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Review

# Chemical pulp from corn stalks☆

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ABSTRACT

This work aimed to carry out chemical cooking of corn stalks, both in a nitrate-alkaline manner and in a soda pulp method. The composition of corn is characterized by cellulose, lignin, ash, and substances extractable into polar and organic solvents. Handsheets were made from the pulp, for which the degree of polymerization, sedimentation rate, and strength properties was determined.

## 1. Introduction

Due to the lack of fibrous raw materials, especially softwood, annual plants are currently used in the paper industry. Today, wood is the primary source of the paper industry. Many countries lack wood resources to meet growing demand. In addition to wood pulp, this causes the use of fibers and waste from the agricultural industry to produce pulp and paper. Approximately 70% of the raw materials in the pulp and paper industry in China and India come from non-wood plants [1].

Hemp, sisal and flax are just among the plants that have been used for many years in the production of paper. According to the crops planted, field planting areas in the Czech Republic have changed significantly in recent years. The largest planting areas are sown with cereals, such as wheat, barley, or rapeseed, but recently there has been a significant increase in areas planted with corn, poppy or mustard. Annual plants have chemical and morphological properties different from wood materials, which is the reason for other conditions for cooking or subsequent bleaching [2].

Non-woody plants are processed by a chemical method. Amaranth, atriplex, Jerusalem artichoke [3], rapeseed straw [4], wheat [5], rice [6] and sugarcane [7, 8] were cooked by using soda pulping.

As with other chemical methods, peracetic acid has been used to cook pulp from wheat, canola, amaranth, and lavatera [9]. In addition to the use of peracetic acid, these authors also considered the use of performic acid to produce pulp from wheat straw. Another widely used option is sulfite semi-chemical pulp from rapeseed straw [10, 11] or rice [6].

Another chemical method of pulp production is the nitrate-alkali process, in which the bond between cellulose and lignin is hydrolytically released during cooking in nitric acid. Lignin is nitrated and simultaneously, partially oxidized to nitrolignin, which can be dissolved in bases [12, 13].

Corn (Zea Mays) is, along with wheat and rice, one of the most cultivated crops in production and consumption [14,15]. It covers up to 147 million hectares of agroclimatic areas worldwide [16]. Corn can provide feed, fuel, food, and fiber in over 3000 products [16, 17]. This work aimed to cook the pulp from corn stalks using the nitrate-alkaline method and the natron industrial method. The general properties of the cooked pulp were determined. At the same time, handsheets were produced from the pulp, and their mechanical properties were examined. This study is focused on research into the chemical production of pulp from agricultural waste. It extends the results to other potential raw materials for producing cellulosic fibers.

## 2. Experimental

Corn straw (Zea Mays) was grown in the lowlands around rivers in the Central Bohemia region. The raw material consisted of stems stripped of the remaining leaves and roots.

Before pulp production, corn straw was chemically analysed

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according to TAPPI methods. In the case of corn straw samples, the inorganic content was first determined in the form of ash according to TAPPI T 211 om-02 [18] and analysis of silica according to TAPPI T 245 cm-21 [19] was also performed. Random samples for further chemical analysis were first ground using the MF 10 BASIC knife mill from IKA (Staufen, Germany) and subsequently sieved. Then followed the determination of the organic part utilizing Soxhlet extraction with binary ethanol-toluene mixture according to Tappi T 280 pm-99 [20]. From the polysaccharide part, the cellulose content was determined according to Seifert method [21] and the lignin proportion according to Klason method using 72% sulfuric acid, i. e. Tappi T 13 wd-74 [22]. Solubility in non-polar solvents was further analysed, specifically in water according to Tappi T 207 om-93 [23] and in 1% sodium hydroxide Tappi T 212 om-02 [24].

The pulp for this study was cooked in two ways. For both methods of pulp production it was necessary to disintegrate the corn straw into 1–2 cm long chips. Before placing them into the container where the batch needs to be made, it was necessary to determine the moisture content, which was around 6% depending on the relative humidity of the air.

In the case of nitric-alkaline cooking, the raw material was boiled in 6% nitric acid for 30 min under total reflux and then washed with water. Washing was followed by extraction in sodium hydroxide with a concentration of 5%; the raw material was cooked for 10 min. Boiling in acid and hydroxide was followed by thoroughly washing, pulping, and neutralizing with 1% acetic acid and washing again.

For cooking soda pulp a laboratory rotary cooker was used, in which 19% sodium hydroxide in the 5:1 hydromodule was used as the cooking liquid. The temperature regime consisted of four phases. First, the raw material was heated to a temperature of 105 °C. It was impregnated at this temperature, followed by further heating to a temperature of 160 °C. In the last phase, cooking was done to the required H-factor of 1250 h. Therefore, the cooking time was around 240 min I this case. After the cooking process, as in the case of the nitrate-alkaline pulp, the pulp was washed with water and pulped.

For the pulp, general properties were first determined, such as the amount of rejects, the degree of cooking and the so-called Kappa number according to ČSN ISO 302 [25] and the total yield. In addition to these general characteristics, the degree of polymerization was measured, which was determined from the limit viscosity number using a capillary viscometer. Viscometric determinations were performed following Tappi T 230 om-08 [26]. However, the test solvent was sodium tartrate with iron complexes, abbreviated FeTNa [27]. Another characteristic was pulp rheosedimentation, a specific type of disturbed sedimentation. This method is based on monitoring the description of the kinetics of spontaneous movement of the network formed from the fibrous components of the suspension during sedimentation or flotation. So that method can be classified as a rheological method. The process of determination was carried out according to Milichovský [28]. It is a time dependence of the position of the interface between the pulp suspension and the clear liquid above the suspension. The final mesh concentration and sedimentation rate were calculated based on the initial pulp concentration in 2 litters of suspension.

The obtained pulps were used to produce handsheets on the RAPID-KÖTHEN laboratory sheeter (Birkenau, Germany), too. Each sheet had a basis weight of approximately 80 $g{\cdot}m^{-2}$. The manufactured sheets were subsequently tested for tensile properties according to the ISO 1924–2 standard [29] using equipment from the FRANK-PTI company (Birkenau, Germany). Mechanical properties include breaking length, tensile strength index, or tensile absorption. In addition, the tensile strength was also analysed as burst that was determined according to ISO 2758:2014 [30] using equipment from the FRANK-PTI company (Birkenau, Germany). All these strength parameters were determined on at least 20 samples and the results were statistically processed. In addition to the strength tests, determination of air permeability according to Gurley method specified in the ISO 5636–5:2013 [31], was also performed using equipment from the Lorentzen & Wettre company (Stockholm, Sweden).

## 3. Results and discussion

### 3.1. Chemical analysis

The chemical composition of the input raw material has a significant effect on the fiber properties and the pulp yield. Table 1 contains the average chemical composition of the individual components of corn straw and other similar raw materials from annual plants for comparison. As is known, the chemical representation in plants depends on the plant's genotype, the plant part, the climate and the place of cultivation. This is also why the values in similar studies dealing with the processing of corn for paper production may differ.

Since the representation of the individual analyzed components is defined as the ratio of the weight of the given component to the absolutely dry sample, the moisture content of the analyzed samples was determined first. For the corn measured by us, the amount of ash, or the mineral substances, was 8.4%, which is similar to the values obtained by Housseinpour et al. [35] 7.5%, but Fagbemigun et al. [32] report only 5.1%. The ash content in corn is similar to the ash value in sunflower, 8.2% [35]. A more miniature representation was achieved for wheat 4.7% [35] and rapeseed straw 4.8% [33]. This is due to the more significant amount of pulp in the stems of the cuckoo. For woody plants, these values are even lower than for annual plants precisely because of a higher amount of inorganic substances in the pith of annual plants. About 0.5% was found in birch and only 0.2% in pine [9].

In our case, the inorganic fraction insoluble in sulfuric acid, which gives us the amount of silicates, was 2.9%. This content was similar to the values for wheat 3.0% and bagasse 2.0% [34]. In professional articles dealing with wood composition, these values are not mentioned because the representation of silicates is generally higher in the annual plants.

Klason's lignin was found in corn stalks at 20.1%. This amount is similar to the lignin content found in other annual crops, i.e., wheat 15.3%, rapeseed straw 20.0%, corn 17.4%, and sunflower 18.2% [35]. Compared to deciduous trees, the value we measured is very similar, e. g., oak contains 21.4% and beech 24.5% of lignin [36]. Conversely, coniferous wood contains more lignin, pine 29.5%, and spruce 30.4% [36].

The Seifert cellulose content of our analyzed corn was similar to corn samples analysed by Faugbemigun et al. [32] and slightly lower than rapeseed straw [33]. On the other hand, there is also reported the cellulose representation of 37.5% for sunflower [35] and 38.2% for wheat straw [4]. Our measured values are close to the wood values reported by Barbash et al. [9], namely 41.0% for birch and 47.0% for pine.

In addition to determining the essential chemical components, the amount of substances extractable into organic, inorganic, and polar solvents were also analysed. A binary mixture of ethanol and toluene, cold water, hot water, and 1% sodium hydroxide were used as solvents. Table 2 shows the contents of extractable substances from the mentioned samples and expresses the percentage representation based on an absolutely dry sample.

The amount of solute transferred into the ethanol-toluene mixture was determined to be 9.9% for corn. A sample from this extraction was then used to determine Seifert cellulose and Klason lignin. The amount

**Table 1**
Chemical composition of the investigated corn straw (in mass% of oven dried samples).

| Raw material / Chemical composition | Ash (%) | Silica (%) | Lignin (%) | Cellulose (%) |
|---|---|---|---|---|
| Corn | 8.4 | 2.9 | 20.1 | 47.6 |
| Corn [32] | 5.1 | – | 15.0 | 44.1 |
| Corn [35] | 7.5 | – | 17.4 | 33.6 |

**Table 2**
Extractive contents of the investigated corn straw and other similar raw materials from annual plants for comparison (in mass% of oven dried samples).

| Raw material / Extractive content in the used solvent | Alcohol-X | Cold water | Hot water | 1% NaOH |
|---|---|---|---|---|
| Corn | Alcohol-Toluene 9.9 | 10.7 | 11.0 | 48.0 |
| Corn [35] | Alcohol-Acetone 9.5 | – | 14.8 | 47.1 |

of extract passing into ethanol-toluene is similar to that when using the ethanol-acetone mixture [35]. However, no author reports the values of annual plants for ethanol-toluene extraction, so at least a similar organic solvent is given for comparison. Compared to wood, this amount of ethanol-toluene extract is similar to pine 10.4% but is significantly higher than for beech 2.6% and spruce 2.7% [36].

In the case of the cold water extract, which is particularly important in the wet pulping process in pulp production, we achieved the same value as Housseinpor et al. [35] for wheat, 10.7%, and rice, 10.7%. However, this value was higher in rapeseed straw, 13.8%, and almost twice as high in sunflower, 16.5% [35].

Despite the expectation that the solubility of investigated sample in hot water would be higher, the value was almost the same (there is only a 0.3% difference). This may be because low molecular weight carbohydrates, polyphenols and dyes are more easily transferred into hot water. The amount of starch insoluble in cold water and partially dissolved in hot water could affect this value more significantly [37]. Barbash et al. [9] measured a similar value for rapeseed straw of 10.1%. Annual plants with a larger amount of pith have higher amount of these extractives (e. g. rice 16.2%, sunflower 15.5%, and corn 14.8%), which could be due to a more significant proportion of salt or carbohydrates in a particular plant [35].

The value of extractives content in 1% sodium hydroxide is slightly lower for corn straw than rice and similar to wheat straw [34]. However, for woody species, the values are significantly lower, namely 19.4% for pine and 11.2% for birch [9]. This extractive content can be high because annual plants contain more pentosans that can only be isolated with dilute alkali. In the same way, it can be assumed that a part of the lignin also passes into the solution.

The composition and amount of extracted substances change depending on the type and age of the plant, but also on the plant's location and the plant's parts (stalks, leaves, roots, etc.).

### 3.2. General properties

At the end of the cooking process, the stalks were washed with water to remove any remaining cooking lye. The waste liquor was collected for subsequent analyses. The results of the analysis of the cooking liquor, so-called black liquor, are shown in Table 3.

The table compares the values for our cooked pulp with kraft liquor. Nitrate-alkaline liquor from corn was obtained after boiling first in nitric acid, followed by extraction with hydroxide. With the help of nitric acid, lignin in the raw material is nitrated to nitrolignin, and then delignification occurs in the hydroxide. The entire process of this cooking takes tens of minutes. However, soda liquor from corn is cooked for several hours only in sodium hydroxide. Kraft black liquor is boiled in a sodium hydroxide and sodium sulfide solution for several hours. Soda pulp and kraft pulp are also commonly used processes in the industry, but similar values can also be achieved with nitric-alkaline cooking. Although such delignification does not occur during corn cooking, the concentration of alkaline lignin, as reported by Potůček et al. [38], so the density of black liquor is very similar to the kraft method.

Table 4 shows the general characteristics of the pulp, such as the yield, the representation of rejects, the degree of delignification, and others. The differences in the delignification processes of the studied corn are conditioned by its anatomical structure, which affects the impregnation process (speed and depth of penetration of delignifying agents into the intercellular space of the corn), as well as different chemical composition (reactivity of lignins with the used delignifying agents).

The total yield in the case of corn is meager compared to the typical total yield for chemical pulp. Generally, the total yield for chemical pulp is approximately 30 – 45%. The no-boil amount is low, so there is almost no waste in these chemical batches. Regarding the degree of delignification, the so-called Kappa number is significantly low when cooking nitrate-alkaline pulp, even if the batch does not last even 1 h. The final mesh concentration is lower for both than for chemical pulps. However, the values differ significantly in the rate of sedimentation. It is clear from the above data that nitrate-alkaline pulp achieves better properties because the lower the sedimentation rate, the better the fiber bonds are formed, which is a value that is important in paper production [28]. The last value is the degree of polymerization, which is almost the same for both pulps.

### 3.3. Mechanical properties

The mechanical properties were measured for laboratory sheets of nitrate-alkaline corn pulp and soda corn pulp. The results are shown in Table 5.

The main objective of this study is to evaluate the mechanical properties of papers made from corn stalk pulp. Better mechanical properties were achieved when the sodium pulping process produced the pulp. Even in the case of the breaking length, when the paper breaks by its weight, the value is almost twice as high as when processing recycled papers [33]. In the case of the tensile strength index, the value of sodium pulp was almost 17 times higher, and in the case of tensile tear strength, the value of sodium pulp was also more favorable. This may be related to the increase in ductility. The tensile work absorption index is an essential quantity that characterizes the properties of sack paper. In particular, it must be strong enough to withstand rough handling. Kraft pulp from conifers is commonly used for production. For sack paper, the authors achieved a tensile strength of 78.4 N•m•$g^{-1}$ and a tensile absorption index of 1.78 J•$g^{-1}$ [39], which are values very similar to corn soda pulp.

**Table 3**
Properties of the black liquor from our investigated soda and nitrate-alkaline pulping process of corn straw compared to black liquor from wood pulping (all measured parameters were determined at the temperature of 22 °C).

| Pulping process, resp. Pulp type / Determined properties | pH | ρ, kg•$m^{-3}$ | μ, Pa•s | γ, mN•$m^{-1}$ | $c_{AL}$, g•$l^{-1}$ |
|---|---|---|---|---|---|
| Nitrate-alkaline corn pulp | 11.84 | 1081.16 | 1.19 | 38.47 | 1.71 |
| Soda corn pulp | 13.20 | 1133.58 | 1.65 | 60.73 | 3.58 |
| Kraft hardwood pulp [38] | 12.20 | 1071.00 | – | – | 27.00 |
| Kraft softwood pulp [38] | 12.60 | 1097.00 | – | – | 56.00 |

pH – pH value, $\rho$ – density, $\mu$ – viscosity, $\gamma$ – interfacial tension, $c_{AL}$ – concentration of alkaline lignin.

**Table 4**
The determined general properties of corn pulp.

| Pulp type / Determined properties | $Y$,% | $x_R$, % | $\kappa$ | $c_F$, kg•$m^{-3}$ | $v_0^S$, mm•$s^{-1}$ | $DP$ |
|---|---|---|---|---|---|---|
| Nitrate-alkaline corn pulp | 14.11 | 0.38 | 22.23 | 8.22 | 7.36 | 703 |
| Soda corn pulp | 16.95 | 1.44 | 45.83 | 8.34 | 34.13 | 687 |

$Y$ – yield, $x_R$ – amount of rejects, $\kappa$ – Kappa number, $c_F$ – final concentration of pulp mesh, $v_0^S$ – sedimentation velocity, $DP$ – degree of polymerization.

**Table 5**
Measured mechanical properties of investigated pulps compared to other pulps and liner.

| Pulp type / Mechanical properties | Air permeance (Gurley), s | Breaking length, km | Relative elongation, % | Tensile index, N•m•g$^{-1}$ | Tensile absorption index, J•g$^{-1}$ | Burst index, kPa |
|---|---|---|---|---|---|---|
| Nitrate-alkaline corn pulp | 78.74 (1.031) | 0.52 (0.027) | 1.54 (0.385) | 5.06 (0.684) | 0.04 (0.019) | 112.66 (3.648) |
| Soda corn pulp | 90.14 (0.483) | 5.42 (0.048) | 3.42 (0.239) | 83.16 (4.425) | 1.34 (0.321) | 257.18 (5.012) |
| Kraft liner from recycled paper [33] | 8.00 | 2.98 | – | 60.70 | – | 159.00 |
| Kraft pulp from conifers [39] | – | 7.99 | 3.20 | 78.40 | 1.78 | – |

## 4. Conclusions

The use of agricultural waste materials is essential, especially in countries with a shortage of primary wood resources. With the increasing consumption of paper, the amount of wood resources in other countries is also decreasing and therefore, the demand for the use of non-wood raw materials for the production of pulp is increasing. The advantage of non-wood raw materials is their annual renewal and low costs compared to wood.

It should be note that the chemical composition of non-wood raw materials varies a lot as specified in this paper. In the case of low lignin content in non-wood raw materials, the energy and chemicals consumption is lower than in the production of pulp from wood, too. Many different delignification procedures are used for producing paper from annual plants, but the soda process is the most suitable for industry.

The summarized findings in this paper show that, in terms of strength properties, the soda pulp is preferable when corn straw is used. Even these values reach values better than those of recycled paper. However, in terms of general properties, they cannot be compared with pulps made from wood, because the yield of these corn straw pulps is much lower than that of wood.

The number of sown areas in the world, especially corn, is an additional benefit. Comparing all the previous aspects, corn appears as a possible potential source of chemical pulp in industrial scale.

In further studies, it would be useful to focus on different degrees of delignification, primarily for the soda pulp, which appears to be more suitable than nitrate-alkaline pulp.

## CRediT authorship contribution statement

**Kateřina Hájková:** Conceptualization, Supervision, Formal analysis, Validation, Investigation, Visualization, Writing – original draft, Writing – review & editing. **Tereza Jurczyková:** Conceptualization, Validation, Investigation, Visualization, Writing – original draft. **Michaela Filipi:** Formal analysis, Investigation. **Jiří Bouček:** Formal analysis, Writing – original draft.

## Declaration of Competing Interest

There are no conflicts to declare.

## Data Availability

No data was used for the research described in the article.

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