

The Impact of Mechanical and Chemico-mechanical Treatment on Natural Fibre Dimensions

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Abstract. The Research aim is to find the most time and energy economic treatment of natural fibres to obtain micro and nanofibres. Alkaline treatment, steam explosion autohydrolysis and ultrasonic treatment effect on hemp fibres and shives dimensions were examined. Fibres length, diameter, form factor and sifting amount were analyzed. The results of the analysis show that an increase in treatment intensity gives a higher amount of fibres being noticeably shorter and thinner than the untreated dew-retted fibres. Fine content of fibres and shives increases, when treatment intensity increases.

Keywords: steam explosion autohydrolysis, ultrasonic treatment, hemp, fibres dimensions, fibres tester.

I. INTRODUCTION

Nowadays there is a trend to replace synthetic micro and nanofibres with natural fibres, not only for economic reasons but also for environmental reasons - natural fibres are degradable, recyclable resource at low cost.

Natural technical fibres are complex formed from fibres bundles. Each elementary fibre of bundle is a complex layered structure composed of cells surrounded by cell walls which are thick, rigid membranes. This layer of cellulose fibre gives the cell most of its support and structure. The structurally strong framework in the cell walls are formed by cellulose macrofibrils located in thin primary cell wall, which clasps broad secondary cell wall whose main components are cellulose macrofibrils embedded in hemicellulose and lignin amorphous matrix [4]. The cell wall also bonds with other cell walls to form the structure of the plant. The cellulose molecules are organised in a hierarchical structure of macrofibrils with a diameter range 0.1–1 microns, which consist of microfibril bundles in a diameter of 10–70 nm. One bundle is usually composed of ~ 70 microfibrils. The primary cell wall contains randomly oriented microfibrils, both outer layers of secondary wall are built from cross-laminated sheaths of microfibrils. Microfibrils of secondary wall inner layer are disposed in a right hand spiral and the thickness of this layer is much greater than that of the primary and outer layers of secondary wall. The orientations of microfibrils in the secondary wall inner layer determine mechanical properties of the fibre. The hemp fibres microfibril angle 6,2° of this layer is the second lowest after corresponding value flax fibres (5°) [6]. Microfibril consists of 30–100 cellulose molecules arranged in chains that ensure the mechanical

strength of fibres [2]. Cellulose microfibrils are linked together with lignin and hemicelluloses matrix [3].

Studies have shown that cellulose micro and nanofibres have higher mechanical properties (stiffness 120 GPa; strength 15000 MPa) than bast fibres (strength 50 MPa, stiffness 800 GPa) and their strength is six times higher than that of carbon fibres [1]. Defibrillated fibres contain a higher percentage of cellulose. Because of these properties cellulose micro and nanofibres are investigated as a potential material for applications in medicine, building constructions and mechanical engineering.

For micro and nanofibre obtaining from elementary plant fibres several treatments must be carried out, this need to be done because fibres cell wall is not a homogeneous membrane [4]. To separate cellulose microfibrils from macrofibrils, matrix degradation should be carried out with chemical, mechanical or chemico-mechanical treatment; however, studies have shown that for natural fibre it is not enough with only one type of treatment. This is why for fibre obtaining pre-treatment and various methods of treatments are used. Post-processing of the fibres is used to normalize the pH level and rinse out harmful substances.

The aim of this research is to find the most efficient and ecological way to obtain natural micro and nanofibres.

II. MATERIALS AND METHODS

Six samples of hemp fibres and two samples of hemp shives with different treatment methods were examined, i.e., pre-treatment, steam explosion autohydrolysis and ultrasonic treatment.

A. Hemp Fibre

In a moderate climate usually are grown hemp fibres or mixed fibres-seed varieties, less varieties for oil production. Technical hemp in Latvian weather in 114-117 days is able to grow from 1,77 m (local breed) to 2,7 m (industrial variety Bialobrzzeskie). Stem yield depending on the variety is about 7,9 to 18,2 t/ha, with the bast content in a range 25 to 30% [5]. Fibres and shives amount depends on the plant density in the field. Hemp plants grown in a low density, they have thick and rich branched stems; it means that such stems carry an increased amount of shives and low fibres outcome.

Hemp fibres applications depend on their physical properties and chemical composition, which depends on the plant variety and growing conditions. Bast fibre diameter is about 17-23 μm , length is 8,3 to 14 mm. The breaking strength $\sim 28\text{-}36$ cN / tex [7], elongation $\sim 2 - 4\%$, Young's modulus 30-60 GPa [6].

The average volume of cellulose content in hemp fibres is 65-70 %, but in shives it is by more than twenty percent less, about 46,3%. Hemicelluloses and lignin content of hemp shives is higher than that of fibres [7].

As a study subject for this research dew-retted (dew soaked) Latvian clone "Purini" hemp fibres and shives are used. Hemps were grown on the experimental fields of the Latgale Agricultural Science Center at 2011 th growing season. Fibre linear density 2,3 tex, the moisture content in a range 9%-12% [8]. This hemp clone is grown as oil product, and hemp fibres and shives as secondary products are not usually utilized in the best way.

B. Pretreatment

Dew-retted hemp fibres and shives were used as the experiment object. After dew-retting one part of samples were treated with 4 wt % sodium hydroxide solution. The process is carried out for one hour at temperature 80 $^{\circ}\text{C}$ to defibrillate fibres and partially remove of hemicelluloses, lignin and pectin [9]. As post-processing method water and alkaline extraction was used by washing samples with boiling distilled water for three hours, followed by the treatment with 0,4 wt % NaOH solution [10] (Table I).

C. Steam Explosion Autohydrolysis

The steam explosion auto hydrolysis treatment provides a partial destruction of fibres lignin [11]. After the steam explosion fibre has rough surface and higher crystallization index [12].

Steam explosion autohydrolysis parameters can be changed in the temperature range from 160 $^{\circ}$ to 250 $^{\circ}\text{C}$, pressure - from 20 to 40bar, the exposition time - from a few seconds up to 10 min. Processing intensity is characterized with $\log R_0$, which is also identified as a reaction coordinate and expressed by the formula (1) [13]:

$$R_0 = t * \exp \left[\frac{T-100}{14.75} \right] \quad (1)$$

Where: t-treatment time in minutes, T- temperature $^{\circ}\text{C}$ that describes treatment intensity relative to a base temperature $T_{\text{base or reference}} = 100^{\circ}\text{C}$ [13].

Four samples of hemp fibres and two samples of shives were subjected to the steam explosion treatment at 32 bars pressure for 1 and 3 minutes. The fibres samples were processed at temperature range from 200 $^{\circ}\text{C}$ to 235 $^{\circ}\text{C}$, shives

were treated at 235 $^{\circ}\text{C}$ temperature. After steam explosion fibres were treated with water and alkaline extraction. The steam explosion treated fibres dimensions were compared with the dimensions of pre-treated (4% NaOH) and after-treated during 45min with ultrasound fibres (see Table I).

D. Ultrasonic treatment

In the ultrasonic process fibres are subjected to a number of processing stages - homogenization, dispersion, disagglomeration and cell structure degradation [14]. As a result defibrillated fibres can be obtained.

Hielshcer ultrasonic homogeniser UP200Ht with the sonotrode S26d14 was used with operating frequency 26 KHz, peak power 200 W and amplitude range from 10% to 100%. Time, power and cycle changes are variables based on the required treatment length and severity [15].

1 wt % fibres water solution was treated with 90% amplitude, 100W power for 45 minutes, ultrasound process was not interrupted during operation, the depth of sonotrode immerse - 7mm.

E. Fibre tester

Lorentzen & Wertte fibre laboratory type analyzer for fibre length, diameter, slack, shape factor and roughness measurements was used. The analysis is fully automatic, suitable for frequent measurements of fibres [16].

Fibre analyzer measures the perimeter of the fibre and the area what fibre occupies. Length and diameter are calculated by area (2) and perimeter (3) formulas. The area and perimeter of each fibre is measured.

$$A = L \times W \quad (2)$$

$$P = 2 \times L + 2 \times W \quad (3)$$

Where: A is area occupied by fibre, L-fibre length, W-fibre diameter, P-fibre perimeter.

Shape factor is determined by a formula multiplying the length of the fibre by the area circle diameter. The form factor ratio is closer to 100%, when fibres wrinkling are low.

F. Experimental parameters

Fibres were cleaned from impurities and shives and cut into 2 mm long pieces. Shives were cleaned from fibres and grounded in to 2mm long pieces.

The plan of experiments is shown in Table 1. All variants of experimental fibres after treatments proposed by experiment plan are prepared for further measurements. 0,130 g of each sample were soaked in distilled water and then tested with fibre tester. From 815 (PuTil 1) to 99 264 (PuTil 6) fibres were measured.

TABLE I
EXPERIMENTAL METHODS AND PARAMETERS

Sample	Material	Dew-retted	4%NaOH, 80 °C, 1h	Steam Explosion		Ultrasonic treatment A=90%, 100 W, min
				Parameters	logR ₀	
PuTil 1	Fibre	+	-	-	-	-
PuTil 2	Fibre	+	+	-	-	-
PuTil 3	Fibre	+	-	1 min, 32 bar, 220 °C	3,53	-
PuTil 4	Fibre	+	+	1 min, 16 bar, 200 °C	2,94	-
PuTil 5	Fibre	+	-	3 min, 32 bar, 235 °C	4,45	45
PuTil 6	Fibre	+	-	-	-	45
THC 1	Shives	-	-	1 min, 32bar, 235 °C	3,67	-
THC 2	Shives	-	-	3 min, 32bar, 235 °C	4,45	-

III. RESULTS AND DISCUSSION

A. Steam explosion auto-hydrolysis

To the steam explosion auto-hydrolysis (STEX) treatment three fibres samples and two samples of hemp shives were subjected with the treatment intensity varied from the medium to the medium severe.

TABLE II

MASS, MOISTURE CHANGES AFTER STEAM EXPLOSION AUTO-HYDROLYSIS

Sample	Log R ₀	Mass before STEX, g	Moisture before STEX,%	Mass after STEX, g	Moisture after STEX,%
PuTil 3	3,53	100	6,4	92,5	4,8
PuTil 4	2,94	100	6,7	93,3	6,1
PuTil 5	4,45	100	6,4	86,2	4,5
THC 1	3,67	100	11,9	79,8	4,4
THC 2	4,45	100	11,9	79,9	4,1

Mass and humidity of the fibres were determined before and after STEX. Steam explosion treatment decreased the dry matter weight. During treatment lignin destruction and loss of volatiles are observed. Increasing intensity of fibres treatment increases mass loss. Hemp shives have the highest mass loss. Fibres with the highest LogR₀ value have the highest moisture level reduction.

B. Ultrasonic treatment results

Fibres samples PuTil 5 and PuTil 6 were treated with ultrasound with the same treatment intensity and time.

TABLE III

MASS, MOISTURE CHANGES AFTER ULTRASONIC TREATMENT (US)

Sample	Mass before US,g	Moisture before US,%	Mass after US, g	Moisture after US,%
PuTil 5	1	7,56	0,98	9,78
PuTil 6	1	5,67	0,85	8,48

Mass and humidity of the fibres were determined before and after ultrasonic treatment. The dry matter weight has decreased – lignin destruction and loss of volatiles are observed. The moisture content of the fibres has increased by more than 2%. The maximum temperature reached in ultrasonic treatment process is ~50°C; this temperature does not affect defibrillation process.

C. Fibres and shives parameter distributions

Fibre lengths were distributed in length classes; fibre and shive modal length (Mo.l.), modal group (M.gr.), short fibre content (Sh.f.), short fibre content that is 1mm or shorter (Sh.c.), mean length (M.l.), mean diameter (M.d.), diameter interval (D.i.), mean shape coefficient (M.sh.c.), shape coefficient interval (Sh.c.i.), fines content (F) and coarseness (C) were determined (Table IV).

TABLE IV

FIBRE TESTER DATA ANALYSIS

Sample	Mo.l., mm	M.gr., %	Sh.f.< 1mm, %	Sh.c.> 1mm, %	M.l., mm	M.d., µm	D.i., µm	M.sh.c., %	Sh.c.i., %	F., %
	1	2	3	4	5	6	7	8	9	10
PuTil 1	1,08	65,7	10,8	76,5	1,17	61,2	4,70	90,4	22,0	7,7
PuTil 2	1,03	56,7	15,5	72,2	1,22	41,7	25,3	83,6	32,9	2,7
PuTil 3	1,04	58,0	16,3	74,4	1,14	45,2	14,3	89,0	19,9	7,9
PuTil 4	1,00	49,5	43,7	93,2	1,54	40,6	50,5	84,3	34,3	8,8
PuTil 5	1,02	53,9	25,8	79,7	1,03	32,4	14,8	84,9	28,5	51,4
PuTil 6	0,5	49,3	49,3	94,8	0,66	35,1	31,0	75,5	30,7	78,8
THC 1	0,98	46,2	37,8	84,0	0,86	47,6	39,0	79,1	19,7	57,7
THC 2	0,57	63,3	63,3	93,7	0,60	48,2	24,8	83,8	10,2	63,1

Fibres and shives length decreases when treatment intensity increases. The lowest modal fibre length of 0,5mm has dew-retted fibres PuTil6 that have been treated with ultrasound for 45 minutes; 94,8 % of these fibres are shorter than 1mm (Table IV, column 1, 2 and 4). In the modal group of shives THC 2 treated with the steam explosion auto-hydrolysis for 3 minutes (severity 4,45) included fibres with length 0,57 mm (63,3% from total), but coarseness 450 $\mu\text{g}/\text{m}$ is still high (Table IV, columns 1,2).

The impact of fibres group with the length 1mm or shorter increases by increasing treatment intensity: dew-retted short fibre content is 76,5%, but short fibre content for ultrasonic treated fibres is 94,8% (Table IV, column 4, Fig.2). The rising steam explosion auto-hydrolysis severity from 3,67 (PuTil 5) to 4,45 (PuTil 6) results in 15 % increase of fibres group with the length 1mm or shorter and decrease by 36 % average fibres length (Table IV, columns 4 and 5). It means that increased treatment improves fibre defibrillation process.

Fibre diameter is the main property that shows defibrillation level. The mean diameter analysis of experimental fibres shows that medium intensive treatment defibrillates fibres better than mediate treatment (Fig. 1). After ultrasonic treatment and medium intensive steam explosion auto-hydrolysis fibre diameter decreases by 28,8 μm (PuTil 5). Diameter distribution varies regardless of the method used. Dew-retted untreated fibres and fibres that have been treated with medium intensive steam explosion have smooth diameter distribution (Fig.1.)

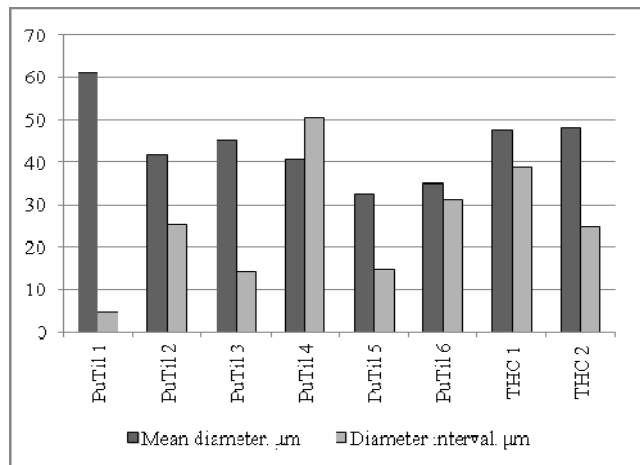


Fig. 1. Fibre mean diameter and interval

Fibres and shives form factor varies irregularly; it does not depend on the treatment or its intensity. Dew-retted untreated fibres have the highest mean values of shape factor as these fibres are the least twisted. The maximal value that mean value of shape factor reaches is 75,5 % (PuTil 6); this shape factor does not affect the quality of the end product.

As the fibre tester does not measure fibres that are shorter than 0,2mm, highest fine content is the aim of treatment. Fine content rises dramatically if fibres are subjected to the medium intensive treatment. Compare to the dew-retted untreated fibres treated with steam explosion auto-hydrolysis and

ultrasonic (PuTil 5) fibres have 43,7% fine content rise. PuTil6 fibres have the highest fine and short fibres content as it fines are exceeding 10 times dew-retted untreated fines content (Fig.2.).

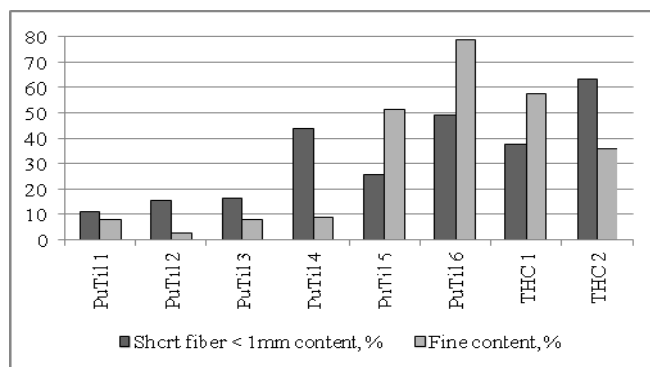


Fig. 2. Short fibre and fines content

The comparison of hemp shives treated with the steam explosion auto-hydrolysis for 1minute ($\log R_0$ 3,67) and for 3 minutes ($\log R_0$ 4,45) in 32 bar pressure parameters shows that more intensive processing leads to the higher defibrillation level: 3 minutes processed shives fibres mean length is by 0,2mm less than that of one minute treated shives; mean diameters are not changed, but 3 minutes treated shives fines content is 63,1% and it is larger than one minute treated shives fine content (57,7%).

Fibres and shives surface coarseness decreases with the increase of treatment severity, with the exception of alkaline and steam explosion treated fibres (PuTil 4) with the largest surface roughness 9161.3 $\mu\text{g}/\text{m}$ (Table IV, column 11). The relatively low surface roughness has fibres subjected to the steam explosion auto-hydrolysis and ultrasonic treatments: PuTil 5 (154,0 $\mu\text{g}/\text{m}$) and the lowest coarseness show ultrasound treated fibres PuTil 6 (0,1 $\mu\text{g}/\text{m}$) (Table IV, column 11).

IV. CONCLUSIONS

The defibrillation level of dew-retted fibres is not sufficient for further processing as fibres diameters are in range from 59,2 to 63,9 μm , average length 1,165 mm and the fines content is only 7,7%. These parameters do not meet the requirements.

Dew-retted alkaline treated fibres do not ensure defibrillation level high enough as obtained fibres diameter range 59,2- 63,9 μm , average length 1,22mm and fine content 2,7% are not sufficient for further electrospinning. At the same time alkaline treated fibres show higher defibrillation level than dew-retted untreated fibres.

Compared to the alkali treated fibres defibrillation level has not changed, mediate steam explosion effects are not noticeable.

The increasing steam explosion auto-hydrolysis severity to 3,53 mean length and diameter decrease are observed, but the increased medium steam explosion treatment does not give sufficient fibres parameters needed for electrospinning.

The combination of severe steam explosion auto-hydrolysis ($\log R_0$ 4,45) and ultrasonic treatment applied to the dew-retted fibres increases the degree of defibrillation as the average fibres length is 1,027mm and diameter - 32,4 μ m, and more than 51% of PuTil 5 fibres are fines.

The ultrasonic treatment effect at the defibrillation express in fibres average length 0,664mm, the mean diameter - 35,1 μ m and fines content of 78%. These parameters offer good perspectives for further processing.

The study shows that for nano-scale defibrillation level at least medium intensive ($\log R_0$ 4,45) steam explosion auto-hydrolysis treatment is necessary. For better results a different treatment method must be used – the ultrasonic treatment gives good results.

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Laima Grave, Silvija Kukle, Anna Šutka, Ausma Viļumsone. Mehānisko un ķīmiski mehānisko apstrāžu ietekme uz dabīgo šķiedru izmēriem

Latvijā kaņepes audzē kā eļļas augus, bet šķiedras ir to blakus produkts, kas parasti netiek izmantots pietiekoši efektīvi, tāpēc nepieciešams atrast tām tautsaimnieciski izdevīgus lietojumus. Rakstā atspoguļoti pētījumu rezultāti, kas vērsti uz efektīvu priekšapstrādes operāciju kombināciju meklējumiem, lai no dabas šķiedrām iegūtu mikro- un nano- līmeņa šķiedras. Eksperimentā kā izejas materiāli izmantotas Latvijas kaņepju klona „Pūriņi” šķiedras un spaļi. Seši šķiedru un divi spaļu paraugi pakļauti dažādu operāciju salikumu un intensitātes tehnoloģijām. Noteikta eksperimentāli 4 wt % nātrija sārna, tvaika sprādziena auto-hidrolīzes un ultraskaņas apstrādes ietekme uz paraugu šķiedru garumu, diametru, formas faktoru un smalkņu īpatsvaru un virsmas raupjumu. Rezultātu analīze parāda, ka, palielinot apstrādes intensitāti, paaugstinās šķiedru defibrilācijas līmenis. Tilinātas neapstrādātas šķiedras, ar sārmu apstrādātas šķiedras un šķiedras, kas apstrādātas ar vidēji intensīvu tvaika sprādziena auto-hidrolīzi, nedod pietiekamu sašķiedrošanas līmeni, lai šķiedras būtu iespējams pakļaut elektrovērpšanai nanošķiedru iegūšanai. Vislabākie rezultāti sasniegti, kombinējot šķiedru sagatavošanas procesā elektrovērpšanai vidēji intensīvu tvaika sprādziena auto-hidrolīzi ar ultraskaņas apstrādi. Iegūto šķiedru vidējais garums ir par 0,51mm mazāks nekā apstrādei nepakļautu tilinātu kaņepju šķiedru vidējais garums, un vidējais diametrs samazinājies par 52%, šķiedru īpatsvars, kas īsāks par 0,2mm, sasniedz 79%; tas ir pietiekami augsts sašķiedrošanas līmenis turpmāko tehnoloģiju piemērošanai.

Лайма Граве, Силвия Кукле, Анна Шутка, Аусма Вилюмсоне. Воздействие механической и химически - механической обработки на размеры натурального волокна

Латвийская конопля выращивается как масляная культура, а волокно является побочным продуктом, который обычно не используется достаточно эффективно, поэтому необходимо найти их наиболее выгодное использование в народном хозяйстве. Целью данного исследования является поиск наиболее эффективного способа обработки натуральных волокон для получения микро- и нано-волокон, которые могут быть подвергнуты дальнейшей обработке, такой, как электропрядение. В эксперименте использованы волокна и костра латвийского конопляного клона "Пурини". Шесть образцов волокна и два образца костры подвержены технологиям обработки различным набором операций и интенсивностью. В исследовании осмотрено влияние обработки 4wt % натрия щелочи, паровым взрывом авто-гидролиза и ультразвуком на размеры – длину, диаметр, фактор формы и удельный вес волокон длиной меньше 0,2 мм и шероховатость поверхности волокон. Анализ результатов показывает, что увеличение интенсивности обработки повышает уровень фибрилляции волокон. Не обработанное волокно, моченое в росе, волокно, не обработанное щелочью, волокно, не обработанное средним паровым взрывом автоматического гидролиза, не обеспечивают достаточно высокий уровень волокнистости для проведения электропрядения. Сочетание парового взрыва автогидролиза средней интенсивности и ультразвуковой обработки дает наилучшие результаты. Волокно, обработанное таким образом, на 0,51 мм короче и диаметром на 52% меньше, чем необработанное волокно конопли, моченой в росе. Удельный вес волокна, которое короче на 0,2 мм, достигает 79%, и это доказательство тому что, достигнут достаточный уровень фибрилляции, и волокна могут быть подвергнуты дальнейшим обработкам.