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

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In this article the review of the methods of lignocellulosic plants degumming is presented. Generally, for the degumming, the following 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 of lignocellulosic fibres; review; osmotic degumming

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 fibre plants. The main component of the fibres is cellulose, while secondary components are lignin, hemicelluloses, pectins, fats and waxes [1–5]. The chemical composition of the fibres is presented in Table 1.

Pectins play an important role in the fibre as a component that binds the fibre into bundles and also determine the luster and touch of the fibre. Pectins are macromolecular compounds of poly-galacturonic acid. They are agglomerated in the middle blades mainly in the above ground plant tissues. 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

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Table 1. Chemical composition (%) of lignocellulosic 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

fibre consists of cells bonded with each other by a lamella, which in turn consists of mostly pectin B with a small amount of pectin A. Fibre bundles, located in the phloem, form rings around the stem, of more or less compact structure, attached to adjacent tissues with a layer of pectin A.

Proper removal of pectin substances during preliminary processing determines the divisibility and, what follows, the fineness of the fibre and its suitability for spinning.

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 the unpleasant touch of fibre, it 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. The presence of lignin is undesirable while processing the fibre as it worsens the touch and flexibility of the fibre. Lignin causes that the fibre is more coarse easily breakable and mechanical parameters such as tenacity and resiliency deteriorate. Moreover, the divisibility of the fibre is reduced 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 e.g.: biological, chemical, mechanical and physical.

Application of proper extraction method will result in fibre with required length, thinness and 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 preliminary processing of raw material i.e. straw (by dew retting, water retting, etc.) followed by enzymatic, chemical or physical processing of fibres. [4–11]

The scheme of the fibre plant degumming methods is shown in Figure 1.

Biological methods. Traditional biological methods, referred to as retting, include dew retting and water retting.

The traditional method of separating fibre from the woody parts of the plant, its extraction and separating from the non-cellulosic components was **dew retting** done in the field. This method is still used in the present days, mainly for economic and environmental

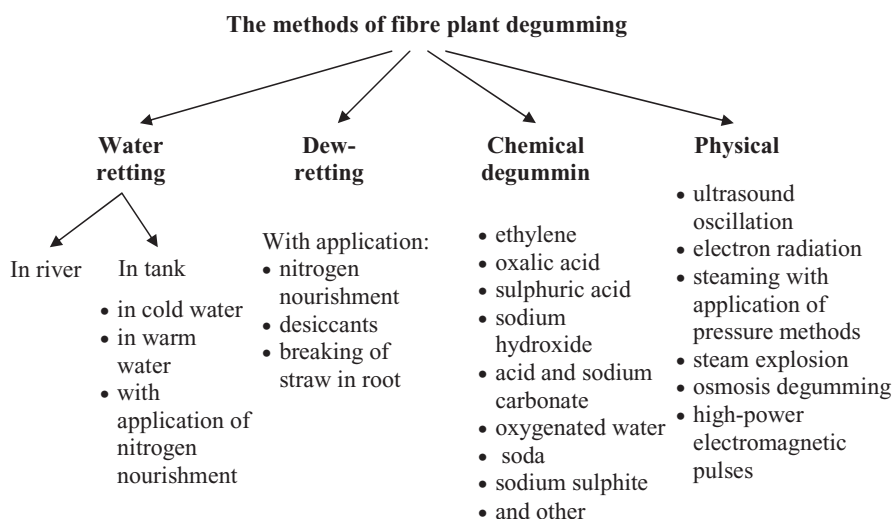


Figure 1. The methods of fibre plant degumming.

reasons – there are no 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 referred to as plant glues. Such retting process is difficult to control and the quality of the fibre varies a lot, depending on atmospheric conditions (season, temperature, air humidity, rainfall, etc.).

Water retting is conducted in retting tanks or natural water reservoirs, in both warm and cold water. Retting in warm water was first described by Szenk in Ireland in 1846, after that the method quickly spread across Europe. The process involves biochemical phenomena occurring in presence of bacteria. Microorganisms cause fermentation of pectin substances and thus separate fibres from the woody parts of the stem. Depending on the retting conditions and the type of pectinolytic organisms, this process can have aerobic or anaerobic character. Retting in warm water instead of cold, allows for reducing the time of the process in controlled conditions (temperature and pH) and conducting it all year round. Water retting conducted with warm water takes between 70 to 100 hours.

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

In order to create optimum conditions for the growth of the “retting” organisms and accelerate the decomposition process of pectins, mineral nutrition was applied (phosphoric, potassium, ammonium salts and urea) or ferments, as well as fluid recirculation, double retting, bedding made of retted tow and enzymes. These treatments are aimed at faster and simultaneous separation of fibres from the woody part, shorter degumming time and better quality of fibre, mostly by improvement of divisibility and fineness of the fibres. The classic warm water retting technology applied earlier in retting industry for flax straw, required large amounts of water and heavily contaminated retting wastes were produced at all stages of the process. The retting wastes included the following: after-retting wastes, polluted water after rinsing the straw retted in the tanks and wastes after pressing water out from the straw. 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, propionic, butyric and valeric). 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 use enzymatic treatment, chemical and physical methods.

Chemical 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 few hours), yet they may result in lower fibre strength parameters and worsening of the 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 to the environment and the need for purchasing of special devices to run the process.

Enzymatic methods. In order to accelerate the retting process, trials to use biologically active substances have been conducted covering pure enzymes characteristic in terms of



(a)



(b)

Figure 2. Warm water retting in Egypt.

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, cellulase and hemicellulase. In the retting process, enzymes from hydrolase group are of the 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 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

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 glycosidic 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

of the 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 methods. 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 enhanced by high moisture content in 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, further softening with a semi-circular squeezer and fibre batching with detergent were conducted after steaming.

This process of flax straw was studied by R.W. 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 short staple flax fibres for use in textile and technical composites.

A new retting technology developed by Migoni and Alessandro Bozzini [14] can be done by using special **electric resonance electrodes** immersed into a water pool, possibly with the addition of specific pectinolytic enzymes, to the water 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 was concentrated on a new **osmotic method** 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

method is patented as an international patent (submission numbers: PCT/PL2006/0000/85 and PCT/PL2008/000081).

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. Peripheral tension – as usually in case of material strength – is twice larger than the longitudinal ones and results 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. This causes dramatic decrease of breaking strength of the gelled pectins that bind the fibre occurs. 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 physico-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.

Experimental

The raw material used for the study was flax straw of AGATA (NL) variety.

The experiment was conducted in laboratory scale – Figure 3. The experiments comprised testing the effect of osmotic degumming parameters i.e. temperature, time and flow of water on quality of flax fibre. 100 g samples of the raw material were used for the tests. The degumming process was carried out at temperature: of 20; 25; 30; 35; 40 and 50°C and time degumming: 24; 48; 72 and 96 h. The experiments were conducted by using the constant, regular and controlled water flow. The reaction of the liquid was maintained at pH 7. After degumming, the straw was scutched and shaken (separation of fibre from the woody part) using the laboratory equipment.

The fibre was subjected to tests: metrological: fineness (tex) acc. to PN-86/P-04676 and tenacity (cN/tex) acc. to PN ISO 1973 and chemical analysis was done to determine the content of: lignin (%) acc. to BN-86/7501-11 and pectins (%) – according to a method developed at INF.

In the next step of the study, the results were confirmed by research conducted on new devices (a periodic and continuous mode) for osmotic degumming – patents No. PCT/PL2006/0000/85 and PCT/PL2008/000081).

Results and Discussion

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. Figure 3 shows laboratory scale for evaluation of fibre content. This is especially important in evaluating progress in breeding new cultivars of fibrous plants.

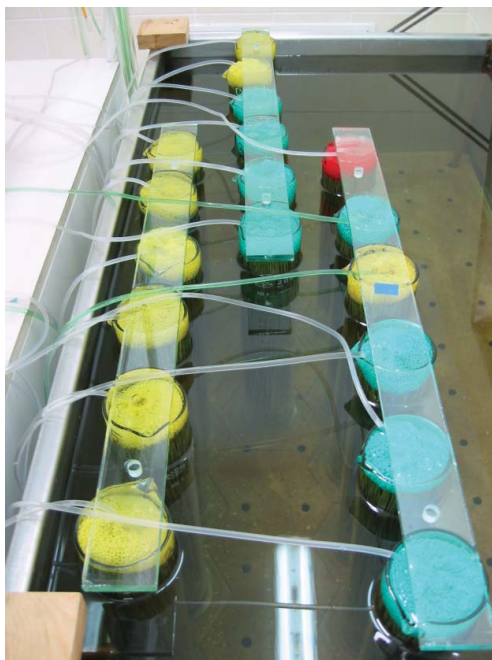


Figure 3. The laboratory evaluation of fibre content.

Figure 4 presents the influence of technical factors of degumming process on the fibre content and the results of statistical analysis.

For all tested factors total fibre content varied between 36.71-48.86% – Figure 4. The highest fibre content (48.86%) was observed for degumming temperature 25°C.

Temperature of 20°C turned out to be insufficient for adequate degumming of fibres, thus the fibre content was not evaluated. During mechanical processing shive cling to the fibre and the sliver does not exhibit capacity for splitting – Table 3. Applying higher

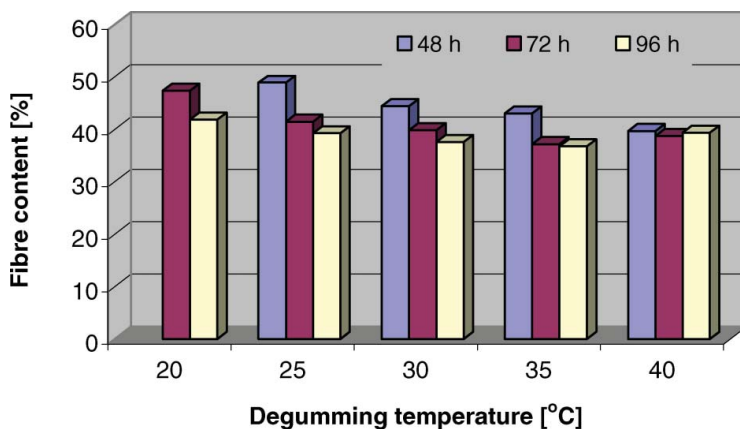
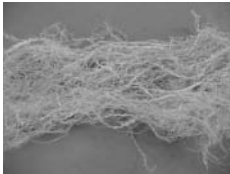
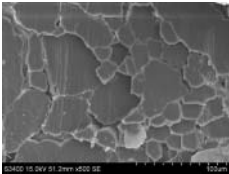
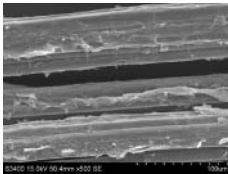

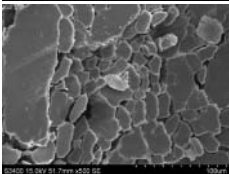
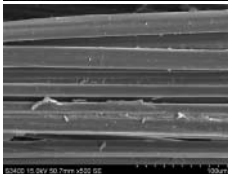

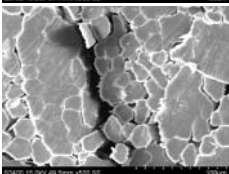
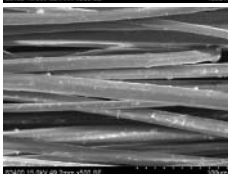


Figure 4. The effect of degumming temperature correlated with the time of degumming on the fibre content.

Table 3. Macro- and micro- views of fibre.

Technological parameters of degumming process	Macro-views	Cross-section	Longitudinal view
20°C 96 h			
30°C 96 h			
40°C 96 h			

temperatures i.e. 30-40°C allow to obtain fibre soft in touch in divided slivers. Further temperature increase to 50°C results in shives not being separated in mechanical processing. Therefore, for this temperature fibre content was not evaluated as well.

Increase of degumming temperature caused decrease of fibre content. The lowest fibre content i.e. 36.71% is observed at the temperature of 35°C and 96 h of the process.

It is known that the fibre content is not correlated with the fibre quality. In the degumming process, the woody part is separated from the fibre, what is caused by the removal of pectins and partially also lignins from the fibre. This leads to improvement of fibre quality, mostly by increase of divisibility and fineness of the sliver. This implies the need for further evaluation of the fibre – metrological and chemical.

The effect of process parameters on tenacity and linear mass are shown in Figure 5 and Table 4.

Table 4. The effect of technological factors on linear mass of the fibre.

	Linear mass [tex]		
	Time [h]		
	48	72	96
Temperature [°C]			
40	0.98	0.84	0.76
35	0.92	0.92	0.84
30	1.26	1.00	0.82
25	2.04	1.12	0.94

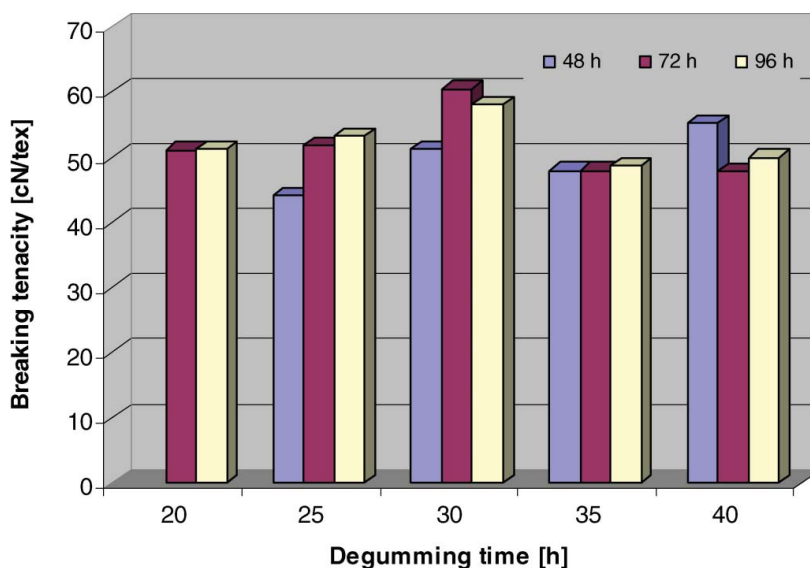


Figure 5. The effect of technological factors on breaking tenacity of the fibre.

The laboratory studies led to the conclusion on the relations between the temperature, degumming time and fibre quality.

From the other side, based on the results of metrological evaluation, elongation of degumming time and higher temperature of the process produced the fibre of better metrological parameters – Figure 5 and Table 4.

The fibre of the best in touch was that obtained at temperature between 30-40°C and degumming time 72 and 96 h. The fineness reached 0.76-0.84 tex whereas breaking tenacity – 48.64-57.98 cN/tex.

The fibre characterized by the best achieved parameters, i.e. fineness of 0.76 tex, was obtained after 96 hours of the process and the at temperature of 40°C.

In case of tenacity, the statistical analysis was proven only for temperature of 30°C. This temperature and degumming time of 96 h allowed for extracting fibre with the highest tenacity i.e. 60.46 cN/tex – Fig. 5, what was confirmed by statistical analysis. From Figure 5 one can conclude that degumming time shorter than 48 h brings about rectilinear rise of tenacity, while elongation of the time to 72-96 h causes the reduction of tenacity.

The effect of technological factors of degumming process on lignin and pectin content are presented in Figure 6 and Table 5.

Drawing from the results obtained for the tested technological parameters (Table 4) it was observed that the highest pectin content reached 5.11% at 25°C and 96 h of the process. The lowest value was achieved (0.66%) at temperature of 40°C and the time of 96 h.

Regarding lignin content in flax fibre, the studied parameters did not have a significant effect on this value. However along with the temperature increase, rectilinear decrease of lignin content was observed – Figure 6.

In the next step of the study, the results were confirmed by research conducted on new devices (a periodic and continuous mode) for osmotic degumming.

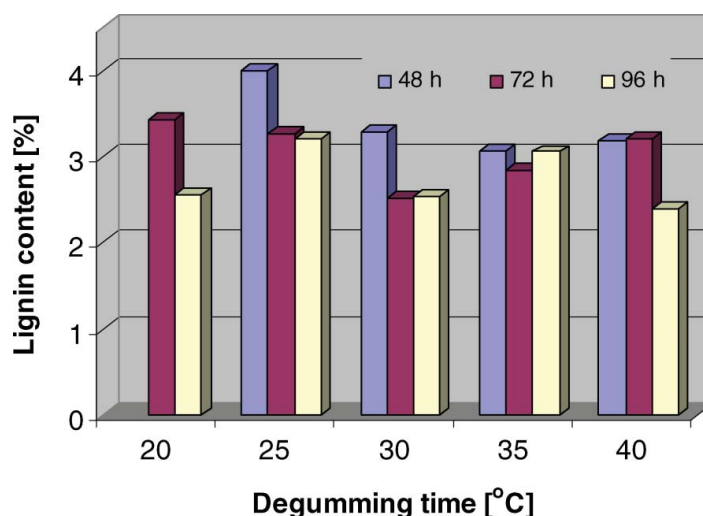


Figure 6. The effect of technological factors on lignin content in the fibre.

The INF&MP (Poznan, Poland) in cooperation with IT&E (Lodz, Poland) has built a device that runs the degumming process in a periodic (Figure 7) and continuous mode (Figure 8).

In the periodic mode the device operation involves the flow of soft water through a straw batch of fibrous plants. The intensity of the degumming process is controlled by the rate of water flow, water temperature and the duration of the process. The technological parameters can be set and recorded *on line* automatically. The process control is fully automatic.

The optimized process parameters have been test-run with the use of specially constructed device that runs fibre degumming in the periodic mode – Figure 7.

The studies allowed for determining the parameters that yielded the optimal quality of flax fibre at the optimal capacity of the device i.e.:

- temperature: 30°C
- time of process: 72 h
- ultrasound: 20 kHz

Table 5. The effect of technological factors on pectin content in the fibre.

Temperature [°C]	Pectin content [%]		
	Time [h]		
	48	72	96
40	1.89	2.65	0.66
35	2.43	2.52	2.68
30	3.87	2.62	2.33
25	5.11	3.15	4.26






Figure 7. The device operating in periodic degumming models.



Figure 8. The device operating in continuous degumming models.

Table 6. Flax fibre after osmotic degumming.

	
Long flax fiber	Hackled flax fiber
Fineness 0.91 [tex]	Fineness 0.71 [tex]
Breaking tenacity 62.79 [cN/tex]	Breaking tenacity 50.13 [cN/tex]
Hackled flax fiber – sliver	
Fineness 0.54 [tex]	
Breaking tenacity 33.75 [cN/tex]	

- batch size – 12-18 kg of flax straw
- total volume of technological liquid inside the device is 0,85 m³
- electric supply – 3 × 380/220 V and maximum power consumption 4,5 kW
- capacity of chemical pump (250PS) – 100 l/min. power consumption 0,25 kW
- device size: 1,6 × 1,4 × 2,6 m
- materials – acid proof steel and fittings

The experience gained during the study when equipment worked in the periodic mode, allowed for designing and constructing a model module device for degumming fibre in the continuous mode – Figure 8.

In continuous mode the line consists of: a feeder, a stem breaker, processing chambers with wringers and ultrasound generator and a system of conveyor belts for moving the raw material. The chambers are equipped with: an anionic column, an additional water container, pumps and filter set and a UV lamp.

The studies allowed for determining the parameters that yielded the optimal quality of flax fibre at the optimal capacity of the device i.e.:

- temperature: 30°C
- time of process: 48 h
- ultrasound in the successive baths: I-20 kHz, II-25 kHz, III-30 kHz
- number of working cycles for the crushing and wringing rollers: 6/1 transporting cycle
- capacity 15 kg of straw/h
- water consumption 30 l/h
- power consumption 0.6 kWh/kg of straw

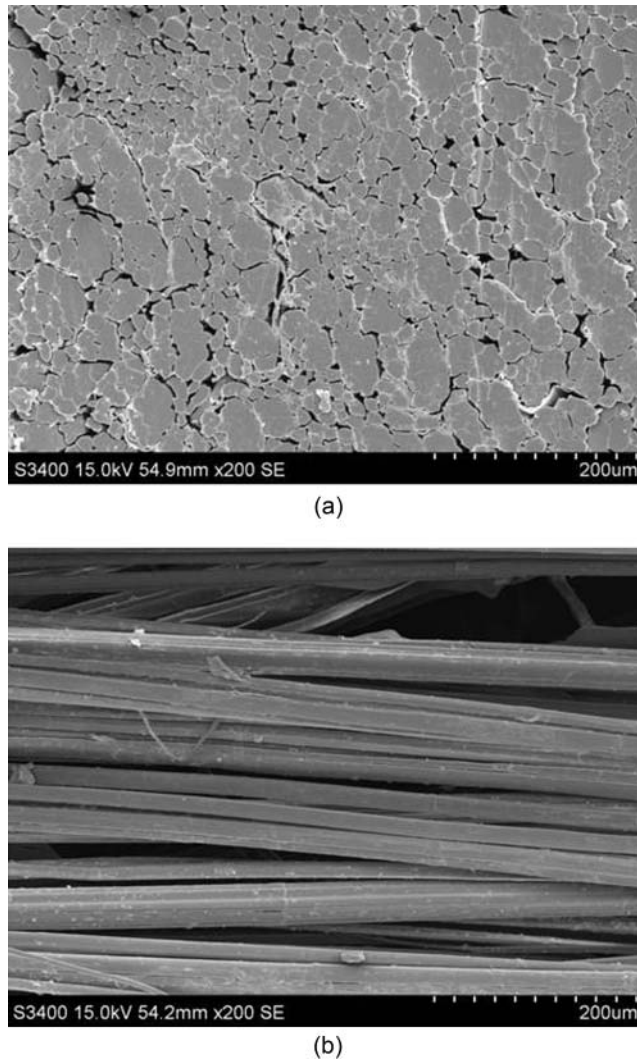


Figure 9. Crosssection (A) and longitudinal (B) images of the fibre after osmotic degumming.

The studies conducted with the use of specially designed devices, confirmed the obtained results in laboratory scale. The best in touch fibre was obtained at temperature between 30-40°C and degumming time of 72 and 96 h. The fineness reached 0.76-0.91 tex whereas tenacity – 48.64-57.98 cN/tex. 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 6. Cross-section (A) and longitudinal view (B) of the fibre is presented in Figure 9.

The new technology of degumming Fibres from bast plants is definitely ecofriendly as it allows for eliminating wastes and unpleasant odour. The water retting extraction processes are very harmful for the environment. Preventing the degradation of flax fibre, which occurs during the retting process, will allow 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.

This allowed to carry out the studies in the 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. The osmotic degumming method has received international awards: Geneva INVENTIONS, Brussels EUREKA, Korea Invention Promotion Association, Poland IWIS, Nurnberg IENA, China INVENTOR FESTIVAL.

Conclusions

1. Retting of lignocellulosic bast fibrous plants is very important for extracting bast fibres with high homogeneity and fineness.
2. 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 water retting process.
3. The osmotic degumming allows to obtain strong fibres characterized by good fineness,, soft touch and whiteness.
4. Applying temperature between 30-40°C of the process is beneficial for the tenacity and fineness of the fibre.
5. Osmotic degumming with ultrasound treatment (20-40 kHz) accelerates this process, making it more cost effective.
6. For economic reason, this method can be applied to extract green decorticated fibres (about 1/4 – 1/3 of total mass).

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