AustroCels Biorefinery in the Course of Time

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Abstract

The exceedance of planetary boundaries accompanied by an environmental crisis and increasing consumer demands for sustainable products calls for a thorough transformation of the industry. In particular, pulp and paper industry is at the forefront of a sustainable bioeconomy, by utilizing renewable raw materials.

In this article we provide a short overview of the AustroCel Hallein transformation from a traditional paper making company toward a modern biorefinery and provide some general considerations regarding the potential of pulp mills as biorefineries with a specific focus on bioethanol production.

History

The AustroCel biorefinery, located in Hallein, dates to the year 1889. In Manchester the industrialist Edward Partington and the Austrian chemist Dr. Carl Kellner founded the company "The Kellner Partington Paper Co. Ltd". One year later in 1890 the Austrian company was registered and the construction of the factory in Hallein started.

The location of the mill in Hallein had three major advantages for pulp and paper production. In the close surrounding there were large amounts of spruce wood available that could be transported on the river Salzach. The river Salzach also provided sufficient energy for the mill and the close salt mine provided brine for the required production of bleaching chemicals.

In 1898 the first paper-making machine was put into operation and in 1914 already 18.000 to of pulp and 4.500 to of paper were produced.

The period of the 1st WW, the interwar period and the 2nd WW posed a substantial challenge for the production.

However, at that time the efforts to establish a bioethanol production on site started. From 1941 on bioethanol based on the fermentation of sugars obtained during pulping was produced. These developments represent the initial groundwork of the novel bioethanol plant of AustroCel ramped up beginning of 2021.

After WW 2 "Kellner Partington" soon became one of the largest pulp producers in Austria. Beginning of the 1960s the mill had to struggle with the general overcapacity in the market for pulp and paper. Hence, the focus was shifted to high-quality print and writing paper. The highlight of this development was the implementation of a new paper-making machine, which was the largest "printing paper machine" in Austria.

Mid of the 1970s the mill reached a capacity of 85.000 to of pulp, 110.000 to of paper and 35.000 to of coated paper.

The 1980s can be summarized as the "environmental decade", including the implementation of a waste-water treatment plant, a novel pulp production, replacement of the chlorine bleaching, etc. and in 1990 the mill was the largest industrial company in the state of Salzburg with 1200 employees.

In 2009, due to the continuous decline of the demand for "coated papers" the paper production at the mill in Hallein was stopped and the last "Tambour" was delivered.

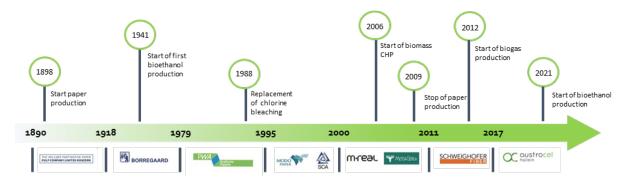


Figure 1: Important Development steps of the AC biorefinery during different ownerships of the mill

After this harsh cut, intensive consideration regarding possible future production scenarios took place and the decision was made to focus on the fabrication of high-quality dissolving pulp. After investments of around 60MEuro production started in 2013.

Within the last 15 years AustroCel invested substantially in biorefinery activities, including a biomass CHP; biogas production; and bioethanol production.

Wood based Biorefinery

The current climate crisis makes a thorough change of our current economic system, that is mainly based on the utilization of non-renewable resources, toward renewable resources necessary. Currently, refined fossil oil is the dominating resource for a multitude of products (e.g., polymers, energy, etc.).

In analogy to this conventional refining, it is the strategy of "biorefinery approaches", to utilize renewable agricultural and forestry feedstocks for material- and energy applications. The various biorefinery concepts mainly differ in the utilized feedstock. For example, there are sugar, or starch-based biorefineries. In the case of lignocellulosic biorefineries wood is utilized as the starting feedstock. Different processing steps of this biomass result in various intermediate or end products, such as chemicals, raw materials, or bioenergy.

Characteristic feature of biorefineries is the coupling of material- energy-based product pathways. Hence, biorefineries contribute to a sustainable transformation from two sides: from a material- and an energy perspective.

In principle, two main biorefinery concepts are used 1) "bottom up" and 2) "top down" systems. "Bottom-up" approaches represent typical extensions of exist-

ing biomass valorization plants such as pulp mills, whereas "top-down" facilities are designed from scratch to obtain a multitude of products from specific raw materials.

In this regard, the European pulp and paper industry is at the forefront of the increasing biorefinery efforts, by utilizing the renewable resource wood from sustainable sourced forests. Apart from the two main products pulp and paper, numerous other bio-based products are already fabricated.

According to a recent study, there are about 139 biorefineries active in Europe, mainly pulp and paper facilities. About 3% of the revenues of the pulp and paper industry result from biorefinery products, with an upward trend. [Cepi 2021]

The following part provides a brief overview and background regarding the potential of pulp mills based on acidic sulfite pulping for biorefinery approaches.

For dissolving pulp, it is necessary to obtain a very pure cellulose fraction. For that, the other wood constituents, including hemicelluloses, lignin and extractives needs to be removed. These constituents account for the potential side-streams and side products.

For example, during the acidic Mg-sulfite pulping around 47% of the mass of spruce wood is removed in side streams. Per to of spruce wood this accounts for 255 kg lignin, 140 kg galacto-glucomannan, 58 kg arabino-glucuronoxylan and 18 kg extractives. (Sixta in Handbook of pulp).

Most of these side products are in the brown liquor after the pulping process. The polysaccharides are hydrolyzed due to the acidic conditions. Different rates of hydrolysis yield oligo or monosaccharides. The hemicelluloses' structure determines the carbohy-

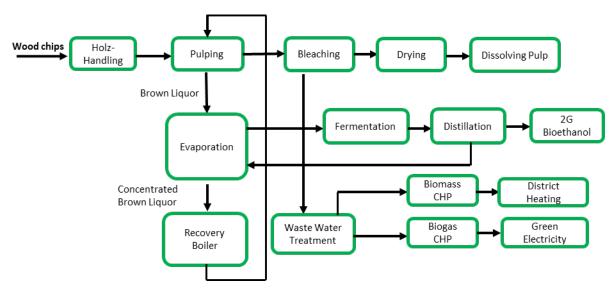


Figure 2: Flow Scheme of the AustroCel Biorefinery

drate profile in spent liquors. Galacto-glucomannan tends to be cleaved down to the monomers, whereas glucurono-xylan is resistant to total hydrolysis due to glucuronic acid side groups.

In the case of spruce wood, the brown liquor is mainly composed of mannose, galactose, glucose, xylose, and arabinose. The C6 sugars represent an ideal feedstock for fermentation approaches, for example bioethanol production.

In contrast to the alkaline Kraft-pulping the lignin is not split at the ether bonds; but rather sulfonated, which results in a solubilization of the Lignin.

The resulting lignosulfonate is characterized by a good water solubility; high MW and can be used for dispersing applications.

The fourth main constituent of wood are so-called extractives, which are lipophilic compounds and specific for the various wood species. For example, spruce extractives are rich in terpenes, including alpha-pinene (Sjöström, 1993). During sulfite pulping alpha-pinene is oxidized to p-cymene (Rydholm, 1965), an aromatic compound, which then can be isolated from cooking liquors and can be utilized in manifold applications.

After removal of the brown liquor the pulp undergoes a bleaching sequence. The hereby removed organic material can be used for biogas production. Typical components found in a bleaching effluent from a sulfite pulp mill are a mixture of hydroxycarboxylic acids, fatty acids, methoxybenzoic acids and other lignin-derived compounds (Bogolitsyna, Holzforschung 2012).

A rather novel development for pulp and paper industry is driven by the need to foster circularity of raw materials and covers strategies to re-use cellulose-based textiles.

In general, separation of cotton or regenerated cellulose fibers from synthetic fibers is complex and costly and the degree of polymerization (DP) of cellulose chains might be small due to the production process and numerous washing cycles.

One strategy for reuse of the cellulose fraction is its hydrolysis and fermentation while the synthetic fraction can be recycled in the fiber production cycle.

Hydrolysis and fermentation of cotton parts of textile waste offers one way to recycle the synthetic fraction in fabrics and convert the used cellulose to fermentable sugars. Deposition or incineration can be avoided, and the fermentation products are capable to replace fossil based raw materials.

AustroCel Biorefinery

The AustroCel Hallein GmbH (AC) currently employs around 290 people and is one of the leading producers of textile pulp from spruce wood. The biorefinery in Hallein produces up to 160.000 to dissolving pulp, 100GWh district heating and 100 GWh green electricity. By that AC provides green energy

for about 25 000 households and district heating for 10 000 households. Since 2021 AC runs the largest 2G bioethanol plant based on wood, with a capacity of up to 35 Mill liters per year.

Bioethanol¹

The current EU policy for renewable energy including bioethanol is based on the EU Energy and Climate Change Package (CCP) and the Fuel Quality Directive (FQD). The so-called Renewable Energy Directive (RED) represents one part of the CCP and provides the specific requirements for liquid biofuels. (Flach, Lieberz, & Bolla, 2019). In 2019 an amendment, the RED-II, was published. It implies, that the share of renewable energy in the final energy consumption must be at least 14% by 2030.

Austrian blending mandates between 2012 and 2020 were 5.75% overall, divided in 6.3% biodiesel and 3.4% bioethanol. Since 2020, the overall percentage is 8.75% without division between fuels. The introduction of E10 was already discussed, but never enforced. Double counting is valid for waste materials and residual products from agricultural and forestry production including fisheries and aquaculture, residues from processing, cellulosic non-food materials or lignocellulosic materials. (Lieberz, 2019)

Further legislation, transposing RED-II into national law has yet to be created and will consitute the framework for targets beyond 2020. Setting specific targets for the use of advanced biofuels (eg. 2G bioethanol), according to RED-II, will increase market demand for advanced biofuels. RED-II foresees following targets for advanced biofuels: 0.2% by 2022, 1% by 2025 and 3.5% by 2030 of final consumption of energy in the transport sector.

The current production of advanced bioethanol in the EU is estimated at around 50 million litres. (Flach, Lieberz, & Bolla, 2019) Most advanced bioethanol producers utilize agricultural residues, such as wheat straw or corn stover. Borregaard and Domsjö Fabriker are utilizing brown liquor from wood pulping for their production, such as AustroCel Hallein. St1 is fermenting organic wastes to bioethanol. (ETIP Bioenergy, 2020)

Table 1 lists operational advanced bioethanol production facilities in Europe. The joint capacity amount to 63,420 t/y (equal 79.9 million litres).

Bioethanol at AustroCel Hallein

Already between 1941–1988 bioethanol was produced in the pulp mill in Hallein with a capacity of 60001/d. This time established experience with brown liquor as substrate. From 2007–2009 a technical preproject, including a conceptual engineering was performed.

In 2011, the transformation from paper-to dissolving wood resulted in an increased sugar content in the spent sulfite liquor, which enabled a higher bioethanol yield.

Hence a new project was established, and it took 3,5 years from concept and basic engineering (July 2017) to full scale production in 2021.

AustroCel Hallein already conducted 60 fermentation and distillation trial runs in lab scale. Substrate and by-products were comprehensively analysed. Different yeast strains and their properties were cultivated and evaluated in a microbiology lab. Process parameters and their effects on sugar conversion rate and yeast viability were tested and a pilot fermentation plant was operating for more than 2 years.

30 million litres bioethanol per year, accompanied by substantial CO₂ saving, could substitute about 1% of gasoline demand by 2025.

¹ Parts of this chapter were previously published in the Biofit Case Study report, a Horizon 2020 project.

 Table 1: Other operational advanced bioethanol production facilities in Europe (status 2020)

Company	Country	Start-up Year	Capacity t/y
Borregaard Industries	Norway	1938	15,800
Domsjö Fabriker	Sweden	1940	19,000
St1 Cellulonix Kajaani	Finland	2017	8,000
St1 Etanolix Jokioinen	Finland	2011	7,000
Chempolis Ltd. Biorefining Plant	Finland	2008	5,000
St1 Etanolix Gothenburg	Sweden	2015	4,000
Clariant Sunliquid	Germany	2012	1,000
St1 Etanolix Hamina	Finland	2008	1,000
St1 Etanolix Vantaa	Finland	2009	1,000
St1 Etanolix Lahti	Finland	2009	1,000
IFP Futurol	France	2016	350
SEKAB Biorefinery Demo Plant	Sweden	2004	160
Borregaard BALI Biorefinery Demo	Norway	2012	110

The bioethanol plant at AC has operational and investment advantages compared to a greenfield scenario. Feedstock and energy supply of the pulp production process provide ideal boundary conditions. Additionally, it is an investment for a sustainable bioeconomy and a step forward to fulfilment of blending mandates for advanced biofuels according to RED-II.

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Lyohemp[™] Fibres from Hemp Shive Dissolving Pulp

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Abstract

Hemp shives, which were grown in various areas and harvested, disintegrated and treated using different methods, were investigated. First sulphur free alkaline digestions of these hemp shives were carried out after physical-chemical characterisation. The obtained pulp was washed and bleached without using chlorine compounds and complexing agents. All required target parameters (residual lignin, degree of polymerization, solubility in Cuoxam) comply with limits for metal contents) could be achieved after optimisation of the conditions during digestion and cleaning. A scale-up into large lab-scale was successful and the resulting pulp is suited for spinning tests. Therefore, the first filaments based on 100% of hemp shives pulp are ready for presentation. The production of a pulp for making continuous filament and staple fibres turned out well. The transfer of all investigated processes in the industrial scale is possible and the pulp can be produced as far as possible by environmentally compatible means.

Introduction

Cellulose man-made fibres (CMMF) relive a strong increase of market demand. About 10 million tons should be enquired until end of this decade [1]. Because of the shortage of wood as resources the interest in using agricultural residues as raw material for pulp production is growing. Hemp as an interesting agricultural plant for soil improvement can be used for nutrition, cosmetics and isolation material.

Figure 1 shows the typical composition of mechanical treated hemp straw [2].

The textile use of fibres has a long tradition, but cotton on one hand and man-made on other hand led to replacement of this material. In a former project the development of LyohempTM based on hemp fibres was successfully investigated [3, 4]. The aim of our research now was to develop an environmentally compatible method for pulping hemp shives as raw material for manufacturing of LyohempTM fibres.

Shives (amount about 55%) as a typical side product of hemp cultivation are the largest share of the hemp straw and so its material use seems very attractive. Actually hemp shives are used as animal bedding or for loam constructions inforcement. Nevertheless, the revenue is low. Goal of a collaboration project with FUDI Futtermittel und Dienstleistungs GmbH & Co. KG (Zeulenroda, Germany), MATRAK Service und Lohnarbeits GmbH (Auma-Weidatal, Germany), IPWC and TITK was the development of a technology for production dissolving pulp based on this raw material. The pulp should be suited for production of Lyocell, or better LyohempTM, and should be characterised by a completely environmentally friendly process. So a sulphur free digestion without anthraquinone as well as washing and bleaching without chlorine components and complexing agents were in the focus.

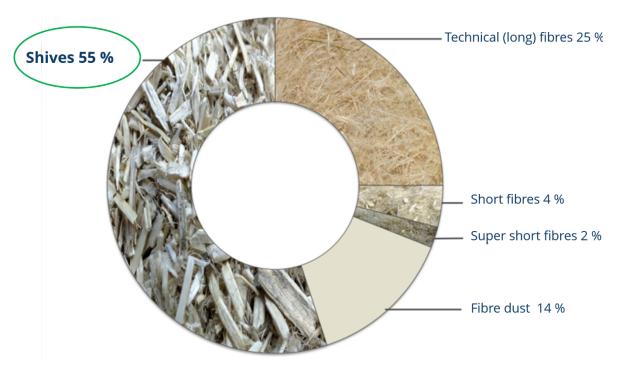


Figure 1: Typical composition of mechanical treated hemp straw [2]

Materials and methods

Characterization of raw material

In the frame of this project more than 20 hemp shive samples were investigated. The raw material was originated from various growing areas in Germany as well as in France and pre-treated in different ways.

For a first characterisation well known wet chemical methods were used. At first the dry content was determined. The following second step was an extraction in a 1:1-mixture of toluene and ethanol. Cellulose content (method by Kürschner and Hoffer, [5]), Klason-lignin content (method by Savard based on TAP-PI T 222 om-83, [6]) and holocellulose content (water-insoluble carbohydrates, method by Wise, [7]) were determined using the extracted samples. The value for hemicellulose content is corresponding to the difference between holocellulose and cellulose. In addition, the ash content at a temperature of 575 °C was determined for all samples.

Digestion

Laboratory tests (2-Liter-autoclave)

For the first tests the digestion of the shredded hemp shives took place in 2-Liter-autoclave with temperature and pressure control as shown in figure 2.



Figure 2: 2-Liter-autoclave (IPWC)

About 170 g of raw material was covered by a freshly prepared sodium hydroxide solution (3.5 - 4.0%) by weight). The liquor ratio, as consumed NaOH per dried raw material was 34% by weight. The optimal temperature ranged over 170 °C, the reaction time was 105 minutes, which leads to a H-Factor of about 1,500. The maximum process pressure increased to about 10 bar. For all tests the H-Factor as comparative value was calculated [8].

After finishing pulping, a multi-stage process of washing and bleaching steps followed. At first, the splinter content was separated. Characteristic is a washing step in 20% acetic acid (A) at the beginning, followed by bleaching applying 5% hydrogen peroxide (P) and repeating the acidic washing step(A). That means the optimal bleach sequence for the hemp shive pulp is A-P-A, carried out at a temperature of 85 °C. Finally, the pulp should be washed by deionized water to available a neutral pH-value.

Scale-up to 10-Liter-Digester

The first scale up took place in a 10-Liter-digester system with engineering and recirculation of alkaline solution control as shown in figure 3.



Figure 3: 10-Liter-digestor (IPWC)

For this digestion 800 g shives are necessary. In contrast to pulping using the autoclave a preheating of cooking liquor is possible as well as an active cooling. So, the optimal parameters are a little bit different. The preferred liquor ratio is 40% by weight and the optimal temperature is 170 °C, too. The reaction time should be 150 min. So, a H-Factor of about 2,500 is necessary for reaching the pursued DP.

Characterization of pulp

The results of digestion were checked by TITK during an iterative evaluation procedure. Most important are the complete solution of the pulp in Cuoxam, a DP in the suited range (550 to 650), low metal ion contents (heavy, alkaline and alkaline earth metals) as well as a high α -cellulose amount [8ff]. Beside other criteria a Kappa-number lower than 5 was pursued.

The Kappa-number was determined following an internal standard at IPWC in accordance with ISO 302:2015-08. The dry content was determined by examining the loss in mass of the samples after drying at 105 °C. Pulp samples as well as cellulose samples regenerated from the dopes and from the spinning tests dissolved in Cuoxam were characterised by capillary

viscometry for determination of the average degree of polymerization (Cuoxam-DP). The α -cellulose content was determined by investigation of the pulp amounts which are resistant to 17.5% sodium hydroxide solution at 20 °C.

The contents of heavy metal (Fe, Cu, Mn, Cr, Ni) as well as alkaline (Na, K) and earth alkaline- (Ca, Mg) ions were measured after microwave digestion according DIN EN ISO 11885 (E22) using ICP-OES. The ash content was determined after incineration at 900 °C.

Measurement of carboxyl group contents has been carried out by means of complexometric titration of zinc ions after removing of the metal ions from the cellulose at first and adding of zinc acetate solution in a second step. The carbonyl group contents were analysed by measurement of the absorbance at 530 nm after reaction with 2, 3, 5-triphenyltetrazoliumchloride solution.

The details of this cellulose characterisation were described in former publications. [9, 10]

Dope preparation and spinning tests

The preparation of cellulose dopes in small laboratory scale was carried out using a special vertical kneader system, linked with a RHEOCORD 9000 (HAAKE). Temperature, torque moment and revolutions per minute (rpm) vs. time were recorded on-line. The dopes were prepared, starting from an aqueous suspension of the treated pulp in 50 wt.-% aqueous NMMO, by removal of the excess water at elevated temperatures, higher shearing stress and low pressure during the dissolution processes (80-95 °C mass temperature, 800-40 mbar pressure, 5-20 rpm). 0.5 wt-% propylgallate, with regard to cellulose, were used for stabilisation of the NMMO solutions. After finishing of the excess water removal (achieving a NMMO monohydrate state), an after-dissolution kneading step (60 min, 90 °C mass temperature, 250 mbar) followed for homogenisation of the prepared dope.

An upscaling into 4 kg dope scale was carried out using planetary mixing machine PML 40 (Netzsch-Feinmahltechnik GmbH).

Small lab spinning tests were carried out by dry-wet spinning experiments for preparation of staple fibres of about 1.7 dtex fineness using a laboratory piston spinning equipment, which is described in former publication [11]. Spinning nozzles, containing 30 holes with capillary diameters of $100 \, \mu \text{m}$ were used for all laboratory spinning experiments. The spinning temperatures

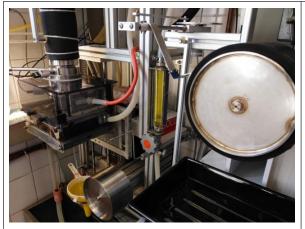
were selected in each case according to the determined rheological properties of the used cellulose dopes.

Further semi-technical spinning tests using spinnerets with 6 x 80 capillaries (90 μ m outlet diameter) were

carried out for investigation of the spinning behaviour and stability. These trials were used for preparation of staple fibres and multifilament samples.

The equipment for the tests is shown in table 1.

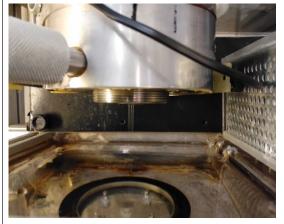
Table 1: Spinning equipment used for hemp shive pulp shaping



Small lab spinning equipment



Large lab spinning equipment



Small lab spinning nozzle (30 capillaries)



Large lab spinning nozzle (6 x 80 capillaries)

Results and discussions

Technical basics and requirements

Some selected and the average values of plant analytics are compiled in table 2.

The cellulose content in shives (< 50%) is significant lower than in technical long fibres (> 70%), but in the same range like in wood or other agricultural residues. The chemical composition is almost independent on place or region of cultivation. A short retting time should be preferred because the cellulose content is higher and the lignin can be separated at lower tem-

peratures and chemical consumption. For a good pulp quality also a satisfied separation from bast fibres, dust and fine content is reasonable. In the frame of our work we mainly used the mechanical pre-treated shives of the project partner FUDI, because they were available in sufficient quantity. Despite of the relative low cellulose content they had a good quality for pulping.

Optimization of digestion

Before optimisation the first challenge was to get a hemp shive based pulp using a sulphur free alkaline process. Compared with other agricultural residues the digestion requires harsher conditions; that means a

Table 2: Plant analysis of different hemp shives

Component/ region	Cellulose [%]	Hemicellulose [%]	Lignin [%]	Extraktives [%]	Ash content [%]
Brandenburg	43.9	33.6	16.4	2.8	1.6
Saxony	41.3	30.1	24.4	4.3	4.2
Mecklenburg	42.9	30.6	22.3	2.8	0.03
France	43.0	33.6	20.2	3.2	1.4
FUDI	37.3	34.5	21.7	4.2	1.6
range	37 - 46	30 - 34	16 - 25	2 - 7	< 0.1- 4

higher NaOH-concentration, higher temperatures and a longer cooking time are necessary. So the strived H-Factor is significant higher than below 1,000 as described in previous works [12, 13].

While in the 2-Liter-autoclave a H-Factor of 1,500 was optimal, in the 10-Liter-digestor a H-factor of 2,000 to 2,500 is required. Three digestions in this scale were required for manufacturing of larger pulp quantities for the large lab spinning test. The pulp could be used as a mixed sample. Therefore, a good reproducibility of pulping was notable.

As described above a three-step procedure for washing and bleaching after pulping is required for getting the target parameters. During the wash steps using de-ionized water and acetic acid (A) the metal ions were removed, target Kappa-number and DP could be adjusted via bleaching (P). Finally, all desired values were achieved.

The developed digestion and bleaching process permitted the preparation of hemp shives based pulps for Lyocell applications. The pulp parameters could be adapted for usage in fibre preparation by dry-wet spinning NMMO processes.

Dope preparation and spinning tests

Dope preparation and fibre spinning tests could be carried out successfully in both, small and large laboratory scale. The cellulose concentration used was in typical range of around 12%, also with regard to the rheological properties.

The prepared fibres and filaments showed well acceptable textile-physical properties, very comparable to industrially produced Lyocell fibres from wooden pulps [14].

Table 3: Hemp pulp properties

Parameter	Unit	Hemp pulp sample 1 2-Liter-autoclav	Hemp pulp sample 2 10-Liter-digestor
Cuoxam-DP		632	624
α-cellulose content	%	89.6	87.7
Carboxyl group content	μmol/g	n.m.	45.4
Carbonyl group content	μmol/g	13.6	18.8
Fe, Cu, Ni, Cr, Mn	ppm	14	45
Na, K	ppm	90 / 47	61 / 7
Mg, Ca	ppm	33 / 415	3 / 14
Ash content	%	< 0.1	n.m.

Table 4: Spinning dope and fibre properties using hemp shive based pulps

Dope characteristics	Unit	Small lab spinning test	Large lab spinning test	Lyocell fibres from wood pulp [14]
Zero shear viscosity (85°C)	Pas	10,620	13,270	
Cellulose concentration	%	11.6	11.9	
Fibre testing				
Fineness	dtex	1.8	1.7ª / 1.6 ^b	≤ 1.7
Fibre tenacity, cond.	cN/tex	36.0	39.7ª / 51.5b	40 - 42
Elongation, cond.	%	16.3	12.3ª / 7.2b	15 - 17
Loop tenacity	cN/tex	17.8	13.0 ^a / n.m. ^b	
Cuoxam-DP		623	560	560 - 620
Photos of prepared fibres / filaments				

^a staple fibre

Conclusions

The aim of the studies was to evaluate the potential of hemp shives in conversion to high purity dissolving pulp grades those could be used for manufacturing Lyohemp™ fibre and further processing into textiles Shives as raw materials were successfully investigated by a modified soda cooking process and additional pulp bleaching and washing steps. So organic impurities and metal salt concentration could be decreased down to those levels which were compatible to the Lyocell process requirements. After adjustment and optimization, the hemp shive pulps prepared from different laboratory scales could be well dissolved in NMMO monohydrate and prepared dopes exhibited

satisfying properties for air-gap spinning. The produced LyohempTM fibres proposed well sufficient mechanical properties for further textile processing. Yarns made of these fibres represent fine counts, high tenacity and low mass variation, which also benefit yarn dyeing and finishing procedures.

Unfortunately, a scale-up in technical standard could not be realized in the frame of this work, but it is assumed that manufactured Lyohemp $^{\text{TM}}$ fibres offer surprisingly good processing properties into yarns and fabrics as well as wearing and draping comfort in apparel application, too.

^b filament, single fibre testing

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