



# Steam Explosion Impact to Technical Hemp Fiber Diameter

Laima Grāve<sup>1</sup>, Anna Putniņa<sup>1</sup>, Silvija Kukle<sup>1</sup>, Veneranda Stramkale<sup>2</sup>

1 - Riga Technical university Department of Design and Textile Products Technology Azenes Str.  
14/24, Riga, LV1048, Latvia

2 - Agriculture Science Centre of Latgale Kultūras laukums 1a, Viļāni, Viļānu novads, LV-5650

**Abstract.** Microfibers and nanofibers from natural products have high mechanical properties. For this reason researchers pay particular attention to the natural fibers and to the method how they are obtained. In the research samples with different treatments (alkaline pretreatment, steam explosion and water extraction) were investigated to evaluate treatment influence on fiber diameters. Fractionation does not provide actual results of fibers diameter as thinner fibers agglomerate to each other and around thicker fibers and cannot be sieved. Fiber diameter measurement with an optical microscope shows that pretreatment and treatment reduced the thickest fiber percentage and increase the number of fine particles. Dew-retted hemp gives 16% of thicker fibers and only 39% fibers with a diameters less than 63 $\mu$ m. 50% of alkaline pretreated and steam explosion treated fibers diameters are less than 63 $\mu$ m and only 8% of diameters fall in range 160-630 $\mu$ m.

**Keywords** – natural fiber, fiber diameter, steam explosion, alkaline pretreatment.

## I INTRODUCTION

In recent years researchers and manufacturers try to replace man-made fibers, such as glass and carbon fibers, with natural fibers. Now they are used to strengthen the materials and as a fillers to make environmentally friendly products.

The natural cellulose-based fibers have several advantages. They are low cost renewable resource with low density, high specific strength and stiffness, high sound absorption capacity and non-abrasive nature [1, 2]. As microfibers and nanofibers from natural products have high mechanical properties researchers pay particular attention to the natural fibers, trying to combine them with polymers in composites and nano-composites [3] for usage in automotive industry, as geotextiles, in medicine and elsewhere. The appeal of natural fiber products should primarily be its quality.

Microfibers are defined as fibers of cellulose of 0.1-1 $\mu$ m in diameter, with a corresponding minimum length in range 5-50 $\mu$ m, and nanofibrils if at least their one-dimension is at the nanometer scale (1-100nm) [4].

The lignocellulosic fibers are made up of cellulose microfibrils bonded together by lignin and hemicellulose matrix. Physical properties of natural fibers are basically influenced by the chemical structure such as cellulose content, degree of polymerization, orientation and crystallinity, which are affected by conditions during growth of plants as well as extraction methods used [2, 4].

Micro and nanofibers can be obtained by chemical, mechanical or chemico-mechanical methods. The main problem is in the way how to obtain micro and nanoscale natural fibers. It is important to clarify

which of them has potential and which technologies produce low yields and are not environment friendly or energy efficient.

The main objective of this study is to investigate the alkaline pretreatment, steam explosion treatment and water extraction impact on technical hemp fibers geometric parameters. The main aim are to determine types of diameter distributions and find out which fraction has the highest share (modal fraction) depending on applied treatments and evaluate their influence on further processing.

## II MATERIALS AND METHODS

### A. Hemp

Hemp (*Cannabis sativa* L.) is an annual herbaceous plant from the hemp family (Cannabaceae). Technical hemp plants in Latvian weather conditions in 114-117 days can grow from 1,77 (local variety) to 2,7 m (industrial variety Bialobrzесьkie) high with the stem yield 1120-1140g/m<sup>2</sup> depending from variety and with 22% to 24% fibers yield [5].

Hemp fibers from hemp varieties grown in Latvia contain about 65-68 % cellulose, 20-22 % hemicelluloses, 6-8 % lignin and 1,4-1,6 % pectin, 0,8-1,25 % fats & waxes [6].

As experimental objects fibers from dew-retted hemp grown on trial fields of Latgalian Agricultural Science Centre at vegetation periods of 2011 and 2012 are used.

### B. Alkaline pretreatment

The 4 wt% sodium hydroxide was used for one hour at 80 degrees as alkaline pretreatment to partially

extract lignin, pectin and hemicelluloses, and separate the fiber bundles [7, 8].

C. Steam explosion treatment

One of techniques that can open the hidden nanostructures is the steam explosion auto-hydrolysis. The process of which includes fast impregnation of the plant material with saturated steam in a closed reactor. The treatment proceeds at moderate temperature and pressure for a desired period of time (from some seconds to some minutes). After that the substance is decompressed within parts of a second [9]. The steam explosion process gives clean fibers with rough surfaces and increased crystallinity index [10]. Hemp fibers in the experiment were treated with 16 bar pressure for 1 minute. Water extraction was used to separate fibers from dissolved lignin.

D. Experiment parameters

In this research dew-retted hemp fibers of Latvian local genotype Purini grown in Agricultural Science Centre of Latgale were used. Fibers were cleaned from impurities and shives and cut into 2 mm long pieces.

TABLE I

Sample	Dew-retted	Alkaline pretreatment	Steam explosion
1.	+	-	-
2.	+	4 wt% NaOH	-
3.	+	4 wt% NaOH	16bar 1min

EXPERIMENT PARAMETERS

Three samples with different treatment were examined (table1):

- 1 - dew-retted hemp fibers without treatment;
- 2 - dew-retted hemp fibers with alkaline (4%NaOH) pretreatment followed by water extraction;
- 3 - dew-retted hemp fibers with alkaline (4%NaOH) pretreatment, steam explosion (16 bar, 1 minute), water extraction.

Dried fibers of all three variants were fractionated through sieves with meshes of three sizes (630µm, 160µm and 63µm).

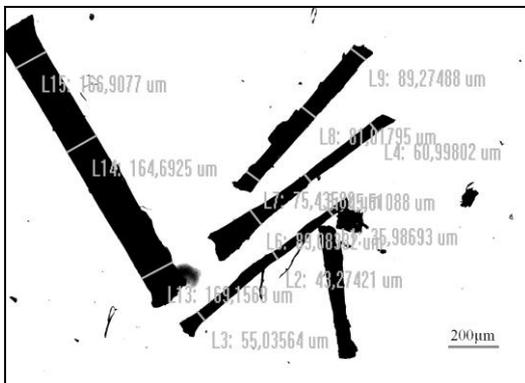


Fig.1. Fibers optical microscope measurements

Fiber diameters of each sample where measured with the optical microscope under amplification 2,5

times. From each sample 100 fibers were measured, each hemp fiber was measured in 3 places - in both ends and in middle (Fig.1.).

III RESULTS AND DISCUSSION

E. Fiber diameter ratio determination by sieving and weighing method

To determine fraction’s share after fractionation through a sieves each fraction was weighted.

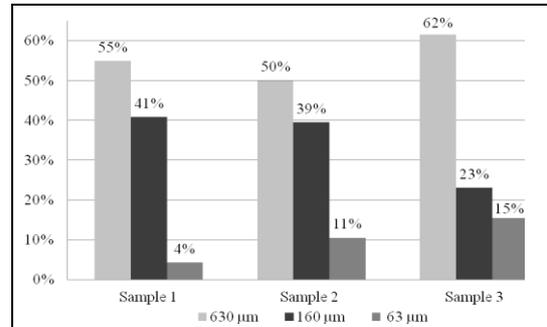


Fig.1. Weight percentage after fractionation

Graphs of Fig.1 shows that modal width percentage in each of three groups exceed 50 % and show the highest value 62% for variant with NaOH pretreatment and the following steam explosion treatment, as well increase group of finest fibers with the diameters less than 63 µm from 4% to 15 %. In spite of noteworthy growth of the finest fibers diameter group with using alkaline pretreatment and steam explosion treatment, it is still too low for further processing.

Cumulative percentage of fibers with diameters 160µm and less for all three variants are high enough (45%; 50% and 39 %) and these fibers are subjected to the further treatment



Fig.2. Dew-retted alkaline pretreated, steam explosion treated fiber agglomeration

There is a large amount of fiber that did not sieve, this is because fiber agglomeration. Fig.2. shows that thinner fibers agglomerate to each other and around thicker fiber.

In order to obtain a homogeneous mass of fibers it is useful to fractionate fiber before pretreatment and

treatment, because thinner fibers agglomerate more than thicker.

*F. Fiber diameters ratio determination by optical microscope*

Dew-retted fiber diameters distribution graph (Fig.3) show clear expressed asymmetry and 39 % of tested fibers diameters fall in modal range 22- 98  $\mu\text{m}$ .

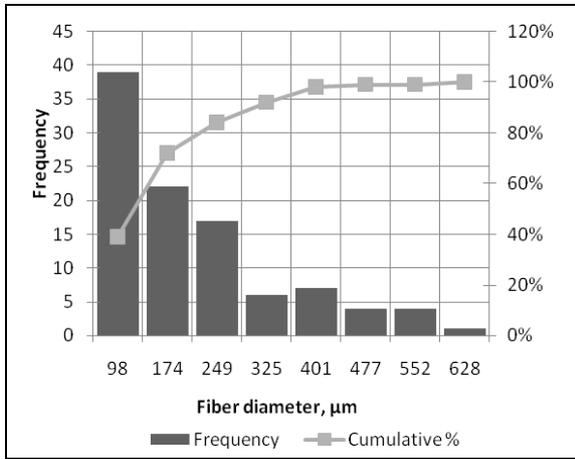


Fig.3. Dew-retted Purini hemp fiber diameters distribution

Diameters of 70% fibers not exceed 174 $\mu\text{m}$ . No one diameter find larger than meshes of chosen sieve. The average diameter (175, 67  $\mu\text{m}$ ) fall into the second interval as distribution has a long right hand tail.

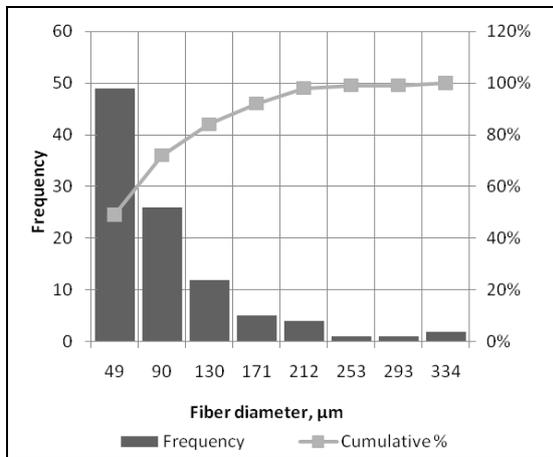


Fig.4. Diameters distribution of the dew-retted Purini hemp fibers with alkaline pretreatment and water extraction

After alkaline pretreatment and water extraction modal interval covers range from 8.268 $\mu\text{m}$  to 49  $\mu\text{m}$  and includes 50 % diameters of measured fibers as well diameters of 82 % fibers not exceed 130  $\mu\text{m}$  witnessed about substantial treatment influence on separation level of fibers complexes (Fig. 4).. Average fibers diameter 64,69  $\mu\text{m}$  is 2.64 times less than that of dew-retted fibers.

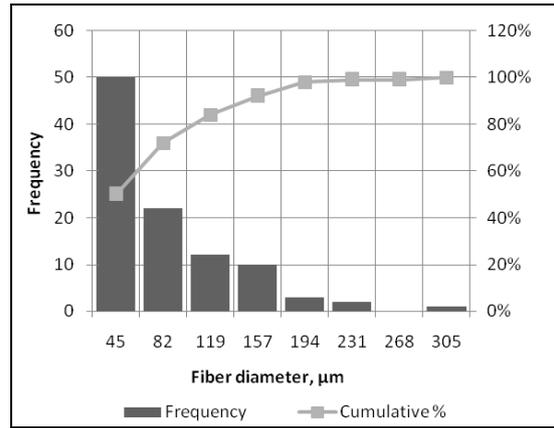


Fig.5. Dew-retted Purini hemp fiber diameters distribution after alkaline (4%NaOH) pretreatment, steam explosion 16 bar, 1 minute, water extraction

Graph of Fig 5 shows that optional treatment by mild steam explosion moves distribution to the direction of smaller diameters to compare with the distribution of sample2. Modal interval corresponds to the diameters range 5,098-45 $\mu\text{m}$  and includes 50% of measured fibers diameters, more then 72% of diameters do not exceed 82  $\mu\text{m}$ . Only 8% of fibers show diameters exceeding 157 $\mu\text{m}$ . Average fiber size 64,690 $\mu\text{m}$ .

TABLE II

Sample	Fiber diameter, %		
	160 $\mu\text{m}$ - 630 $\mu\text{m}$	63 $\mu\text{m}$ - 160 $\mu\text{m}$	$\leq$ 63 $\mu\text{m}$
1.	16%	45%	39%
2.	8%	43%	49%
3.	2%	48%	50%

THE PERCENTAGE OF FIBER DIAMETER

Table II shows that in result of alkaline pretreatment and steam explosion treatment the share of fine particles increases, the share of thick fibers decrease substantially.

IV CONCLUSION

After 4 wt% sodium hydroxide pretreatment substantially increases hemp fine fibers share, decreases modal diameters range, increases from 40% to 50% fibers share with diameters fall in modal range and decreases share of thick fibers bundles.

Optional steam explosion treatment following alkaline pretreatment does not change very much two larger distribution groups impact, but decrease substantially share of thick fiber bundles.

Comparison of alkaline treatment influence on hemp technical fibers decomposition with the steam explosion treatment with different treatment severities as parallel methods could be investigated in future

For further experiments to avoid fiber agglomeration before any treatment full fractionalization is necessary.

Further investigations are needed to compare fibers after various steam explosion treatment conditions to

determine which of them gives the best result of fiber defibrillation.

#### V ACKNOWLEDGMENTS

This work has been supported by the European Social Fund within the project «Support for the implementation of doctoral studies at Riga Technical University». The authors are grateful to the staff of Laboratory of Biomass Eco-Efficient Conversion, Latvian State Institute of Wood Chemistry.



INVESTING IN YOUR FUTURE



#### VI REFERENCES

- [1] Siqun Wang, Qingzheng Cheng, T. G. Rials and Seung-Hwan Lee, Cellulose Microfibril/Nanofibril and Its Nanocomposites, Proceedings of the 8th Pacific Rim Bio-Based Composites Symposium, 301-308.
- [2] M. Fan Elementary hemp fibres and strength. *BioResources* 5 (4), 2010., 2307-2322
- [3] Berglund, L., Cellulose-Based Nanocomposites. In: A.K.M. Mohanty, M.; Drzal, L. (Editor), *Natural fibers, biopolymers, and biocomposites*. Taylor & Francis, pp., 2005., 807-832.
- [4] Jayamol G.I, Sreekala M. S., Sabu T. A Review on Interface Modification and Characterization of Natural Fiber Reinforced Plastic Composites. *Polymer Engineering and science*, Vol. 41, No. 9, 2001. 1471-1485.
- [5] V. Stramkale, I. Nagle, V. Zepa, I.Kroiča Linu un kaņepju selekcijas materiāla novērtēšana integrēto lauksaimniecības kultūraugu audzēšanas tehnoloģiju ieviešanai, *Latgales Lauksaimniecības Zinātnes centrs*, 2012., 24
- [6] Kukle S., Stramkale V., Kalniņa D., Soliženko R. Comparison of HEMP Fibres Properties //6th International Textile Clothing and Design Conference "Magic World of Textiles" (ITC&DC): Book of Proceedings, 2012. – pp. 76-80. ISSN 1847-7275
- [7] M.Kostic, B. Pejic, P.Skundric Quality of chemically modified hemp fibers. M. Kostic et al. / *Bioresource Technology* 99 (2008) 94–99
- [8] Kukle S., Gravitis J., Putniņa A. Processing Parameters Influence on Disintegration Intensity of Technical Hemp Fibres. *Journal of Biobased Materials and Bioenergy*, 2012, Vol.6, No.4, p 440-448.
- [9] Kallavus U., Gravitis J. A Comparative Investigation of the Ultrastructure of Steam Exploded Wood With Light, Scanning and Transmission Electron Microscopy. *Holzforchung*, 49 (2), 1995., 182-188.
- [10] Leonard Y. Mwaikambo, Martin P. Ansell Chemical Modification of Hemp, Sisal, Jute, and Kapok Fibers by Alkalization *Journal of Applied Polymer Science*, Vol. 84, 2002., 2222–2234.