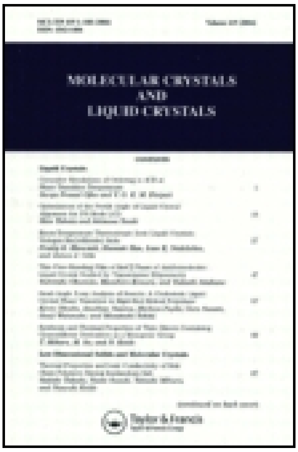

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Konczewicz Wanda [a], Kryszak Natalia [a], Nowaczkiewicz Ewa [a], Kozlowski Ryszard [b], Wojtysiak Jan [c] & Podsiedlik Wladyslaw [c]

[a] Institute of Natural Fibres and Medicinal Plants, Poznan, Poland

[b] Institute for Engineering of Polymers Materials and Dyes, Piastow, Poland

[c] Institute for Sustainable Technologies, Textile Technologies Department, Radom, Poland

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Degumming Bast Fibrous Plants by Osmosis Phenomena as a Promising Method in Primary Processing

KONCZEWICZ WANDA,[1,*] KRYSZAK NATALIA,[1]
NOWACZKIEWICZ EWA,[1] KOZLOWSKI RYSZARD,[2]
WOJTYSIAK JAN,[3] AND PODSIEDLIK WLADYSLAW[3]

[1]Institute of Natural Fibres and Medicinal Plants, Poznan, Poland
[2]Institute for Engineering of Polymers Materials and Dyes, Piastow, Poland
[3]Institute for Sustainable Technologies, Textile Technologies Department, Radom, Poland

This article reviews the methods of degumming lignocellulosic plants. The following degumming methods are used: biological, chemical, mechanical and physical – retting in warm and cold water retting, dew retting, chemical treatment by acid and base, use of electromagnetic pulses and most promising - osmotic degumming with ultrasound. The paper presents the newest methods of degumming, especially the method of osmotic degumming combined with ultrasound. This new method, developed by Institute of Natural Fibres and Medicinal Plants, is a typical pro-ecological method, because degumming water after cleaning has to be recycled. The retting water has no odour and biochemical oxygen demand and chemical oxygen demand parameters are not high. For better cost-efficiency the osmotic degumming method can be applied also for degumming green decorticated fibres.

Keywords Degumming; lignocellulosic fibres; osmotic degumming; review

Introduction

Structure and Chemical Composition of Lignocellulosic Fibres

The group of fibrous plants include: flax, hemp, jute, ramie, sisal, kenaf, manila, coir and others belong to the bast plants. The main component of the fibres is cellulose, while secondary components are lignin, hemicellulose, pectins, fats and waxes [1–5]. The chemical composition of the fibres is presented in Table 1.

Pectin plays an important role in the fibre as a component that binds the fibre into bundles and also determines the lustre and touch of the fibre. Pectins are macromolecular compounds of poly-galacturonic acid. They are agglomerated in the middle blades mainly in the ground tissue. In fibrous plants two pectin fractions can be found i.e. fraction A which is soluble in water and fraction B – non-soluble in water. Technical flax fibre consists of cells bonded with each other by a lamella, which in turn consists of mostly pectin B with

*Address correspondence to Konczewicz Wanda, Institute of Natural Fibres and Medicinal Plants, Poznan, Poland. Fax: +480618417830. E-mail: wanda.konczewicz@iwnirz.pl

Table 1. Chemical composition (%) of lignocellulolsic fibres

Fibre	Cellulose	Lignin	Pectins and hemicelluloses
Abaca	60–80	6–14	13
Bamboo	26–43	21–31	—
Cabyua	80	17	—
Coir	36–43	41–45	3–4
Cotton	83–99	6	5
Curaua	70–80	13	—
Flax	64–84	0,6–5	19
Hemp	67–78	3.5–5.5	17
Henequen	60–78	8–13	4–28
Isora	75	23	—
Jute	51–78	10–15	37
Kenaf	44–57	15–19	—
Nettle	53–82	0.5	0.9–4.8
Pineapple	80	13	—
Pita	80	17	—
Ramie	67–99	0.5–1	22
Sisal	60–80	6–14	13

a small amount of pectin A. Fibre bundles, located in the phloem, form rings around the stem, of more or less compact structure, which bundles are attached to adjacent tissues with a layer of pectin A.

Proper removal of pectins substances during preliminary processing determines the divisibility and, what follows, the fineness of the fibre and its suitability for spinning. In retting process it is the easiest to remove pectins A, which is decomposed by bacteria and fungi, while pectin B remains within the fibre and is decisive for the compactness of the fibre. Too excessive removal of pectins and natural waxes will cause that the fibre has unpleasant touch, will be dry and coarse. Complete removal of pectins will result in disintegration of fibre bundles into elementary fibres.

Waxes and fats are also important in terms of technological parameters. They determine soft touch, low friction and thus ease of moving the fibre. In flax fibre, waxes are present mainly in the outer part of the stem, in epidermis, and in smaller amounts in fibre cells.

Lignin functions as inlaying component within amorphous areas of occurring cellulose and causes that the cellulose is rigid. In the elementary fibre, lignin occurs in the primary wall and outer part of the secondary wall. While processing the presence of lignin is undesirable as touch and flexibility of the fibre become worse. Lignin causes that the fibre is more easily breakable and mechanical parameters of tenacity and resiliency deteriorate. Moreover, the divisibility of the fibre lowers as a result of presence of lignin.

The structure of elementary fibre and its microscopic, mechanical, physical and chemical properties also have effect on the overall quality of the fibre. Single fibres that constitute plant fibrous tissue are referred to as elementary Fibres, either when they are a part of a compact bundle or are loosely located within the cortex tissue. The structure of elementary fibres is similar in all lignocellulosic fibres. The quality of the extracted fibre depends on the extraction method and the amount of elementary fibres, their distribution and binding within the bundles and between the bundles. In order to separate the fibres from other tissues various mechanical treatments are used – decortication, hackling, scutching etc. Better results are achieved by preliminary processing such as water or dew retting, as well as enzymatic, chemical or physical processing of the fibre.

Fibre Extraction Methods

There are many commonly known methods for plant fibre degumming. Generally for this degumming are used the method: biological, chemical, mechanical and physical. Application of proper extraction method will result in fibre with required length, thinness and breaking tenacity as well as high purity, optimal efficiency and homogeneity [4,5]. The extraction of fibres from fibrous plants is carried out mainly by mechanical processes. Separation of non-cellulosic components from the fibre can be achieved by application of preliminary processing of raw material i.e. straw (dew retting, water retting, etc.) and enzymatic, chemical or physical processing of fibres [4–11].

The scheme of the methods of fibre plant degumming is shown on the Fig. 1. Traditional biological methods, referred to as retting, include dew retting and water retting. The traditional method of separating fibres from the woody parts of the plant, its extraction and separating from the non-cellulosic components was dew retting that underwent on the fields. This method is still used in the present days, mainly for economic and environmental reasons – there are not wastes produced in the process. The dew retting involves the growth of microorganisms, mostly fungi, which by growing mycelium penetrate the stem and produce enzymes that decompose pectin substances, commonly named as plant glues. Such retting process is difficult to control and the quality of the fibre varies much, depending on atmospheric conditions (season, temperature, air humidity, rainfall etc.).

Water retting is conducted in retting tanks or natural water reservoirs, both in warm and cold water. Retting in warm water was first described by Szenk in Ireland in 1846, after that the method quickly spread in all Europe. These processes involve biochemical

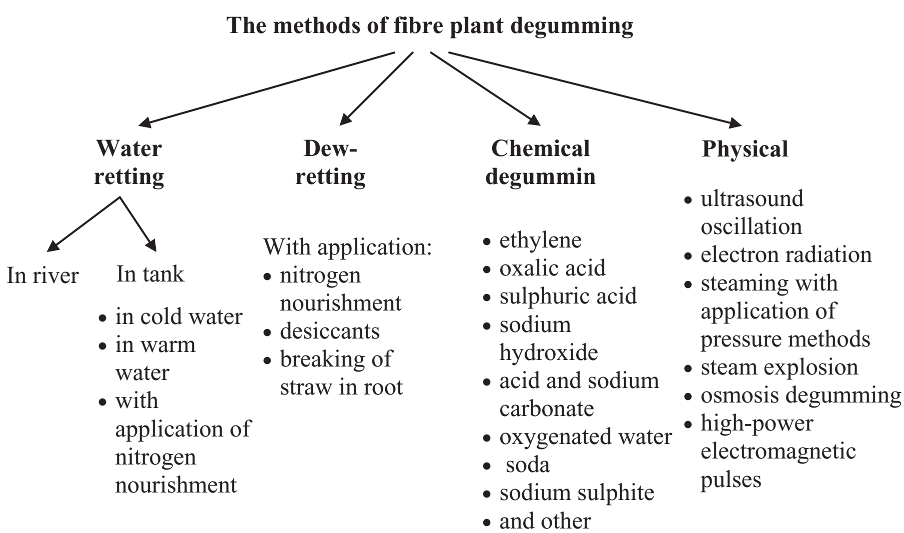

Figure 1. The methods of fibre plant.

phenomena occurring in presence of bacteria, which microorganisms cause fermentation of pectin substances and thus separating fibres from the woody parts of the stem. Depending on the fermentation conditions and the type of pectinolytic organisms, this process can have aerobic or anaerobic character. Applying retting in warm water instead of cold, allows for reducing the time of the process in controlled conditions (temperature and pH) all year round. Water retting conducted with warm water takes between 70–100 hours.

Nowadays the warm-water retting process is used only in Egypt (Fig. 2), where the retting process is conducted in natural conditions without heating water in the tank. The straw is pressed down by stones. After the retting the straw is dried on the field.

In order to create optimal conditions for the growth of the "retting" organisms and accelerating the decomposition process of pectins, mineral nutrition was applied (phosphoric, potassium and amon salts) or ferments, as well as fluid recirculation, double retting, bedding made of retted tow and enzymes. These treatments aimed at faster and simultaneous separating fibres from the woody part, shorter degumming time and better quality of fibre,

Figure 2. Warm water retting in Egypt.

mostly by improvement of divisibility and fineness of the fibres slivers. The classic warm water retting technology applied earlier in retting industry for flax straw, required using large amounts of water and heavily contaminated retting wastes were produced at all stages of processing. The retting wastes included the following: after-retting wastes, polluted water after rinsing the straw retted in the tanks and wastes after wringing. The after-retting wastes were red-brownish, turbid, characterized with unpleasant odour of organic decay, mainly of volatile fatty acids that form during fermentation such as: acetic, butyric, propionic, formic and valeric acids). Apart from that the wastes contained the products of protein decomposition, mostly amino acids. The after-retting wastes are characterized with high values of chemical oxygen demand COD and biological oxygen demand BOD. Because of that this technology was phased out.

Other methods of extracting fibres are using enzymatic treatment, chemical and physical methods. The process of chemical degumming involves using such chemical substances as: acetylene, acetic acids, sodium chlorite, hydrogen peroxide, sodium carbonate and others. The aim of chemical degumming is to shorten the process (down to a few hours), yet they may result in lower strength parameters and worsening of the fibre colour. The process must be monitored carefully to prevent damage to the fibre. Although the chemical methods have been known for years, nowadays they are not commonly used. Application of chemicals in degumming is hindered by their high cost, harmful effect on the environment and the need for purchasing specialist devices to run the process.

In order to accelerate the retting process, trials to use biologically active substances have been made – namely pure enzymes characteristic of capacity to catalyze the decomposition process in pectins. These processes of enzymatic degumming can be controlled.

Thus in the textile industry enzymatic concentrates have been produced especially designed for degumming plant fibres. These preparations combined the properties of amylase, pectinase, celulase and hemicelulase. In the retting process, enzymes from hydrolase group are of highest importance since they cause fermentation of pectins. The most common pectinolytic enzymes are presented in Table 2 [12].

The most basic factors that determine the kinetics of enzymatic reactions are temperature, ambient pH, its oxy-reductive potential, ion concentration, presence of inhibitors (or activators) and the concentration of the enzyme and substrate.

Enzymatic preparations allow for faster and simultaneous degumming of fibre from the woody part and decomposition of pectin in the fibre itself. This leads to shortening of the

Table 2. Most common pectinolytic enzymes (causing pectin fermentation)

Enzyme	Activity
Protopectinase (pectozymase)	Catalyzes the hydrolysis of protopectin, turning it into soluble pectin
Pectinopoligalacturonase (pectinase – PG)	Catalyzes the hydrolysis 1–4 glicozydic bonds in galacturonide, what leads to forming polymers or possibly monomers of galacturonic acids as a result of enzymatic decomposition
Pectin depolymerase (DP)	Catalyzes decomposition of pectinic acid to monogalacturonic acid
Pectinometyoesterase (pectase – PM)	Catalyzes the hydrolysis of metozylic groups at the last i.e. sixth carbon atom in esterified units of galacturonic acid

degumming time and improvement of fibre quality, mainly higher divisibility and fineness of fibre strands. Enzymatic treatment enables to apply the preparations that undergo complete biological degradation in sewage, what leads to developing an eco-friendly technology saving water and energy.

Physical processing of fibres comprises the following treatments: ultrasonic, steam explosion, high frequency electromagnetic radiation, electro-osmosis and osmosis.

At manufacturing plants steaming of hemp fibre has been used. The steam treatment relies on hydrolysis of the substances that bond the fibre bundles with the woody part, in an autoclave under pressure. The hydrolysis is enhancing moisture of the stems, thus before steaming the straw was soaked in warm water for about 30 minutes and then steamed in the autoclave at the pressure of 2 atm for about 2.5 hours. As a result of hydrolysis the bonds between the bast bundles and the wood and the fibre were easily separable. To obtain higher quality steamed fibre, after steaming further softening with a semi-circular squeezer and batching the fibre with detergent were conducted.

This process on flax straw was studied by Kessler in 1998 [13]. He claims that steam explosion treatment of flax following dew retting can be controlled to give a well-defined severity of treatment which leads to good fibre quality with minimum loss in fibre yield. The high potential of steam explodes shot staple flax fibres for use in textile and technical composites.

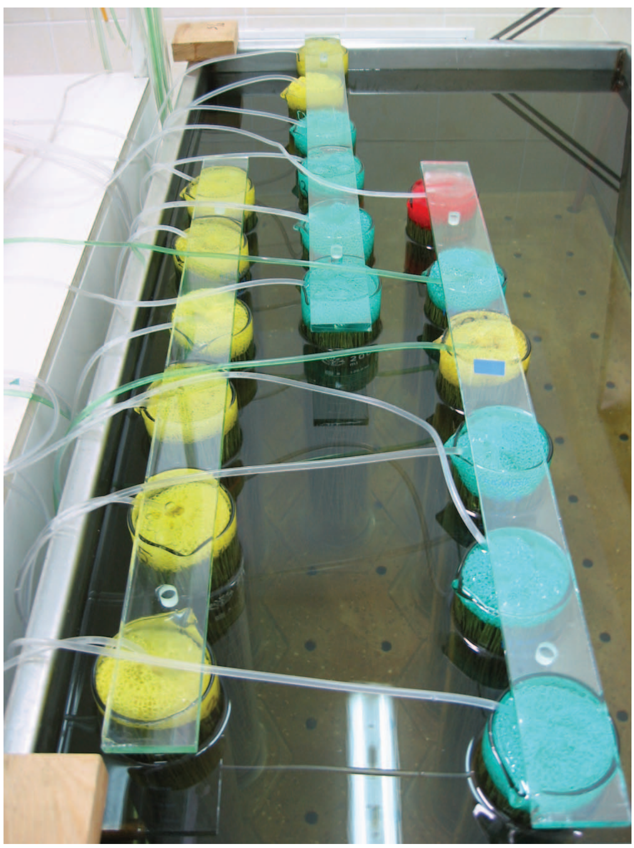

Figure 3. The laboratory evaluation of fibre content.

A new retting technology developed by Migoni and Bozzini [14] can be done by using special electric resonance electrodes immersed into a water pool, possibly with the addition into the water of specific pectinolytic enzymes, to speed up the process. The special electrodes, used for retting fibre crops in a pool, develop an electric field, without the production of electric current, but diffusing into the water a particular resonance signal.

At the INF&MP, the research concentrates on new methods of fibre degumming that allow for obtaining higher quality of fibre. An example of such methods is extraction of fibre by using the natural physical laws: water diffusion, osmosis and osmotic pressure [4–7,15–19]. All fibrous plants, especially flax and hemp, can undergo degumming process, where multiple water change or continuous water flow is used.

The degumming mechanism operates in the following order – water diffuses into the stem where the fibre and wood absorb the water and swell, while pectins (being 'super-absorbents') increase in volume several times, what leads to considerable growth of the stem diameter. At the same time the hydrostatic pressure within stem increases substantially. The pressure inserted on the epidermis causes tensions both longitudinal and peripheral.

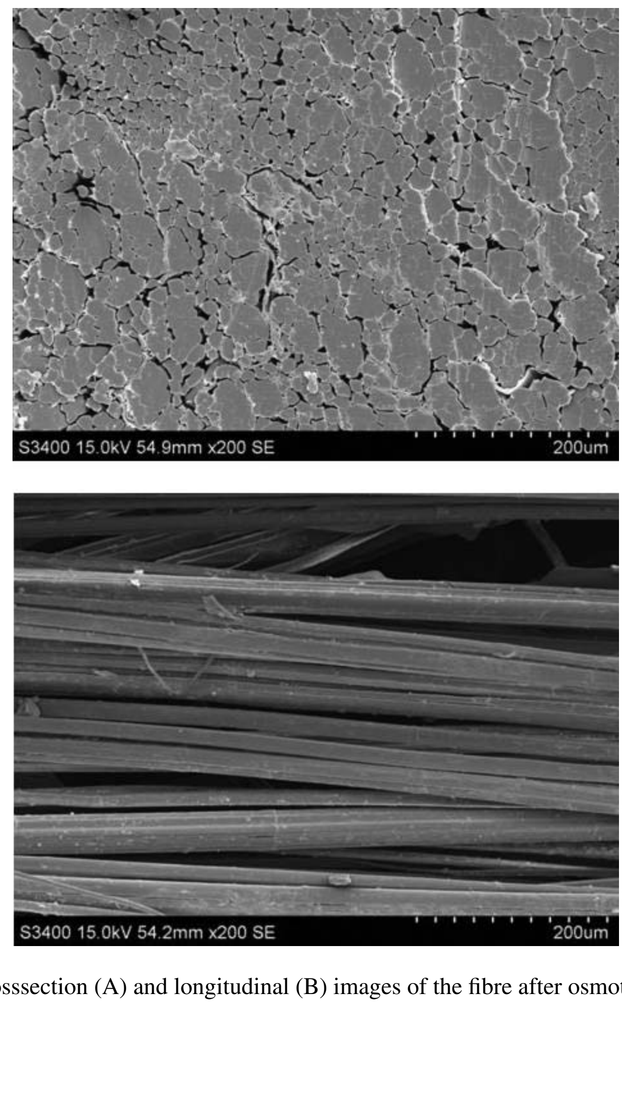

Figure 4. Crosssection (A) and longitudinal (B) images of the fibre after osmotic degumming.

Peripheral tension – as usually in case of material strength – is twice larger than the longitudinal ones and result in longitudinal breaking of epidermis, without breaking and shortening the fibres. This can be particularly attributed to the natural properties of the fibre structure, where longitudinal bonds of polymeric chains are several hundred stronger than transversal bonds. Dramatic decrease of breaking strength of the gelled pectins that bind the fibre occurs then. Later the hydrostatic pressure inside the stem pushes out gelled pectins outwards through the cracked longitudinal epidermis. The pectins become diluted and dissolved (along with other substances present in phloem) in the flowing water. This results in degumming of the fibre, what leads to obtaining high quality fibres. Generally, all soluble substances containing mineral salts, pectins, bacteria and dyes are removed from the stem.

Utilization of physical-chemical phenomena, especially osmosis, occurring inside the fibrous plants when they are exposed to water, allows for extraction of fibres without affecting the natural features of fibre. The fibre obtained by this method is delicate, thin and has colour adequate to the quality of used raw material.

Using osmotic degumming allows for obtaining homogeneous fibre and allows for objective evaluation of the amount and quality of fibre contained in the raw material – Fig. 3. This is especially important in evaluating progress in breeding new cultivars of fibrous plants.

During the studies the effect of technological parameters of the degumming process i.e. temperature, time and water flow rate, on the quality of fibre was evaluated. The best in touch fibre was obtained at temperature between 30–40°C and degumming time 72 and 96 h. The fineness reached 0.76–0.91 tex whereas breaking tenacity 48.64–57.98 cN/tex. The optimized process parameters have been test-run with the use of a specially constructed device that runs in the periodic mode of degumming fibres – Fig. 5.

Table 3. Flax fibre after osmotic degumming

Long flax fiber

Fineness	0.91 [tex]
Breaking tenacity	62.79 [cN/tex]

Hackled flax fiber

Fineness	0.71 [tex]
Breaking tenacity	50.13 [cN/tex]

Hackled flax fiber – sliver

Fineness	0.54 [tex]
Breaking tenacity	33.75 [cN/tex]

Figure 5. The device operating in periodic degumming models.

In the studies conducted with the use of a specially designed device, the fibre of similar metrological parameters have been obtained i.e.: linear mass at 0.60–0.91 [tex] breaking tenacity 49.66–62.00 [cN/tex] – Table 3. Cross-section (A) and longitudinal view (B) of the fibre is presented in Fig. 4.

In cooperation with the Institute of Sustainable Technologies in Lodz we have built a device that runs the degumming process in a periodic (Fig. 5) and continuous mode (Fig. 6).

The new technology of degumming Fibres from bast plants is definitely eco-friendly as it allows for eliminating wastes and unpleasant odour. In the extraction processes the very harmful for the environment, that both are present in traditional retting process of flax straw. Preventing the degradation of flax fibre, which phenomenon occurs during retting, will enable to obtain higher share of long fibre in the total mass of extracted fibre as a result of applying the new technology. Irrespective of increased share of long fibre in the total extracted fibre, the quality parameters will also be improved.

The experience gained with the degumming device, which equipment worked in the periodic mode, allowed for designing and constructing a model module device for degumming fibre in the continuous mode – Fig. 6. This enabled to carry out the studies in the

Figure 6. The device operating in continuous degumming models.

semi-technical scale. The studies aimed at investigating the degumming process depending on various parameters of physical factors, time and analysing the process in terms of economic feasibility and quality. This device is suitable also for degumming of "green" decorticated bast fibres. This device is suitable also for degumming of green decorticated bast fibres.

This osmotic degumming method has received international awards: Geneva INVENTIONS, Brussels EUREKA, Korea Invention Promotion Association, Poland IWIS, Nurnberg IENA, and China INVENTOR FESTIVAL. The method is patented as an international patent.

Conclusion

Retting of lignocellulosic bast fibrous plants is very important in the degumming process and extracting bast fibres with high homogeneity and fineness. The technology of osmotic degumming of fibres from bast plants is very eco-friendly – it will allow for elimination of environmentally harmful wastes and unpleasant smell, which are present in traditional retting process. The osmotic degumming enables to obtain fibres characterized with good fineness, strong, soft in touch and whiteness. Applying temperature between 30–40°C of the process is beneficial for the breaking tenacity and fineness of the fibre. Osmotic degumming with ultra sound treatment (20–40 kHz) accelerates this process, making it more cost effective. For economic reason, this method can be applied to degumming green decorticated fibres (about 1/4–1/3 of total mass).

References

[1] Siwcow, A. N. (1953). *Primary Processing of Bast Plants*, PWT: Warszawa, Poland.
[2] Schilling, E., & Muller, W. (1951). *Flax*, PWT: Warszawa, Poland.
[3] Zyliński, T. (1958). *Nauka o Włoknie*, PWLiS: Warszawa, Poland.
[4] Kozlowski, R., Batog, J., Konczewicz, W., Kozlowska, J., & Sedelnik, N. (2001). *J. Natural Fibres*, *45*, 85.
[5] Kozlowski, R., Batog, J., Konczewicz, W., Kozlowska, J., & Sedelnik, N. (2001). *J. Natural Fibres*, *45*, 93.
[6] Kozlowski, R., Batog, J., Konczewicz, W., Mackiewicz-Talarczyk, M., Muzyczek, M., Sedelnik, N., & Tanska, B. (2005). 11th International Conference for Renewable Resources and Plant Biotechnology, June 6–7, 2005, Poznan, Poland.
[7] Kozlowski, R., Batog, J., & Konczewicz, W. (2003). International Conference of FAO European Research Network on Flax and Allied Fibre Plants for Humanity Welfare, December 8–11, 2003, Cairo, Egypt.
[8] Kozlowski, R., Batog, J., Konczewicz, W., Mackiewicz-Talarczyk, M., Muzyczek, M., Sedelnik, N., & Tanska, B. (2006). *Biotechnol. Lett.*, *28*(10), 761.
[9] Kozlowski, R., Batog, J., Konczewicz, W., Mackiewicz-Talarczyk, M., Muzyczek, M., Sedelnik, N., & Tanska, B. (2006). *J. Natural Fibers*, *3*(2/3), 113.
[10] Kozlowski, R., & Tabisz, J. (1967). *Practitioners Library, IPWL, 3/67*, Poznan, Poland.
[11] Sharma, H. S. S., & Van Sumere, C. F. (1992). *The Biology and Processing of Flax*. M Publications: Belfast, Northern Ireland.
[12] Szalkowski, Z., et al. (1978). *The Textile Engineer's Manual*, WNT: Warszawa, Poland.
[13] Kessler, R. W., Becker, U., Kohler, R., & Goth, B. (1998). *Biomass Bioenergy*, *14*(3), 237.
[14] Bozzini, A. (2010) [private communication].
[15] Konczewicz, W., Kozlowski, R., Heller, K., & Byczynska, M. (2006). International conference on NATURAL FIBRES: Vision 2020, December 8–9, 2006, New Delhi, India.
[16] Konczewicz, W., & Kozlowski, R. (2007). In: *Textiles for Sustainable Development, Chapter 9*, Nova Science Publishers: New York, USA, p. 95.
[17] Kozłowski, R., Batog, J., Konczewicz, W., Mackiewicz-Talarczyk, M., Muzyczek, M., Sedelnik, N., & Tanska, B. (2005). *Len i Konopie*, Polska Izba Lnu i Konopi: Poznan, Poland, p. 5, 27.
[18] Konczewicz, W., & Kaniewski, R. (2008). *Przemysl Chemiczny*, *87/12*, 1206.
[19] Konczewicz, W., Kozłowski, R., Kryszak, N., & Nowaczkiewicz, E. (2009). 10th International Cotton Conference: Natural Fibres-Their Attractiveness in Multi-Directional Applications, 3–4 September 2009, Gdynia, Poland, 92.