Tatjana Rijavec<sup>1</sup>, Svjetlana Janjić<sup>2</sup>, Darja Kocjan Ačko<sup>3</sup> <sup>1</sup>University of Ljubljana, Faculty of Natural Sciences and Engineering, Aškerčeva 12, 1000 Ljubljana, Slovenia <sup>2</sup>University of Banja Luka, Faculty of Technology, Vojvode Stepe Stepanovica 73, 78 000 Banja Luka, Republic of Srpska, Bosnia and Herzegovina <sup>3</sup>University of Ljubljana, Biotechnical Faculty, Jamnikarjeva 101, 1000 Ljubljana, Slovenia

Revitalization of Industrial Hemp *Cannabis sativa* L. var. *sativa* in Slovenia: a Study of Green Hemp Fibres

Oživljanje navadne konoplje Cannabis sativa L. var. sativa v Sloveniji: raziskava zelene konoplje

Original Scientific Article/Izvirni znanstveni članek Received/Prispelo 02-2017 • Accepted/Sprejeto 02-2017

# Abstract

The importance of industrial hemp as a source of highly valuable textile fibres is briefly presented through its use for textiles and composites and its increasing cultivated areas in the 21st century. On the territory of present Slovenia, about 160 ha of agricultural area was cultivated with hemp before WWII, then it quickly began to decline and at the end of the 1970s, it was no longer cultivated. Revitalization of industrial hemp in Slovenia with field experiments started already in the years 2000/2001 for producing seeds, whereas hemp fibres were used only as an insulation for buildings. The textile technological properties of hemp fibres from different varieties grown in Slovenia have not been examined till now. They are important for using hemp fibres in highly valuable textile products. The properties of green hemp fibres extracted mechanically from non-retted hemp stems of Cannabis sativa L. var. sativa (varieties: Novosadska, Juso-11, Bialobrzeskie, Unico-B and Beniko) were determined. All the analysed varieties except Beniko had stem height over 200 cm. The highest yield of green fibres was 33.1% (Novosadska). The analysed green fibres' content was 1.24–3.26% of ash, 7.77-8.50% of moisture regain, 10.69-13.92% of water-soluble substances and 8.45-10.83% of pectin. Through a biodegradation process of retting green hemp fibres in tap water at temperature 35°C, 9.01– 18.89% of dry mass was removed after ten days. Average linear density of green hemp fibres was very high, around 200 tex. Tenacity of fibres' bundles was in the range of 167–272 MPa, but tenacity of elementary fibres was 548-672 MPa. From the curves of specific stress-strain, it is seen that green hemp fibres from all five varieties had similar superstructure. All analysed green hemp fibres had high linear density and low mechanical properties. For textile application, they should be further processed into finer fibres in order to increase their tensile stress and become also more flexible and soft.

Keywords: Cannabis sativa, non-retted hemp fibres, green hemp fibres, hemp in Slovenia, revitalization of industrial hemp

# Izvleček

V članku sta na kratko predstavljena pomen navadne konoplje kot vira dragocenih tekstilnih vlaken za uporabo v tekstilstvu in v kompozitih in povečevanje kmetijskih površin, zasejanih z navadno konopljo v 21. stoletju. Še pred drugo svetovno vojno je bilo na ozemlju današnje Slovenije s konopljo zasejanih okrog 160 hektarov kmetijskih površin, te površine pa so potem začele hitro upadati in konec 70. let konoplje niso več gojili. Revitalizacija navadne konoplje v Sloveniji se je s poljskimi poskusi začela v letih 2000/2001 za pridelavo semen, pri čemer so se konopljena vlakna uporabljala le za toplotno izolacijo stanovanjskih hiš. Tekstilno tehnološke lastnosti konopljenih vlaken iz različnih sort navadne konoplje, ki so gojene na območju Slovenije, do zdaj še niso

Corresponding author/Korespondenčna avtorica: Assoc Prof DrSc Tatjana Rijavec Tel. +386 1 200 32 24 E-mail: tatjana.rijavec@ntf.uni-lj.si Tekstilec, 2017, **60**(1), 36-48 DOI: 10.14502/Tekstilec2017.60.36-48 bile proučevane. Pomembne so za izdelavo visokovrednih tekstilij. Lastnosti zelene konoplje, to je vlaken, pridobljenih z mehansko obdelavo negodenih stebel navadne konoplje Cannabis sativa L. var. sativa, so bile ugotovljene za sorte Novosadska, Juso-11, Bialobrzeskie, Unico-B in Beniko. Vse analizirane sorte, razen Beniko, so imele stebla visoka več kot 200 cm. Največji izkoristek zelenih vlaken je bil 33,1-odstoten (Novosadska). Analizirana zelena konoplja je vsebovala 1,24–3,26 % pepela, 7,77–8,50 % standardne vlage, 10,69–13,92 % v vodi topnih snovi in 8,45–10,83 % pektina. Po desetdnevni biorazgradnji vlaken zelene konoplje v vodovodni vodi pri temperaturi 35 °C se je odstranilo 9,01–18,89 % suhe snovi. Povprečna dolžinska masa zelene konoplje je bila zelo visoka, okrog 200 tex. Trdnost snopov konopljenih vlaken je bila v območju 167–272 MPa, trdnost elementarnih vlaken pa 548–672 MPa. Iz krivulj specifična napetost–raztezek je razvidno, da so imela vlakna zelene konoplje ne glede na sorto podobno nadmolekulsko strukturo. Ker imajo vlakna zelene konoplje iz vseh analiziranih sort visoko dolžinsko maso in slabe mehanske lastnosti, bi jih za uporabo v tekstilne namene morali z nadaljnjo obdelavo čim bolj razgraditi v finejša vlakna, s čimer bi se jim zvišala natezna trdnost, postala pa bi tudi bolj upogibljiva in mehka.

Ključne besede: Cannabis sativa, negodena konopljena vlakna, zelena konoplja, konoplja v Sloveniji, revitalizacija navadne konoplje

# 1 Introduction

Hemp (*Cannabis sativa* L. var. *sativa*) is an increasingly attractive industrial plant for food, textile and constructional industries because of its valuable seeds, fibres and shives. After removal of seeds, hemp stems (also called straw) remain as a cheap by-product, which can be technologically converted into highly valuable products. Dried hemp straw consists of about 28% of extremely long, strong bast fibres (textile fibres) and about 55% of short, woody fibres (called also hurds or shives) [1].

Bast fibres present a sclerenchyma supporting tissue of stems. A hemp stem cross-section with primary (main) and secondary fibres' layers is presented in Fig. 1a. Primary and secondary fibres' layers (Fig. 1b) consist of dead elongated plant cells (elementary fibres) bonded together into bundles (technical fibres) (Fig. 1c) with intermediate cells containing pectin and a small amount of lignin, usually called middle lamella [2]. Fibres' bundles, running from the root to the top of a stem, are around 1500–2000 mm long and have 10–40 elementary fibres in cross section [3].

Dry non-retted hemp stems (green hemp) can be mechanically separated into bark and shives. Since bast fibres in bark contain a preserved whole middle lamella, they are very rough and stiff and should be softened for further uses. On the contrary, fibres from retted hemp stems (dew, water, tank, enzyme retting) are softer and more flexible, because pectin in middle lamella degrades through retting [6].

## 1.1 Use of hemp bast fibres in textiles

Hemp is a plant of genus *Cannabis*, which originates from India and Central Asia [7]. In ancient times,



Figure 1: Cross-section of hemp stem and fibres: a) cross-section of a hemp stem with a bark tissue (A), a woody part (B) and a pith (C) [4]; b) fragment of cross-section of hemp stem with a primary (main) fibre layer (F), a discontinued secondary fibre layer (f) and a woody tissue (B) [5]; c) cross-section of hemp fibres' bundle (a) and a longitudinal view of elementary fibres (b) [5].

hemp was regarded more as a source of drug than as a textile fibre plant. In a Neolithic site Yuan-Shanu (today Taiwan), the remains of hemp ribbons dated from 10,000 BC were found [8]. In 2,800 BC, the Chinese emperor Sei Nung ordered the cultivation of cannabis for the purpose of producing fabrics [5, 9]. In 500 BC, the Scythians used hemp fibres for cordages [5]. The Scythians also brought hemp to Europe [7]. In the 15<sup>th</sup> century, hemp was transferred from Europe to America [10], where later, in the 19th century, one of the most popular trousers was made from hemp fabric (sail cloth). Those trousers were patented by Jacob Davis and Levi Strauss in 1873 [11]. Despite economic success, a prohibition of industrial hemp cultivation was enforced in the USA in 1937. It coincided with the emergence of the first synthetic fibre, nylon 6.6. The prohibition of cultivating industrial hemp was later enforced in other parts of the world.

Hemp bast fibres can be exploited for textiles, papers, composites and biomass materials. Different fine woven and knitted fabrics for apparel, denim, decoration fabrics for interior, cordages (strings, twines, cords, ropes), nets, canvas, carpets, insulation etc. can be produced from hemp bast fibres [3]. New uses of hemp fibres include geotextiles [12], composites (NFC, natural fibres composites) [13, 14] and biocomposites [15, 16] with biodegradable matrices, like polylactide resins [17], wheat gluten plastics [18] and biobased linear low density polyethylene [19, 10].

#### 1.2 Industrial hemp in the 21<sup>st</sup> century

Only industrial hemp (*Cannabis sativa* L. var. *sativa*), which contains very low percentage of psychoactive drug delta-9-tetrahydrocannabinol (delta9-THC) and is not suitable for medicinal or recreational uses, is today allowed for cultivation [20, 21]. In the European Union, the cultivation of industrial hemp varieties with up to 0.2% THC content in dry mass is allowed [22], in Canada, the limit is up to 0.3% [23], but in Switzerland, it is even up to 1% of THC [24]. The cultivation of industrial hemp is today still illegal in most parts of the USA [23]. Only about thirty countries in the world legally cultivate industrial hemp [1, 25]. The world's largest producers of industrial hemp are China, Canada and France.

The global harvested area for industrial hemp was about 85.000 hectares in the year 2011, approximately 60,000 hectares (70.6%) for fibres and 25,000 hectares for seeds [26]. The production of hemp fibres in the year 2011 was around 77,000 tons [25]. In the European Union, 25,224 hectares of cultivated areas were sown with industrial hemp in the year 2015, with almost half of the planted areas located in France (Fig. 2) [27].



*Figure 2: Cultivated areas with industrial hemp in the European Union in 2015 [27]* 

## 1.3 Industrial hemp in Slovenia

The beginnings of cultivation of industrial hemp on the territory of present Slovenia go back at least 400 years [28] when hemp fibres were processed mainly into cordages and woven fabrics. In the year 1939, industrial hemp was planted on over 160 hectares of land, after the WWII, the cultivation was gradually abandoned to be completely terminated in the 1970s (Fig. 3).



Figure 3: Cultivated areas with industrial hemp and stem yields in the period from 1939 to 1980 in the territory of present Slovenia [29, 30]

Revitalization of industrial hemp in Slovenia started in the years 2000 and 2001 by the Department of Agronomy at Biotechnical Faculty at the University of Ljubljana in cooperation with an incentive development centre in Murska Sobota (Spodbujevalni razvojni center v Splošni kmetijski zadrugi Murska Sobota) [31]. In field experiments in Markiševci near Murska Sobota [20, 32], several varieties from different provenances were included: from Poland (Beniko, Bialobrzeskie), Hungary (Unico-B), Ukraine (Juso-11), and Serbia (Novosadska), which all were in the years 2000 and 2001 in the European List of Varieties except the variety Novosadska, which has never been included into the European List of Varieties. In the year 2011, the same hemp varieties were seeded in a field experiment on the location of the Department of Agronomy at Biotechnical Faculty at the University of Ljubljana. In all three experiments, a seeding technology for fibres was used. The results of an average yield of stem from all three experiments are given in Fig. 4. The experiments in 2000 and 2001 showed a statistically significant influence of a variety on the yield of stem [33]: Novosadska and Unico-B proved to be the best varieties with an average yield of stem of 7,164 kg/ha, but the varieties Bialobrzeskie and Beniko proved to be the worst with 20-30% lower yield of stem per hectare. The experiments also show that in wet environment (in the year 2000, rainfall was more intense in the beginning of the growing period than in the year 2001 [31]) all the varieties performed much better than in dry environment: the average yield of stem in the year 2000 was by 30-69% higher than in the year 2001. The variety Novosadska performed the best in wet weather conditions, but in dry weather conditions, the variety Unico-B performed the best.

The average yield of stem in the experiment 2011 in Ljubljana was by 9–29% lower than in the year 2000 in Markiševci, but in comparison with the average yield in the year 2001 in Markiševci, it was by 24–63% higher (Fig. 4). From the average values of yield of stem of all three years, it is seen that the variety Novosadska gave the highest yield with 7,164 kg/ha, followed by Unico-B with 6,599 kg/ha, Juso-11 with 6,094 kg/ha and Bialobrzeskie with 5,608 kg/ha and Beniko, which gave only 4,823 kg/ha.

Current legislation in Slovenia enables anyone who has a minimum cultivated area of 0.1 hectare to be engaged into the cultivation of industrial hemp [22]. In 2016, four hundred forty-five registered farmers in Slovenia cultivated industrial hemp on 360 hectares (Fig. 5), which represented about 3% of all production area in Slovenia. Industrial hemp in Slovenia is mainly cultivated for seeds, which brings farmers the main income, but straw remains mostly unused [34].



Figure 4: Average yield of stem of industrial hemp varieties grown in main crop in the years 2000 and 2001 in Markiševci [33] and in the year 2011 in Ljubljana



*Figure 5: Cultivated areas of industrial hemp in Slovenia in the 21<sup>st</sup> century [29]* 

In Slovenia, like in other countries in the European Union, all the varieties of industrial hemp that are included in the European List of Varieties are allowed for sowing. In the year 2016, there were 54 authorized varieties of *Cannabis sativa* L. – Hemp [35]. Slovenian autochthonous varieties of industrial hemp are not included in the European List of Varieties, because their content of THC exceeds the allowed values of 0.2% [36].

In the year 2016, the following sowing varieties of industrial hemp were introduced in Slovenia: one Ukrainian variety Uso-31, two French varieties (Fedora 17 and Santhica 27), and four Hungarian varieties (KC Dora, Kompolti hibrid TC, Monoica and Tisza) [37].

By using genetic engineering, the diversity of varieties has increased significantly which influences the authorized varieties that change all the time. This puts the farmers and processors in front of a number of issues regarding the suitability of cultivation in a given territory and yield of plants. Hemp varieties have different ability to adapt to growing conditions (weather conditions, soil, harvest time etc.), which influences stem and fibre yields and their quality.

The cultivation of industrial hemp in Slovenia is mainly intended for the production of seeds and therefore, non-retted hemp stems are available as a by-product. The aim of our study was to determine the properties of green hemp bast fibres obtained from non-retted stems, which are not available in professional literature.

## 2 Experimental part

## 2.1 Materials

Stems of five industrial hemp varieties Bialobrzeskie, Beniko, Unico-B, Juso 11, and Novosadska<sup>1</sup> from the field experiment in Ljubljana in the year 2011 (Fig. 4) were included in the experiment<sup>2</sup>. Each variety was sown on the area of  $2 \times 5$  m<sup>2</sup>, with a seed quantity 25 kg/ha. Hemp seeds were

<sup>2</sup> Experimental field with a medium-deep silty-clay soil was arranged with installed drainage system. The soil was medium stocked with macro nutrients. The field was plowed and aligned seedbed. Sowing was carried out on 20 April 2011 on a plowed and aligned seedbad. Hemp was cultivated without adding nutrients and pesticides. The plants were healthy, with no infections or injuries to pathogens and pests. Young crops were dug and later individual weeds were manually pulled.

sown in a row at distance of 20 cm, a row distance from each other in a series was 10 cm, which quite restricts plants spatially for growing branches only at the top of stems. Precipitation in the year 2011 was lower in spring and considerably higher in autumn than the average of the last thirty years; the temperatures were slightly higher than the average of the last thirty years (Fig. 6).

Growing of hemp crop was initially poor due to drought in the spring months. It improved in May to July due to higher rainfall. In the late summer, the weather was dry. At the beginning of autumn, the growth stopped and the seeds developed.



*Figure 6: Precipitation and temperatures in Ljubljana in the year 2011* 

From each hemp variety, ten whole stems were selected for analyses. Dried non-retted stems were, after harvesting seeds and removing leaves, decortificated by beating with a plastic hammer. Green hemp fibres (Fig. 7a) were manually separated from broken wooden core (shives) (Fig. 7b).



Green hemp fibres

Hemp shives

*Figure 7: Mechanically treated dry stems were separated into green hemp fibres and broken wooden core (hemp shives)* 

#### 2.3 Methods

Volume density of a stem ( $\rho_s$ ) was calculated according to Eq. 1, where a stem volume ( $V_s$ ) according to

<sup>&</sup>lt;sup>1</sup> The varieties Bialobrzeskie and Beniko originate from Poland, from the Institute for Natural Fibers in Poznan. Compared with the variety Unico-B, which is a Hungarian variety of GATE Institute - Rudolf Fleischmann (Agricultural Research Institute), the varieties Bialobrzeskie and Beniko flower 3 to 4 weeks earlier and reach maturity at least 14 days in advance. Juso 11 is a Ukrainian variety (the Institute of Bast Crops). The variety was obtained by hybridisation of Dneprovskaya 4, Juso 21 and Dneprovskaya Odnodomnaya 6. It was registered in 1984 in the former Soviet Union. A Serbian variety Novosadska originates from the Institut za ratarstvo i povrtarstvo in Novi Sad. The variety was bred in the middle of the 20th century based on Flajšman hemp. Officially, it was recognized as Novosadska hemp in 1989. It is known for its large crop of stems and seeds.

Eq. 2 was simplified as a truncated cone with height  $(H_s)$ , thickness in the bottom  $(2R = d_1)$  and thickness at the top  $(2r = d_2)$ :

$$\rho_s = \frac{m_s}{V_s} \, \text{kg/m}^3 \tag{1}$$

$$V_s = \frac{1}{3} \pi H \left[ Rr + R^2 + r^2 \right]$$
 (2).

Yield of green hemp fibres per stem ( $\eta_f$ ) was calculated from the ratio of dry mass of extracted green hemp fibres ( $m_f$ ) to dry mass of stem ( $m_s$ ) (Eq. 3):

$$\eta_f = \frac{m_f}{m_s} \times 100 \,(\%) \tag{3}$$

Tensile properties of green hemp fibres were determined on a dynamometer Instron 5567 (Instron, GB) at temperature 25 °C and relative humidity 60%. The initial length of samples was 100 mm (at testing velocity 50 mm/min), and 5 mm (at testing velocity 0.3 mm/min). For each hemp variety, 20 measurements were done.

Microphotographs of green hemp fibres were taken on a stereomicroscope Nikon SM Z800 (Nikon Instruments).

Moisture regain of green hemp fibres was determined according to standard SIST EN ISO 139:2005 Textiles – Standard atmospheres for conditioning and testing.

Ash content was defined after burning and heat treatment of green hemp fibres. The samples were burned in ceramic bowls in open air and the remaining ash was heat treated for three hours in a furnace at temperature of 600 °C.

To remove water-soluble substances, green hemp fibres were boiled one hour in distilled water. To remove pectin matter, green hemp fibres were boiled one hour in a one percent aqueous solution of ammonium oxalate  $(NH_4)_2C_2O_4$  [38].

## 3 Results and discussion

#### 3.1 Hemp stems

The average stem height of analysed varieties was over 200 cm, only the variety Beniko was significantly shorter, i.e. 137.2 cm (Tab. 1). The stem height of hemp from the experiments in Ljubljana in the year 2011 was on the average by 6 to 20% lower than that from the experiment in Markišavci in the year 2001 (Fig. 8). The exception was the variety Juso-11, the stem height of which was equal in both locations.

The hemp varieties differed significantly from each other in stem thickness (Tab. 1): at the bottom of stems, the thickness was in the range of 6.8-25.4 mm, at the top of stems, it was 2.5-4.65 mm.

Batra [39] specified the best hemp stems for fibres production as those with height over 200 cm and diameter over 0.5 cm. It can be concluded that all the varieties included in the experiment met these criteria except the variety Beniko the stems of which were too low. For the variety Beniko, Jankauskienė [40] reported that in the experiments in Lithuania in the year 2006, their stems reached an average height of 213 cm. These experiments confirm again the importance of environmental factors and quality of soil on the yield of industrial hemp.

An average mass of air-dried hemp stems varied from 17.6 g to 37.8 g per stem (Tab. 1) because the varieties had different volume densities. Stems with lower volume densities had larger pith. Pith converted into lumen by drying and the space filled with air. It influenced the volume density of hemp stems, which was in the range from 27.5 kg/m<sup>3</sup> (Novosadska) to 218.1 kg/m<sup>3</sup> (Beniko). The stems with lower volume density were more hollow and as such more suitable as a potential thermal insulation material than the stems with higher volume densities.

Table 1: Properties of hemp stems (mean  $\pm$  error of the mean values): height of a stem (H<sub>s</sub>), thickness at the bottom (d<sub>1,s</sub>) and at the top (d<sub>2,s</sub>) of a stem, mass (m<sub>s</sub>) and average volume density ( $\rho_s$ ) per stem

Hemp variety	$H_{\rm s}[{\rm cm}]$	$d_{1,s} [{\rm mm}]$	$d_{2,s} [{\rm mm}]$	<i>m</i> <sub>s</sub> [g]	$ ho_{s}$ [kg/m <sup>3</sup> ]	
Novosadska	204.0±8.3	25.4±9.6	4.65±0.6	34.84±4.7	27.5	
Juso-11	216.0±11.7	17.8±6.6	4.3±0.7	37.77±6.0	50.2	
Bialobrzeskie	201.9±11.0	9.6±0.7	2.5±0.3	30.73±4.8	106.9	
Unico-B	201.8±8.7	10.2±0.5	4.4±0.7	31.82±3.6	112.7	
Beniko	137.2±15.3	6.8±1.1	2.5±0.7	17.62±6.9	218.1	

Revitalization of Industrial Hemp Cannabis sativa L. var. sativa in Slovenia: a Study of Green Hemp Fibres



Figure 8: Comparison of hemp stem height from the experiments in Ljubljana in the year 2011 with those from the experiment in Markišavci in the year 2001 [33]

## 3.2 Green hemp fibres

3.2.1 Average yield of green hemp fibres per stem The highest average yield of green hemp fibres per stem of 33.1% was determined for the variety Novosadska. For the varieties Juso-11, Unico-B and Beniko, it was lower, i.e. about 31% (Tab. 2), but for the variety Bialobrzeskie, the lowest average yield of green hemp fibres per stem of 22.5% was determined because of its large branching stems.

For unbranched stems, a high linear correlation exists between the mass of stems and the mass of green hemp fibres (correlation coefficient  $R^2 = +0.9889$ ).

*Table 2: Mass of air dried green hemp fibres*  $(m_f)$  *and its yield per stem*  $(\eta_f)$  *(mean*±*error of the mean values)* 

Hemp variety	<i>m</i> <sub>f</sub> [g]	$\eta_{ m f}$ [wt.%]	
Novosadska	11.54±3.8	33.1	
Juso-11	11.75±4.0	31.1	
Bialobrzeskie	6.92±1.9	22.5	
Unico-B	10.10±3.2	31.7	
Beniko	5.48±1.5	31.1	



Figure 9: Longitudinal view of green hemp fibres in a stereomicroscope: (a) non-retted stem; (b) one hour in distilled water at boiling; (c, d) one hour in 1% solution of ammonium oxalate at boiling; (e, f) ten days in tap water at 35 °C. Fibres in Fig. 9d–9f were gently mechanically treated with a needle before microscopy. Microscope magnification: 12.6-x. (1 – cuticle, 2 – remains of non-fibrous cortex material, 3 – destroyed remains of non-fibrous cortex material)

Tekstilec, 2017, 60(1), 36-48

#### 3.2.2 Longitudinal view of green hemp fibres

Non-retted green hemp fibres (Fig. 9a) are rough and of different thickness, which formed at peeling of stalks when the stem bark was mechanically disintegrated. On the darker fibres in Fig. 9a, a stem cuticle is seen. In Fig. 9a-9c, some remains are seen of non-fibrous cortex material<sup>3</sup>, which is firmly connected with fibres and has not been removed by the treatment in boiling water or in a solution of ammonium oxalate, but was destroyed through microbiological retting as it is seen in Fig. 9e and 9f. Thick fibres were mechanically easily split longitudinally into finer fibres' bundles after treatment in ammonium oxalate (Fig. 9d) and after ten days retting in tap water of 35 °C (Fig. 9e and 9f) and also after treatment. Hemp fibres' bundles are composed of elementary fibres, which are bound to each other with pectin and lignin. Pectin can be removed through microbiological retting or by boiling in a 1% solution of ammonium oxalate, whereas lignin is hardly destroyed in these procedures.

#### 3.3.3 Chemical properties of green hemp fibres

Hemp fibres consist of cellulose (74–78%), hemicellulose (4–20%), lignin (2–11%), water and solvent soluble substances, wax, ash, pectin, protein and water [42].

The content of water-soluble substances of absolutely dry green hemp fibres (Tab. 3) was in the range of 13.10–13.92% for the varieties Juso-11, Bialobrzeskie and Beniko; for the varieties Novosadska and Unico-B, it was lower, 10.69% and 11.57%. The content of the removed pectin after the treatment for one hour in a 1% solution of ammonium oxalate was in the range of 8.49–10.83%. In biodegradation process of ten days of retting in tap water at temperature 35°C 9.01–18.89% of dry mass, mainly pectin, was removed (Tab. 3). The reasons for great differences should be specifically examined. The moisture regain was in the range of 7.77–8.50%. The content of ash, which represents a quantity of inorganic substances in fibres, was in the range of 1.24–3.26%.

In absolutely dry green hemp fibres, 21.5% non-fibrous material (water-soluble substances and pectin) and 78.5% fibres were determined on average.

# 3.3.4 Physical-mechanical properties of green hemp fibres

Linear density (Fig. 10) of each fibre was measured before measuring its tensile properties (Tab. 4). Average linear density of measured green hemp fibres was in the range from 149.8 tex (Juso-11) to 220.5 tex (Beniko) with a high coefficient of variation, between 27% and 52%.



Figure 10: Linear density of green hemp fibres

Hemp variety	Ammonium oxalate soluble substances [%]	Water-soluble substances [%]	Removed substances at retting in water at 35°C [%]	Ash [%]	Moisture regain [%]	
Novosadska	8.49	10.69	9.01	2.71	8.02	
Juso-11	9.52	13.92	16.49	3.26	8.19	
Bialobrzeskie	9.89	13.71	18.86	2.68	7.77	
Unico-B	8.76	11.57	18.89	1.24	8.5	
Beniko	10.83	13.10	14.12	3.12	8.07	

Table 3: Chemical properties of green hemp fibres

tion is well described in literature [43].

<sup>3</sup> Botanical explanation of dicotyledonous stem constitu-

Tensile properties of hemp fibres' bundles depend on initial length of a fibre, clamped between two grips of a dynamometer before testing. Smaller initial lengths result in higher tenacities [44]. Tensile properties of green hemp fibres were studied at two different initial length, 100 mm and 5 mm. Breaking force measured at initial length of 100 mm was in the range of 17.4–29.8 N, but at initial length of 5 mm, it was 2.7–4.8-times higher, in the range of 80.5–96.2 N. Specific breaking stress of green hemp fibres measured at initial length of 100 mm was 167–272 MPa, but at initial length of 5 mm, it was 548–672 MPa, i.e. 2.5–4-times higher than at initial length of 100 mm (Tab. 4).

The differences in average breaking stress measured at initial length of 100 mm varied a lot between the varieties: the strongest variety was Juso-11, the weakest Bialobrzeskie green hemp fibres. Tensile stress measured at initial length of 5 mm was for all varieties very similar except for Beniko the tensile stress of which was the lowest.

Because the length of elementary hemp fibres is only 50–55 mm [45] or less, tensile properties measured at initial length of 100 mm demonstrate primarily the tenacity of links between hemp fibres and pectin lamella which glues elementary hemp fibres together into bundles. Much higher tenacity measured at initial length of 5 mm demonstrates the tenacity of elementary fibres, and depends on the strength of cellulose fibrils that form elementary fibres.

Breaking elongation (Tab. 4) measured at initial length of 100 mm was in the range 0.9–1.0% and confirms a brittle nature of green hemp fibres. Breaking elongation measured at initial length of 5 mm was in the range of 5.8–7.2%. It is typical for elementary hemp fibres, which are much tougher fibres than green hemp fibres.

*Table 4: Breaking force* ( $F_{br,f}$ ), specific breaking stress ( $\sigma_{pr,f}$ ), breaking elongation ( $\varepsilon_{br,f}$ ) (mean±error of the mean values) of green hemp fibres

	Initial length = 100 mm			Initial length = 5 mm				
Hemp variety	F <sub>br, f</sub> [N]	$\sigma_{\rm pr, f}$ [cN/tex]	σ <sub>pr, f</sub> [MPa]	€ <sub>br, f</sub> [%]	F <sub>br, f</sub> [N]	$\sigma_{\rm pr, f}$ [cN/tex]	σ <sub>pr, f</sub> [MPa]	ε <sub>br, f</sub> [%]
Novosadska	20.03±2.9	12.7	191	0.9±0.1	96.03±8.4	42.4	636	6.6±0.6
Juso-11	27.76±4.2	18.1	272	1.0±0.1	96.24±9.4	44.6	669	7.2±0.6
Bialobrzeskie	17.44±2.4	11.1	167	1.0±0.1	85.43±6.2	44.8	672	6.1±0.5
Unico-B	29.77±3.6	15.3	230	1.0±0.1	80.46±8.8	44.5	668	5.8±0.9
Beniko	27.25±4.6	14.0	210	1.0±0.1	93.11±12.5	36.5	548	6.0±0.4



*Figure 11: The correlation between (a) breaking force and linear density and (b) between breaking stress and linear density of green hemp fibres (all measured at initial length of 100 mm)* 

Tekstilec, 2017, 60(1), 36-48

Duval et al. [46] confirmed that a diameter of a fibre near its rupture point influenced the hemp fibre tensile strength: a general observation was that tensile strength decreased with increasing fibre diameter. This interdependence was confirmed also by Shahzad [15] who attributed such behaviour to the amount of flaws in the fibres (kinks, dislocations [2]) and to the number of elementary fibres which decreased with lower diameters of fibres and resulted in the increase of tensile properties of fibres. In our experiment, a correlation between tensile force and linear density  $(R^2 = 0.330251)$  and also between tensile stress and linear density ( $R^2 = -0.17197$ ) (Fig. 11) of green hemp fibres was low and showed only a weak tendency to the interdependence between the studied properties. The curves of specific stress/strain of green hemp fibres from the tensile test at initial length of 100 mm (Fig. 12a) are almost linear, which means that the tensile deformation of fibres was near elastic in the range of less than 1%. The variety Juso 11 with the steepest curve resists to strain the most and has the highest modulus of elasticity, the variety Bialobrzeskie with the least steep curve has the lowest modulus of elasticity.

On the contrary, the curves made on fibres at initial length of 5 mm (Fig. 12b) show linear behaviour up to strain of about 2% where helically packed fibrils in S2 layer at an angle of  $5-30^{\circ}$  [41] to the fibre axis align along the fibres' axis. After the alignment of fibrils is finished, the fibres' resistance to tensile force increases suddenly and the stress/strain curve bends towards the ordinate, which causes a deflection of the curves. With increasing tensile force, the fibres show again an elastic deformation till they break at the end.

## 4 Conclusion

The quality and yield of hemp fibres depend on many factors, which include hemp variety, growing conditions (growing plan, temperature, raining/watering, plant nutrients in soil, pests etc.), the way of retting of stems and the way of extracting fibres from stems. Retting facilitates the extraction of bast fibres from stems, but it usually causes the deterioration of textile fibres' mechanical properties [47].

In our study, the following properties of green hemp fibres extracted mechanically from non-retted stems were determined:

- analysed varieties except Bialobrzeskie had a comparable stem height and quantity of green fibres, but they differed in volume density, which was for Novosadska only 27.5 kg/m<sup>3</sup>, but for Beniko, it amounted to 218 kg/m<sup>3</sup>;
- analysed green hemp fibres differed in content of ash, but Unico B and Novosadska deviated in lower content of pectin and water soluble substances;
- average linear density of green hemp fibres was very high, around 200 tex;
- tenacity of fibres' bundles was comparable with literature [42], i.e. between 167 MPa (Bialobrzeskie) and 272 MPa (Juso-11);
- tenacity of elementary fibres was between 548 MPa (Beniko) and 672 MPa (Bialobrzeskie), which is typical for hemp elementary fibres;
- curves of specific stress-strain of fibres from all five varieties are similar, which means that fibres from different varieties have similar superstructure.



*Figure 12: Average stress-strain curves of green hemp fibres bundles, measured at initial length of (a) 100 mm and (b) 5 mm* 

Tekstilec, 2017, **60**(1), 36-48

Green hemp fibres from all analysed varieties have high linear density and low mechanical properties. They can be used for technical applications, like ropes and geotextiles. For textile application, they should be further processed into finer fibres (e.g. retted, treated in autoclave with alkalis – cotonisation) to increase their specific tensile stress, flexibility and softness.

## References

- CARUS, Michael. EIHA. European Industrial Hemp Association. Market data on industrial hemp – fibres, shivs and seeds. October 2013 [online] [accessed 20.10.2016]. Available on World Wide Web: <a href="http://www.fibrafp7.net/">http://www.fibrafp7.net/</a> portals/0/michael\_carus\_day\_1.pdf>.
- FAN, Mizi. Characterization and performance of elementary hmp fibres: factors influencing tensile strength. *BioResources*, 2010, 5(4), 2307–2322.
- SPONNER, J., TOTH, L., CZIGER, S., FRANC, RR. Hemp. In *Bast and other plant fibres*. Edited by Robert R. Franck. Boca Raton [etc.] : The Textile Institute ; CRC Press ; Cambridge : Woodhead, 2005, 176–206.
- WOODHOUSE, Thomas, KILGOUR, P. Cordage and cordage hemp and fibers. Isaac Pitman & Sons, 1919, 12–15 [online] [accessed 14.01.2017]. Available on World Wide Web: <a href="http://hempology.org/ALL%20HISTORY%20ARTICLES">http://hempology.org/ALL%20HISTORY%20ARTICLES. HTML/1919%20CORDAGE%20HEMP.html>.</a>
- MERRITT, Matthews Joseph. *The textile fibers. Their physical, microscopical and chemical properties.* Forth edition. New York : John Willey & Sons, London : Chapman & Hall, 1924, 2.
- 6. SUNG, Wing L., WOOD, Mark, HUANG, Fang. Extraction of hemp fibers. *United States Patent Application Publication*, US 2010/0147472 A1, *Jun. 17*, 2010.
- KOČEVAR, Franjo. *Tekstilne surovine*. Ljubljana : Državna založba, 1956, 126.
- ROBINSON, Rowan. Velika knjiga o konoplji. Ljubljana : A. Urbančič, 2000, 54.
- 9. ARSENIJEVIĆ, Miro L. *Tekstilna vlakna*. Beograd : Naučna knjiga, 1965, 71.
- SINGH, Sukhdeep, DEEPAK, Dharmpal, AG-GARWAL, Lakshya, GUPTA, VK. Tensile and flexural behaviour of hemp fiber reinforced virgin-recycled HDPE matrix composites. *Procedia Materials Science*, 2014, 6, 1696–1702, doi: 10.1016/j.mspro.2014.07.155.

- 11. DAVIS, Jacob W. Improvement in fastening pocket-openings. US Patent 139,121, 20 May 1873.
- LEAO, A. L., CHERIAN, B., M., DE SOUZA, S. F., KOZLOWSKI, R. M., THOMAS, S., KOT-TAISAMY, M. Natural fibres for geotextiles. In *Handbook of natural fibres. Volume 2: processing and applications*. Edited by R. M. Kozlowski. Oxford et al. : The Textile Institute, Woodhead Publishing, 2012, 280–311.
- HRISTIAN, L., OSTAFE, M. M., MANEA, L. R., LEON, A. L. The study about the use of the natural fibres in composite materials. *IOP Conference Series: Materials Science and Engineer ing*, 2016, **145**, 032004.
- BECKERMANN, Gareth. Performance of hempfibre reinforced polypropylene composite materials : PhD thesis. Hamilton : The University of Waikato, 2007.
- SHAHZAD, Asim. A study in physical and mechanical properties of hemp fibres. *Advances in Materials Science and Engineering*, 2013, article ID 325085, 9 pages, doi: 10.1155/2013/325085.
- TAMBYRAJAH, Dilip. Indulge and explore natural fiber composites. *An invitation to product designers*. March 2015 [online] [accessed 20. 10. 2016]. Available on World Wide Web: <a href="http://eiha.org/media/2015/05/Natural-Fiber-Composites-for-designers-book-by-Dilip-Tambyrajah-March-2015.pdf">http://eiha.org/media/2015/05/Natural-Fiber-Composites-for-designers-book-by-Dilip-Tambyrajah-March-2015.pdf</a>>.
- SAWPAN, Moyeenuddin Ahmad. Mechanical performance of industrial hemp fibre reinforced polylactide and unsaturated polyester composites : PhD thesis. The University of Waikato, Hamilton, New Zealand, October 2009 [online] [accessed 20.10.2016]. Available on World Wide Web: <a href="http://research.commons.waikato.ac.nz/">http://research.commons.waikato.ac.nz/</a> handle/10289/3974>.
- WRETFORS, C., CHO, SW., HEDENQVIST, MS., MARTTILA, S., NIMMERMARK, S., JO-HANSSON, E. Use of industrial hemp fibers to reinforce wheat gluten plastics. *Journal of Polymers and the Environment*, 2009, 17, 259–266, doi: 10.1007/s10924-009-0147-6.
- KUKLE, S., VIDZICKIS, R., ZELCA, Z., BE-LAKOVA, D., KAJAKS, J. Influence of hemp fibers pre-processing on low density polyethylene matrix composites properties. 3rd International Conference on Competitive Materials and Technology Processes (IC-CMTP3) IOP Publishing IOP Conf. Series: Materials Science

and Engineering 123, 2016, doi:10.1088/1757-899X/123/1/01202.

- MALEK, Aljaž. Morfološke in nekatere gospodarsko pomembne lastnosti navadne konoplje (Cannabis Sativa L.) : diplomski projekt. Ljubljana : Univerza v Ljubljani, Biotehniška fakulteta, 2010, 3–7.
- HILLING, Karel W., MAHLBERG, Paul G. A chemotaxonomic analysis of cannabinoid variation in Cannabis (Cannabaceae). *American Journal of Botany*, 2004, **91**(6), 966–975, doi: 10.3732/ajb.91.6.966.
- 22. Pravilnik o pogojih za pridobitev dovoljenja za gojenje konoplje in maka. *Uradni list RS*, 205, 36/15, 25. 5. 2015.
- 23. JOHNSON, Renée. Hemp as an agricultural commodity. CRS Report, February 2, 2015. Congressional Research Service 2013 [online] [accessed 20.10.2016]. Available on World Wide Web: <a href="https://www.google.si/url?sa=t&rct=j&q=&esrc=s&source=web&cd=2&ved=0ahUKEwid9dGo99nPAhWqAMAKHbdjDdMQFgglMAE&url=https%3A%2F%2Fwww.fas.org%2Fsgp%2Fcrs%2Fmisc%2FRL32725.pdf&usg=AFQjCNHhGEaohsUt5WJSgyO66Oootm-7mg>.</a>
- 24. Ordonnance du DFI sur les tableaux des stupéfiants, des substances psychotropes, des précurseurs et des adjuvants chimiques (Ordonnance sur les tableaux des stupéfiants, OTStup-DFI) du 30 mai 2011 [online] [accessed 20.10.2016]. Available on World Wide Web: <a href="https://www.admin.ch/opc/fr/officialcompilation/2011/2595.pdf">https://www.admin.ch/opc/fr/officialcompilation/2011/2595.pdf</a>>.
- 25. Countries growing hemp. Hemp University. Learn what industrial hemp is and how to grow hemp [online] [accessed 20.10.2016]. Available on World Wide Web: <a href="http://www.hempuniversity">http://www.hempuniversity</a>. com/hemp-university/growing-hemp/countriesgrowing-hemp/#US>.
- 26. Top worldwide growth in industrial hemp fibres, shivs, seed & oil and pharmaceuticals.
  28. Maj 2014. *Bio-based news* [online] [accessed 20. 10. 2016]. Available on World Wide Web: <a href="http://news.bio-based.eu/worldwide-growth-industrial-hemp-fibres-shivs-seed-oil-pharmaceuticals/">http://news.bio-based.eu/worldwide-growth-industrial-hemp-fibres-shivs-seed-oil-pharmaceuticals/</a>>.
- 27. Total hemp cultivated area in Europe. EIHA 2015 [online] [accessed 20.10.2016]. Available on World Wide Web: <a href="http://eiha.org/media/2014/10/15-08-24-Industrial-Hemp-Cultivation-Area-2015-EIHA1.pdf">http://eiha.org/media/2014/10/15-08-24-Industrial-Hemp-Cultivation-Area-2015-EIHA1.pdf</a>>.

- GESTRIN, Ferdo. Industrijske rastline (lan, konoplja, murve) na Slovenskem. *Kronika (Ljubljana*), 1991, **39**(3), 39–44.
- 29. *Republika Slovenija. Statistični urad* [online] [accessed 15.3.2016]. Available on World Wide Web: <www.stat.si>.
- MAČEK, J. Predivnice. Zbornik Biotehniške fakultete Univerze v Ljubljani. Ljubljana : Biotehniška fakulteta, 1995, 185–187.
- 31. KOCJAN AČKO, Darja, BARIČEVIČ, Dea, RENGEO, Dean, ANDRENŠEK, Samo. Economical important characteristics of five hemp varieties (Cannabis sativa L. var. sativa) in fields trials at Markišavci near Murska Sobota. Zbornik Biotehniške fakultete Univerze v Ljubljani, Kmetijstvo, Agricultural issue, 2002, **79**(1), 237–252.
- 32. RENGEO, Dejan. Praktične izkušnje iz projekta »Pridelava in predelava navadne konoplje v Sloveniji 2000/2001. Murska Sobota, 2002 [online] [accessed 20.10.2016]. Available on World Wide Web: <www.konoplja.org/web/Konoplja/ Industrija/Gradivo\_Rengeo.doc>.
- 33. KOCJAN AČKO, Darja, BARIČEVIČ, Dea. Hemp (*Cannabis sativa* L. var. *sativa*) stem and seed yield in dependence of production technology and sawing dates in field trials at Markišavci near Murska Sobota. *Sodobno kmetijstvo*, 2004, 37(1), 33–39.
- IVANČIČ, Anita. Konoplja tabu ali poslovna priložnost? *Podjetnik*, 2014, 6 [online] [accessed 20.10.2016]. Available on World Wide Web: <http://www.podjetnik.si/clanek/konoplja-tabuali-poslovna-prilo%C5%BEnost-20140609>.
- 35. Skupni katalog sort EU. 1. Skupni katalog poljščin. 34. dopolnjena izdaja (2015/C 404/01) *Uradni list Evropske unije*, 4. 12. 2015, 229–230 [online] [accessed 20.10.2016]. Available on World Wide Web: <http://www.uvhvvr.gov.si/si/ zakonodaja\_in\_dokumenti/rastlinski\_semenski\_ material/ostali\_dokumenti/skupni\_katalogi\_ sort\_eu/>.
- SVETE, Tatjana. Rastlina, ki obeta odprtje tisočev delovnih mest. Konoplja osvaja Slovenijo. *Misteriji*, 2013, **20**(237), 12–13.
- BRDNIK, Žiga. Industrijska konoplja v Sloveniji. Konoplja.net. 2016 [online] [accessed 20.10.2016]. Available on World Wide Web: <a href="http://konoplja.net/industrijska-konoplja-v-sloveniji/">http://konoplja.net/industrijska-konoplja-v-sloveniji/</a>.
- 38. SHARMA, H. S. Shekhar. *The biology and processing of flax*. Edited by HS. Shekhar Sharma

and CF. van Sumere. Belfast : M Publications, 1992, 323.

- BATRA, Subhash K. Other long vegetable fibers. Abaca, banana, sisal, henequen, flax, ramie, hemp, sunn, and coir. In *International Fiber Science and Technology Series/7. Handbook of Fiber Science and Technology: Vol. IV. Fiber Science*. Edited by Menanchem Lewin and Eli M. Pearce. New York, Basel : Marcel Dekker, 1983, 745.
- JANKAUSKIENĖ, Zofija, GRUZDEVIENĖ, Elvyra. Beniko and Bialobrezskie – industrial hemp varieties in Lithuania. Environment. Technology. Resources : proceedings of the International Scientific and Practical Conference, 2009, 1, 176–182, doi: 10.17770/etr2009vol1. 1096.
- 41. THYGESEN, Anders. Properties of hemp fibre polymer composites – an optimization of fibre properties using novel defibrillation methods and fibre characterization : PhD Thesis. University of Denmark, 2006.
- BALTINA, I., ZAMUŠKA, Z., STRAMKALE, V., STRAZDAS, G. Physical properties of Latvian hemp fibres. *Environment. Technology. Resources : Proceedings of the 8<sup>th</sup> International Scientific and Practical conference*, 2011, **11** [online] [26.1.2017]. Available on World Wide Web: <a href="http://org.daba.lv/LLZC/Zinatniska\_darbiba/L-8356.pdf">http://org.daba.lv/LLZC/Zinatniska\_darbiba/L-8356.pdf</a>>.
- 43. KONING, Ross E. 1994. Stems. *Plant physiology information website* [online] [accessed 31.1.2017].

Available on World Wide Web: <a href="http://plant-phys.info/plant\_biology/stems.shtml">http://plant-phys.info/plant\_biology/stems.shtml</a>.

- 44. THAMAE, T., AGHEDO, S., BAILLIE, C., MA-TOVIC, D. Tensile properties of hemp and Agave americana fibres. In *Handbook of tensile properties of textile and technical fibres*. Edited by A. R. Bunsell. Boca Raton [ect.] : The Textile institute; CRC Press ; Oxford; Cambridge; New Delhi : Woodhead Publishing, 2009, 80.
- 45. THOMSEN, Anne Belinda, RASMUSSEN, Søren, BOHN, Vibeke, VAD NIELSEN, Kristina, THYGESEN, Anders. Hemp raw materials: the effect of cultivar, growth conditions and pretreatment on the chemical composition of the fibres. 2005 [online] [accessed 24.1.2017]. Available on World Wide Web: <a href="https://www.votehemp.com/">https://www.votehemp.com/</a> PDF/ris-r-1507.pdf>.
- DUVAL, Antoine, BOURMAUD, Alain, AUGI-ER, Laurent, BALEY, Chistophe. Influence of the sampling area of the stem on the mechanical properties of hemp fibers. *Materials Letters*, 2011, 65, 797–800, doi: 10.1016/j.matlet.2010.11.053.
- 47. LIU, Ming, FERNANDO, Dinesh, DANIEL, Geoffrey, MADSEN, Bo, MEYER, Anne S., ALE, Marcel Tutor, THYGESEN, Anders. Effect of harvest time and field retting duration on the chemical composition, morphology and mechanical properties of hemp fibers. *Industrial Crops and Products*, 2015, **69**, 29–39, doi: 10.1016/j.indcrop.2015.02.010.