason lignin value for black mustard is approximate to values of conifers, pine 29.5%, spruce 30.4%.[16] Housseinpour et al.[15] states the values for wheat 15.3%, rapeseed 20.0%, corn 17.4% and for sunflower 18.2%. These values are very similar to the amount of lignin measured by us.

The ash amount is similar for both plants, lower in comparison to the other non-wood plants. Barbash et al.[7] states the amount of ash 4.5% for the wheat and rapeseed and Carvalho et al.[17] measured 2.3% for bagasse. Houseinpour et al.[15] analyzed the values for sunflower and corn which were significantly higher, sunflower ash was 8.2% and corn ash 7.5% which can be caused by the larger pith in stems. The amount of ash in analysis of the trees was significantly lower than the values measured for non-wood plants, for birch 0.5% and for pine only 0.2%.[7]

The amount of silicates differs. Camelina contains as twice as much more than black mustard. In comparison to rapeseed 0.0064% is significantly higher.[6] The values are higher in comparison to wood which may be caused by fact that the whole plant with fruits not only the stems was used for the chemical analysis in our case. The paper[18] presents the values for pine 0.04%, for beech and spruce 0.01% which is higher than the values of rapeseed but significantly lower than the values for camelina and black mustard.

The alpha-cellulose values are significantly lower than for other plants, for rapeseed 28.8%[6]. The difference for other plants is even more substantial, for wheat 38.2%, for bagasse 51.90, for cotton stems 48.8% and for rice 41.2%.[5] Other authors unfortunately does not state the amount of the alpha-cellulose for other plants neither they state the amount of beta-cellulose and gamma-cellulose.

The amount of extractable solutions soluble in cool water is important for paper production mainly for process of wet pulping. In comparison to the other authors, the solubility for camelina is lower than for the other field plants but the black mustard has similar solubility to wheat 10.7% and to rice 10.7%.[15] In contrary the values for the other plants were higher, for rapeseed 13.8% and the value for the sunflower was closely as twice as higher as camelina 16.5%.[15]

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One would expect that solubility in hot water is higher than in cool water because the polysaccharides of low molecular mass which are non-soluble in low temperature can transition into hot extract. The lower result corresponds to fact that the mixed polysaccharides, polyuronic acids and polyuronides are leaching into cool water and alkaline solution. These easily hydrolyze and already dissolve in cool water. Barbash er al.[7] states the values for rapeseed 10.1%, wheat 6.0%, pine 6.7% which are the values similar for those measured by us. In contrary he states the value for birch only 2.2%. The plants which have larger pith such as sunflower 15.5%, rice 16.2% and corn 14.8%[15] have significantly larger amount of these extractives. It could cause larger part of salts or saccharides in particular plants.

The amount of plants measured by us in 1% of sodium hydroxide was 29.75% in case of black mustard and 36.97% in case of camelina. These values are similar to ones for rapeseed stems 30.8%[6] and for sunflower 29.8%.[15] However the values for wood referred by Barbash et al.[7] are significantly lower, for the birch 11.2% and for pine 19.4%. The composition and the amount of extractives changes based on the specie and the age of plant even the location where the plant is being grown and its part. The hemicelluloses of non-wood plants mainly consist of pentosans which can be isolated by extraction in diluted alkaline.

This may be the reason why the amount of extractable solutions in 1% sodium hydroxide is higher than in other solvents. We can also assume that even the lignin is being partly dissolved in the solution of sodium hydroxide.

The values of solubility in acetone are similar to the case of the rapeseed stems 2.6%,[6] cotton stems 2,9%,[5] bagasse 3.4%[5] or of rice 3.5%.[15] Low values can be caused by the fact that the mentioned plants do not contain such amount of the resin acids which are soluble in acetone.

The transitional amount of dissolved solution in binary ethanol-toluene mixture was only compared to Potůček et al.[6] who states the values for rapeseed 5.3% which are similar to camelina. The other authors focused on the chemical analysis does not state the solubility values of this mixture.

Since the objective of this study has been the production of nitrate-alkaline pulp for non-bleached paper making or the utilization of pulp as the substitute for regenerated brown material with utilizing the crop residues.

The lab sheets were made from nitrite-alkaline pulp from black mustard and camelina and were compared to commonly available papers according to the mechanical properties.

Better properties were achieved for the pulp made from black mustard. Even in case of the breaking length we achieved 7.16 km which came out better than for sulfite wood paper. In comparison to the other authors, the breaking length in our case of black mustard was higher than soda pulp from rapeseed,[6] soda pulp from corn[5] or chemical pulp made by peracetic acid from wheat.[7] In comparison with the kraft pulp 11.15 km, our pulp did not come out with such properties.

The other determined value was the relative elongation or so-called tensile stretch. That was in our case similar to the soda pulp from rapeseed.[6] Unfortunately the other authors who pursued the pulp production from non-wood plants do not state this value.

In case of the tensile index, the value for nitrate-alkaline pulp from black mustard was significantly lower than the values for soda pulp from okra[20] or the pulp produced by the ethanol-soda method from bagasse.[17] in comparison to industrial pulps, it achieved lower values than kraft non-calender paper 59.42 Nmg^{-1} or parchment paper 52.46 Nmg^{-1} while the sulfite wood paper had even lower values 20.68% Nkg^{-1}.

The last determined value was the tensile absorption index which was compared only with the values of rapeseed cooked by the soda method[6] and these values were lower for the black mustard but higher than in case of the camelina pulp.

5. Conclusions

Non-wood materials are important raw material in countries where the wood for paper and paper products production is not available in sufficient quantity. However along with growing consumption of paper and cellulose products the quantity of wood sources is reducing and the usage of non-wood raw materials for pulp production is growing.[22] Among the non-wood plants advantages are annual renewal and low costs of their renewal in comparison to the wood.[7]

In case of non-wood plants, the morphological and chemical composition is different than for. In case of low content of lignin in non-wood plants there is a need for less energy and chemicals in bleached pulp production than in the wood pulp production.[23,24]

The non-wood plants are usable for paper production but for now only by the soda cooking method which represents alkaline pulp production method without sulfur and during which the raw material is delignified in the sodium hydroxide solution in relatively drastic conditions while alkaline degradation of polysaccharides occurs. To avoid

The alkaline degradation of polysaccharides, anthraquinone, ethylendiamine, oxygen or methanol are being added to the solution. The positive impact of anthraquinone on delignification degree rises was published already in 1977.[25] Anthraquinone is applied for two main reasons, to accelerate alkaline cooking and to stabilize saccharides along with conservation of the yield. It is possible to assume that anthraquinone stabilizes saccharides and also interact with lignin which leads to fast and intensive delignification.[26] from the environmental point of view, the presence of the anthraquinone in the black liquor is reflected by lower air pollution because the sulphur-free cooking process can be chosen. Another important aspect is the reduction of bleaching alkaline need when the pool is cooked to lower delignification degree.[27]

However, the mechanical properties of the non-wood pulp are of lower quality than the kraft pulp but even the non-wood pulp has its benefits. Particularly the nitrate-alkaline cooking method because the black liquor shall be used as fertilizer because it contains large quantity of nitrates.

The European pulp and paper industry produce 11 million tons of waste a year.[28] The solution of composting of waste materials in paper production consists in discharging waste resp the sludge settles until most of the paper fibers and organic materials are stabilized (odor / chemically) through exposure to microorganisms with minimal carbon loss. Sometimes fertilizers are added to waste or sludge to increase the nutrient content. This creates a humus material that can be used for application to agricultural land, houseplants and in greenhouses. This is one of the lowest disposal costs by-products in the paper industry. Except for the requirement of large land to spread the sludge, there are several other composting costs.[29, 30, 31, 32] Commercial composts must meet several technical requirements such as degree of maturity or suitability for plant growth. Composts made from organic waste mixed with various amounts of recycled paper and waste from the paper industry meet these requirements. Furthermore, some parameters of compost, such as salt and organic content and process specific leachate emissions are positively affected by the additional waste from the paper industry into the composting matrix. The concentrations of harmful substances, in particular heavy metals should be considered as a limiting factor.[33]

The application of composted MSW to agricultural land has several beneficial effects. Generally, compost increases soil fertility by adding nutrients such as N, P and K, thus substituting mineral fertilizers.[34, 35] The addition of compost also increases plant health primarily by protecting against plant pathogens.[36, 37] Furthermore, the addition of organic materials and the associated increase in soil organic matter (SOM) has been associated with many positive effects such as improved soil structure, increased water holding capacity and infiltration, increased workability and reduced erosion.[38, 39]

Several already issued and other foreseen European Union directives have a great influence on the waste management strategy of paper producing companies. Through legislation, the landfill option is restricted, although it has not phased out on-site landfills. Due to the large quantities of waste generated the high moisture content of the waste and the changing composition, some recovery methods, for example, conversion to fuel components, are simply too expensive and their environmental impact uncertain. The thermal processes, gasification, and pyrolysis seem to be interesting emerging options, although it is still necessary to improve the technologies for sludge application. Other applications, such as hydrolysis to obtain ethanol, have several advantages (use of wet sludge and applicable technology to sludges) but these are not well developed for pulp and paper sludges. Therefore, at this moment, the minimization of a waste generation still has the highest priority.

The summarized knowledge in this paper describe that non-wood plants are usable to pulp production, unfortunately by soda cooking method, yet. But also, because the produced pulp mechanical properties are below the values for the bleached pulp made from wood by as yet mostly used kraft method.

Even though the crop fields in the Czech Republic are mostly sown under corn or rapeseed this article has been devoted to other plants which remains in the field and their sown areas has been growing in the last years.

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