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# Cottonisation of Decorticated Flax Fibres

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## Abstract

The commonly used flax process of decortication allows the mechanical extraction of fibre from plant stems without prior retting. The one-type fibre obtained in this process is characterised by very low quality, as it is poorly divided, has high linear mass and high amounts of impurities. This paper presents a description of a newly developed method of obtaining high quality flax cottonized fibre from low quality decorticated fibre by application of a wet degumming process for fibre. The experiment involved studying the parameters of flax fibres after each step of the technological process i.e. after decortication, wet degumming and final mechanical cottonisation. The study covered tests of the following fiber parameters: linear mass, length, impurities, chemical composition as well as thermogravimetric analysis, Fourier transform infrared spectroscopy analysis and scanning electron microscopy images. The results confirm the efficiency of the method applied for obtaining high quality fibre from decorticated flax fibre.

**Key words:** decorticated fibres, fibre elementarization, cottonization, fibre degumming, degumming device.

treatment is applied, the fibre is dried and finally mechanically cottonised.

Decortication, commonly used for flax, allows for the mechanical extraction of fibres from plant stalks without the retting process [1-3]. During decortication, fibre is separated from the woody parts of the stalk, including shives, and the initial division of fibres and shortening takes place. The one-type fibre obtained in such a way is characterised by very low quality: it is poorly divided, has high linear mass, and a lot of impurities. Therefore decorticated fibres have limited applicative potential and are used only for technical purposes, where high quality is not a prerequisite. Decortication, despite its drawbacks, is used because it eliminates the long dew retting process (several weeks) and allows for substantial shortening of extraction and processing technologies of fibre. The traditional process of retting fibrous plants in tanks filled with water has been abandoned in Europe due to its strong negative impact on the environment.

One of the solutions proposed so far is the cottonisation process for flax fibres, carried out mechanically for the fibre extracted with the dew retting method, with the retting lasting 3 to 7 weeks on average, depending on the weather conditions [2-4]. Dew retted fibre is of good quality and divides easily from the woody parts of the stalks into smaller fibre complexes; therefore using mechanical cottonisation brings about positive results. This way, which makes use of the traditional retting technique, leads to obtaining long and short fibres. The drawback of dew retted fibre, however, is the strong relation be-

tween weather conditions and fibre quality, which makes the reproducibility of fibre parameters impossible.

The new method of obtaining high quality cottonised flax fibres from low quality decorticated fibres enables to speed up the technological process by elimination of the time consuming retting and makes the process independent of weather conditions. The elimination of dew retting is an important advantage in the management of arable land, as in the solution proposed straw is taken from the field directly after cutting or pulling, while in the case of dew retting the straw is left on the field for 3 to 7 weeks, which prohibits any other field work at that time.

The existing methods of flax fiber cottonization have been applied mainly for noils coming from dew retted fibers, which means that the cottonization process is used for fibers characterised by good quality at the initial stage, contrary to the low quality of decorticated flax fibers. There are few methods of flax cottonisation, the most often used being the mechanical process. Several researchers have developed enzymatic treatment for the cottonisation of dew retted fibers [5-9]. There are no available scientific publications describing the cottonization of decorticated flax fibers.

The novelty of the technology proposed lies in the pioneering use of a wet process after decortication for the degumming of one-type fibre with the use of a closed device with adjustable pressure, which allows to obtain high quality cottonised flax fibres, which so far has been impossible from decorticated fibres [10].

## Introduction

The paper presents a new method of obtaining high quality cottonised flax fibres from low quality decorticated fibres by elementarisation/cottonisation conducted with the use of the degumming process under conditions of liquid flow in a closed device with adjustable pressure. The new method of obtaining high quality cottonised flax fibres from low quality decorticated fibres involves a few consecutive processing steps, set in a specific sequence i.e. after decortication wet

## Materials and methods

The initial material for the experiment was flax straw of the Modran and Nike varieties, harvested by pulling by Polish farms in 2014. The flax straw was decorticated, which resulted in obtaining poorly divided one-type fibre with a high content of impurities. In order to produce high quality fibres, the osmotic degumming process was applied in a closed device with adjustable pressure, followed by ultrasound processing in an open device. After wet processing, mechanical treatment was applied for the elementarization/cottonization of decorticated fibres. Ultrasounds were used only at the wet degumming stage, while the final cottonization was carried out mechanically without using any additional devices. The Figure below shows the course of the treatments (Figure 1).

At stage I of the experiment, flax straw of the Modran and Nike varieties was subjected to the mechanical process of decortication on a technological line for decortivating bast fibres for the textile industry. The decortication leads to obtaining one-type fibre. The technological line for bast fibre decortication consists of several devices combined in a line that performs alternate cycles of breaking, shaking and scutching. The decortication process of the Modran and Nike varieties was conducted at the Experimental Plant of INF&MP 'Lenkon'.

At stage II of the experiment, decorticated fibres in the form of reeled sliver were subjected to the wet degumming process

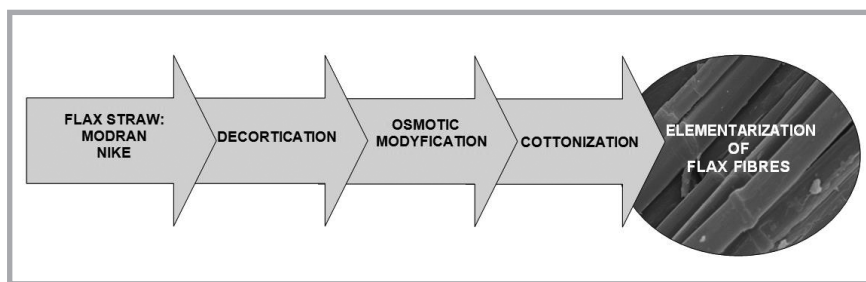


Figure 1. Course of treatments in the technology of producing flax fibre with alternative quality.

in a closed device with liquid flow at the maximum speed for the device at 30 °C for 24 hours (Figure 2). After that stage the fibre was degummed further in open degumming devices (in water at 30 °C) equipped with an ultrasound generator of 24 kW constant power. The fibre batches moved at a speed of 6 m/h (Figure 3). The device developed is a prototype and its productivity is about 100 kg/day. The degummed fibre was dried and then mechanical cottonization (stage III) was carried out on a carding machine adapted to the needs of the technology developed in order to elementarise it and adjust the parameters to values typical of cotton fibre.

The main material for the study was flax fibre obtained after each stage of processing in the experiment. All types of fibres were subjected to quality tests and their chemical composition determined.

## Methods

In the experiment, flax fibre of the Modran and Nike varieties was tested after each processing stage i.e. after decortication (I), after wet degumming (II) and

after final mechanical cottonisation (III). The linear mass, length, impurity content and chemical composition were determined, and also a Thermogravimetric study (TGA), Fourier transform infrared spectroscopy (FTIR) and Scanning Electron Microscope (SEM) analyses were conducted.

The following testing methods were applied for fibre evaluation:

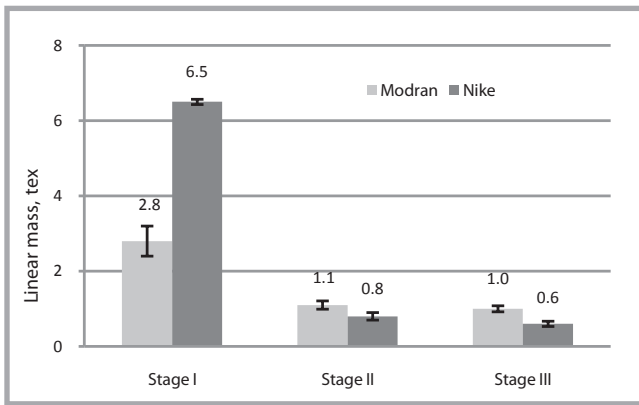
- Linear mass of fibres (tex) according to PN-EN ISO 1973:2011.
- Length (mm) according to PN-ISO 6989:2000.
- Wax and fat content (%) according to BN-86/7501-10.
- Lignin content (%) according to BN-86/7501-11.
- Pectin content (%) tests were conducted by a gravimetric method according to a technique developed at INF&MP. The percent share of pectins was determined by dissolving them in ammonium citrate, then precipitating from the solution with calcium chloride, and finally measuring the weight



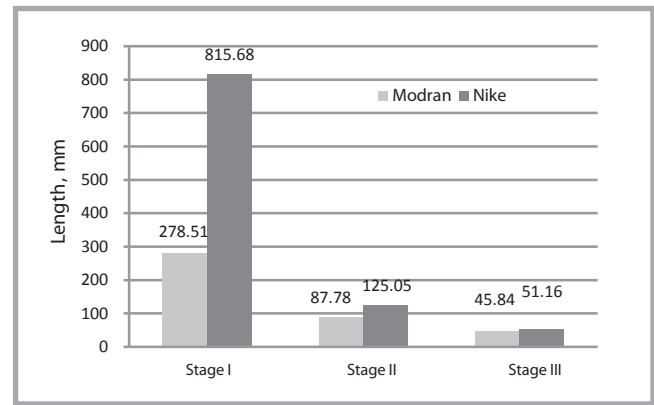
Figure 2. Closed device with liquid flow (rate of water flow 30 l/min) produced by the Institute for Sustainable Technologies, Radom.



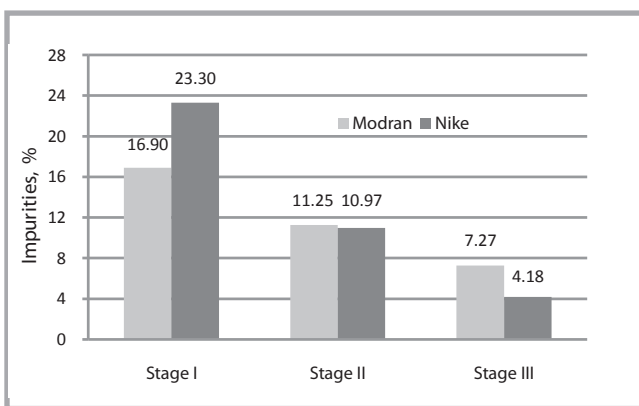
Figure 3. Degumming in an open devices with water and ultrasound. Batch size: 4.5 m x 1.5 m x 0.6 m, produced by the Institute for Sustainable Technologies, Radom.



**Figure 4.** Linear mass of flax fibres of Modran and Nike varieties after each processing stage.



**Figure 5.** Length of flax fibres of Modran and Nike varieties after each processing stage.



**Figure 6.** Impurity content of flax fibres of Modran and Nike varieties after each processing stage.

of the calcium pectinate precipitated from the solution.

- Hemicellulose content (%) according to BN-77/7529-02.

Moreover we conducted the following:

- Determination of the impurity content in the fibre according to the decree of the Minister of Agriculture and Rural Development of 5th May 2011 on the method of determining the percentage content of impurities in short flax fibre or hemp fibre.
- Microscopic test – photos of longitudinal views and cross sections of flax fibres were taken with a S-3400N SEM (produced by Hitachi, Japan) in the high vacuum mode (a secondary electron detector SE). The fibres were sprayed with a gold layer prior to the tests. For both the longitudinal views and cross sections, the following parameters were set: magnifications of x250, working distance 20 mm and the value of accelerating voltage 15-20 kV.
- Thermogravimetric study (TGA) – performed with a TA Instruments, Analyser Q50, produced by Thermo Scientific, USA. A test sample (about 20 mg) was subjected to heating with-

in the temperature range 30 to 650 °C and at a heating rate of 15 °C/min in nitrogen atmosphere at a constant gas flow rate of 90 mL/min.

- Fourier transform infrared spectroscopy (FTIR) – was performed on a TA Instruments, iZ10 model, produced by Thermo Scientific, USA. The spectrum of the released gases contained 32 scans per second at a resolution of 4 cm<sup>-1</sup> within the range from 600 to 4000 cm<sup>-1</sup>.

## Results and discussion

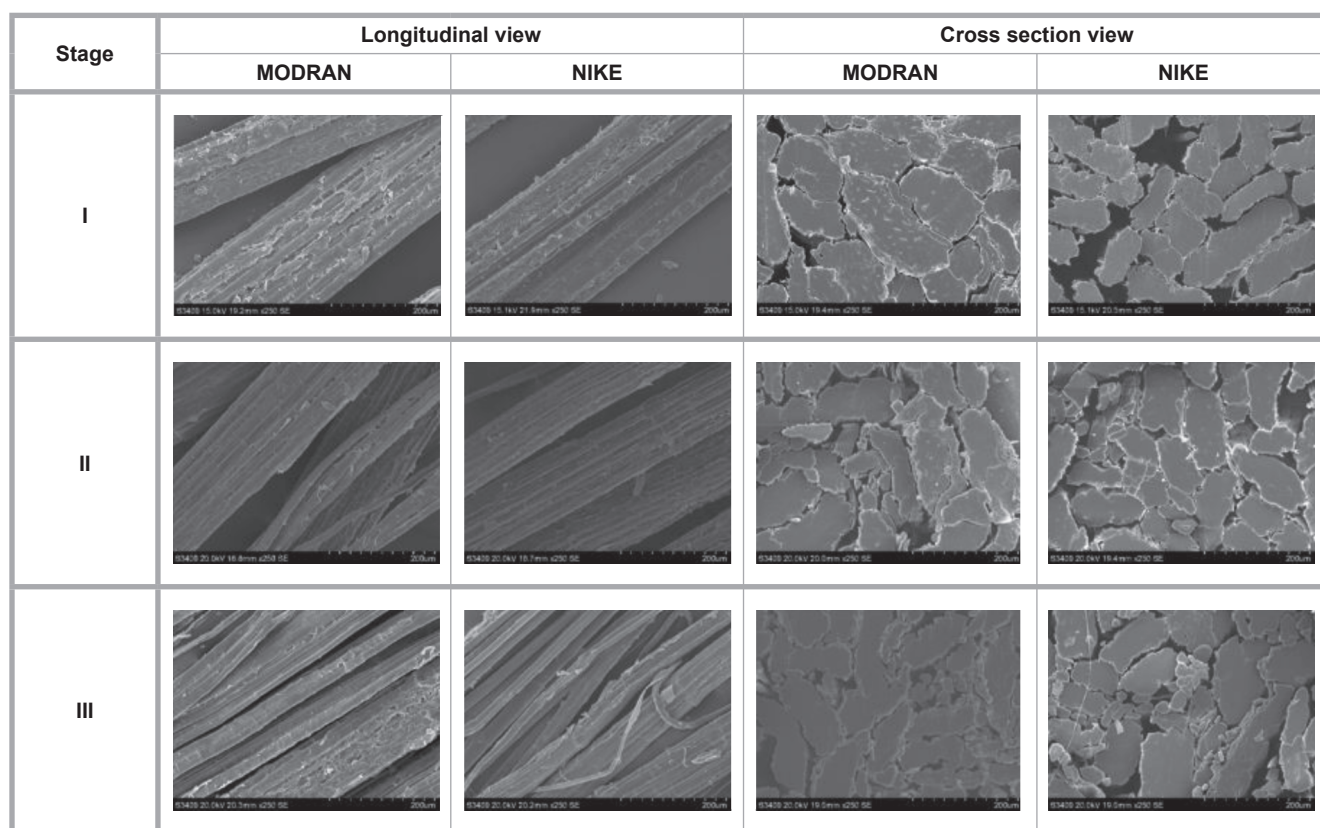
In order to obtain high quality cottonized flax fibres from low quality decorticated fibres, a physical degumming method in a water medium was applied. The degumming process of fibrous plants is known [11], based on using physical phenomena such as the diffusion and osmosis that occur in fibre in contact with water. The solution presented in this paper involved the physical degumming of the fibre in water by the action of forced water flow, temperature, ultrasounds and by the hydrodynamic action of water on the fibre. Thus by increasing the process

temperature faster, the removal of pectins, which glue the fibres together, was achieved; the use of ultrasound forced water flow, and the hydrodynamic action of water led to better removal of the leached substances.

### Metrological and microscopic analyses

The fibre after each stage of the technological process, i.e. after decortication (I), after wet degumming (II) and final mechanical cottonization (III), was subjected to metrological and microscopic analyses. **Figures 4-7** present graphically the results of the analyses

**Figure 4** shows the effect of the method developed on the linear mass of decorticated flax fibres of the Modran and Nike varieties. The initial average linear mass for the Modran variety was 2.8 tex. After degumming in the closed device and in the open device equipped with ultrasounds, the fibre was thinned to a linear mass of 1.1 tex. At the last cottonisation stage the fibre was further divided and reached the final value of linear mass of 1 tex. For the Nike variety fibre. the initial average linear mass was 6.5 tex, and after the wet degumming it was 0.8 tex, while after cottonization it reached 0.6 tex. Such a considerable decrease in the linear mass for both varieties indicates that the wet process of degumming allowed for the division of thick fibre complexes into elementary fibres and fibre complexes of very low linear mass, which was also visible in the microscopic analysis of longitudinal and cross section views of the fibre after each processing stage (**Figure 7**). This proves that the technology developed for the cottonization of decorticated flax fibres causes the efficient thinning of the fibre to parameters similar to those of cotton fibres.



**Figure 7.** Longitudinal and cross section views of flax fibres of Modran and Nike varieties after each processing stage.

The method developed led to a significant reduction in average fibre length of Modran and Nike fibres, as can be seen in **Figure 5**. The initial average fibre length for Modran was 278.51 mm; after the wet degumming it was reduced to 87.78 and after cottonization to 45.84. In the case of Nike, the initial average fibre length was 815.68 mm; after stage (II) the fibre was shortened to 125.05 mm, and after cottonization it reached an average length of 51.16 mm, which is similar to the length of cotton fibres.

Similar to changes in the linear mass and fibre length as a result of applying the technology developed, after each processing stage a significant reduction in the impurity content took place for both Modran and Nike fibres (**Figure 6**); for Modran – from an initial value of about 17% to a final value of about 7%, and for Nike – from about 23% to about 4%, while a value of 4% is satisfactory.

### Chemical analysis

Cellulose is the main component of plant fibres, and it is often found with the following substances: waxes and fats, hemicellulose, lignin and pectins. The percentage share of specific substances in the fibre may vary depending

on the plant variety. It is known that each extraction process, such as decortication, water retting, dew retting as well as enzymatic, chemical, physical or any other treatment of fibre affects, to varying degrees, the degumming of fibre from the woody part, which is caused by the reduction in pectins, lignin, hemicellulose, waxes and fats in the fibre itself, leading to the removal of impurities and the division of fibre slivers into smaller thinner complexes. Therefore fibre parameters such as: length, linear mass, tenacity, homogeneity and efficiency depend on the extraction technique [12]. The results of chemical analyses of the fibre composition after each stage of the processing are presented in **Figures 8-12**.

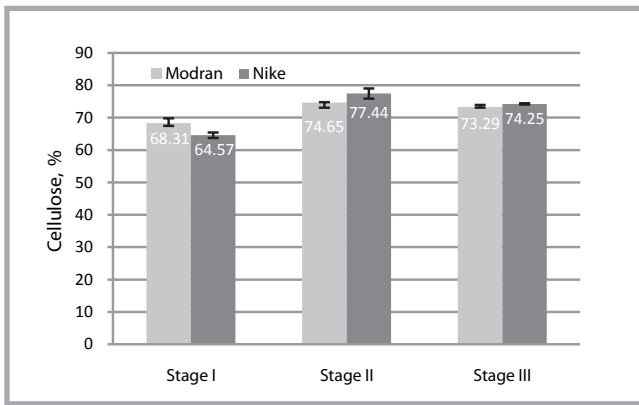
In the case of the cellulose content (**Figure 8**) in the flax fibres, the initial value reached 68.31% for Modran and 64.57% for Nike. The application of wet treatment of the fibre affected the removal of the non-cellulosic component ( e.g. lignin), which contributed to a relative increase in the cellulose content up to 74.65% for Modran and 77.44% for Nike. The cottonisation that followed did not significantly affect the levels of cellulose in the fibre, where the value for Modran fibres was 73.29% and for Nike

fibres 74.25%. The effect was similar for the lignin component.

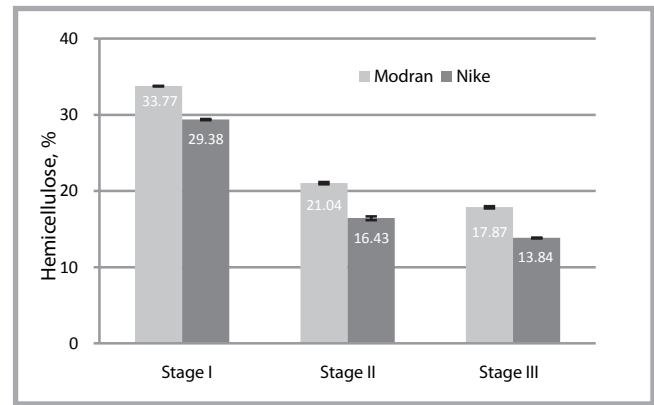
The hemicellulose content (**Figure 9**) in the decorticated fibres (I) was 33.77% for Modran and 29.28% for Nike fibres. The application of further stages of the processing decreased hemicellulose levels to the following:

- For Modran fibres 21.04 in stage II and 17.87% in stage III
- For Nike fibres 16.43% in stage II and 13.84% in stage III.

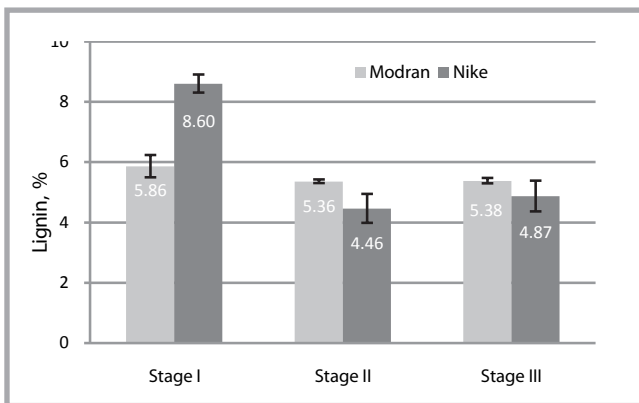
The lignin content in the decorticated fibres (I) was 5.86 % for Modran and 8.60% for Nike (**Figure 10**). The use of the further processes i.e. wet degumming (II) caused the lowering of lignin levels to 5.36% for Modran and 4.46% for Nike. However, the last stage (III) of the processing, i.e. mechanical cottonization, did not have an effect on lignin removal from the fibre, which might indicate the non-invasive activity of the process for the lignin present in the fibres. Biologically lignin in a plant plays an inlaying role in the areas of amorphous cellulose, making it stiff. In the technological process of obtaining fibre, lignin is a unwanted substance, which worsens the handle and fibre elasticity, making



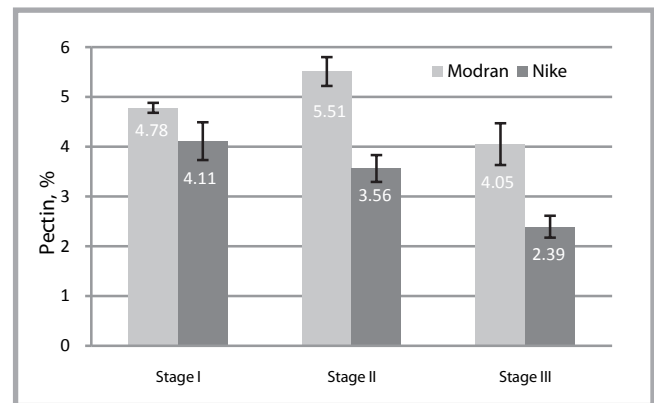
**Figure 8.** Cellulose content in flax fibres of Modran and Nike varieties after each processing stage.



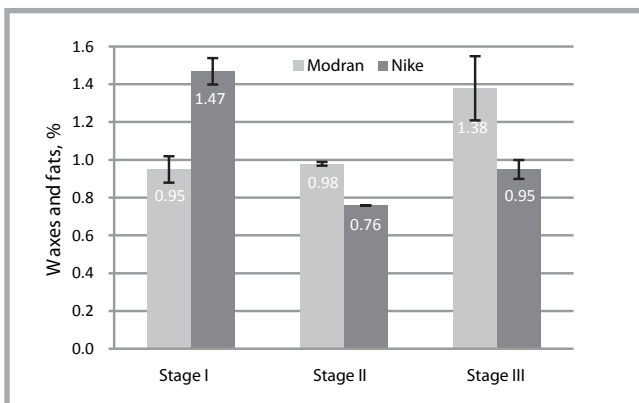
**Figure 9.** Hemicellulose content in flax fibres of Modran and Nike varieties after each processing stage.



**Figure 10.** Lignin content in flax fibres of Modran and Nike varieties after each processing stage.



**Figure 11.** Pectin content in flax fibres of Modran and Nike varieties after each processing stage.



**Figure 12.** Wax and fat content in flax fibres of Modran and Nike varieties after each processing stage.

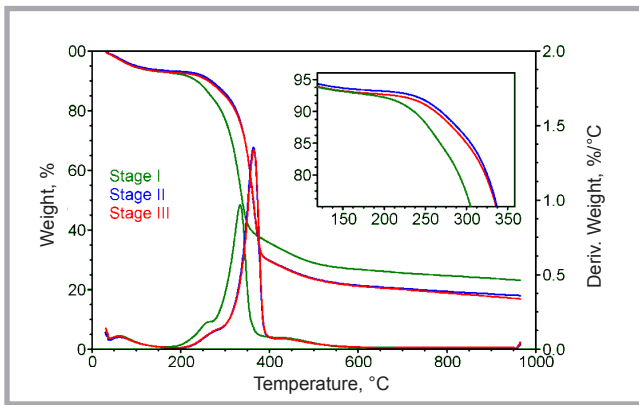
the fibre brittle, and the tensile strength and elasticity become worse. Moreover lignin content reduces fibre divisibility.

The pectin content (**Figure 11**) in the decorticated fibres (I) was more than 4%, (4.78% Modran and 4.11% Nike), while for the fibre after stage III, it was 4.05% for Modran and 2.39% for Nike fibres. Analysis of the pectin content showed that along with the next technological step, the pectin content dropped for both varieties tested. Only in the case of Modran, for the wet degumming process

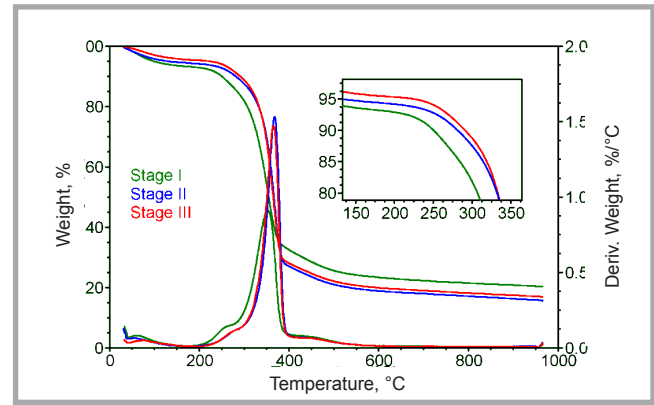
(II), did the relative percentage of pectins increase to 5.51%, which is caused by the non-invasive character of the process. It must be mentioned that pectins play an important role in flax fibre as they glue fibres together into bundles and give fibre lustre and handle. Fibrous plants contain two pectin fractions: A – the fraction soluble in water, and B – the fraction insoluble in water. Technical flax fibre consists of cells glued together with a lamella, built mainly from pectin B, and to a lesser extent from pectin A. Fibre bundles are distributed in the bast layer around the

stem, making rings, more or less compact, glued to adjacent tissues with pectin A. Thus the removal of pectin substances in the pre-treatment determines the divisibility and later the fineness of the fibre, as well as its suitability for spinning. Excessive removal of pectins will cause that the fibre will be rough, dry and unpleasant. Total pectin removal will cause the destruction of the fibre bundles into elementary fibres.

On the basis of the tests, it is clearly visible that for Modran fibres with each technological process applied, the percentage value of waxes and fats increased from 0.95% (stage I) to 1.38% (stage III) (**Figure 12**). The relative growth observed for fats and waxes resulted from the decrease in the contents of other components in the fibre. For Nike fibres the content of waxes and fats fell from 1.47% (stage I) to 0.76% (stage II), and finally to 0.95% (stage III). The results may indicate that for the Modran variety wet degumming and mechanical cottonisation does not cause the removal of waxes and fats from the fibre. In terms of the technological process, waxes and fats constitute a very



**Figure 13.** TGA/DTG curves during thermal degradation of flax fibres of MODRAN variety.



**Figure 14.** TGA/DTG curves during thermal degradation of flax fibres of NIKE variety.

important component of the fibre as they reduce the friction coefficient, facilitating the movement of fibres themselves and in working elements of the devices used for breaking, shaking, stretching and forming yarn.

#### TGA/FTIR analyses

Thermogravimetric curves (TGA/DTG) of flax fibres of the Modran and Nike varieties tested after decortication (I), after wet degumming (II) and final Mechanical cottonization (III) are presented in **Figures 13-14**. Thermal properties of the fibres tested are presented in **Table 1**.

According to the literature data [13], four stages are observed during the thermal degradation of flax fibres:

- 1st stage – temperature about 100°C, representing evaporation of water;
- 2nd stage – temperature between 185 and 300°C; the degradation is linked to hemicellulose degradation;
- 3rd stage – temperature about 350°C, linked to cellulose degradation;
- 4th stage – temperature between 250 – 600°C, which represents the slow degradation of lignin.

Analysis of the TGA curve and DTG derivate showed that for both flax varieties, the highest mass loss was observed at the 3rd stage of thermal decomposition, which is linked to cellulose, confirmed by chemical analyses. However, linking the TGA curves illustrating the mass loss and change in the signal for the remaining fibre components is not the same as for the content of these compounds determined with chemical analysis (**Figures 8-12**). Moreover thermal analysis indicated that for the fibres tested, the use of wet degumming (II) and mechanical cottonization (III) im-

proves the thermal stability of the fibre as compared with the decorticated fibres (I). For the decorticated fibres, the decomposition temperature was 297.00 °C (Modran) and 308.96 °C (Nike), whereas for the fibres from stages II and III, it increased to values between 332.75 and 335.47 °C. This relation is confirmed by fibre degradation analysis for 10% and

60% mass loss – **Table 1**. However, for a temperature of 950 °C of fibre degradation, a higher percentage value for the sample residue was observed for the decorticated fibres (stage I) rather than for the modified fibres after stage II and stage III. For Modran fibres, the mass loss of the samples was about 5.19% – 6.2% and for Nike 4.62% – 3.39%. Parallel to the

**Table 1.** Thermal properties of flax fibres of Modran and Nike varieties obtained at the three stages of the technological process: after decortication (I), after wet degumming (II) and final mechanical cottonisation (III).

Fibre		$T_{onset}$ , °C	Mass loss, %	% of residue at 950 °C, %	$T_{10\%}$ , °C	$T_{60\%}$ , °C
Modran	Stage I	297.00	49.02	23.26	235.22	358.62
	Stage II	332.75	57.91	18.07	267.89	370.76
	Stage III	333.70	57.06	17.06	262.26	371.46
Nike	Stage I	308.96	55.70	20.45	250.04	366.62
	Stage II	333.18	61.92	15.83	281.43	372.85
	Stage III	335.47	62.85	17.06	291.41	371.51

**Table 2.** List of compounds identified during the thermal degradation of the fibres tested and the stage of the degradation when they occur.

Compound	Formula	Functional group	Wave number $cm^{-1}$	Decomposition stage
Water	H <sub>2</sub> O	OH	3735-3737	1-4
Carbon dioxide	CO <sub>2</sub>	CO <sub>2</sub>	2355-2369; 669-671	1-4
Carbon oxide	CO	CO	2182	2-4
Acetic acid	CH <sub>3</sub> COOH	-CH <sub>3</sub> OH C=O C-O	2971-2974; 2922-2927 3587-3590 1791-1795; 1765-1770, 1172-1175; 1105-1109; 1034	3
Formic acid	CHOOH	-CH OH C=O C-O	2971-2974; 2922-2927 3587-3590 1791-1795; 1765-1770 1172-1175; 1105-1109; 1034	3
Methanol	CH <sub>3</sub> OH	OH C-H C-O	3587-90 2931-2934; 2858-2864 1172-1175; 1034; 1105-1109	2-4
Formaldehyde	CH <sub>2</sub> O	C-HO C=O	2814-2819; 2725-2728 1744-1746	3
Methane	CH <sub>4</sub>	-CH	3016-3018	4
Ester	RCOOR	-CH CO-O-C C=O	2922-2932; 2860-2867 1051-1061; 1112 1727-1730	4

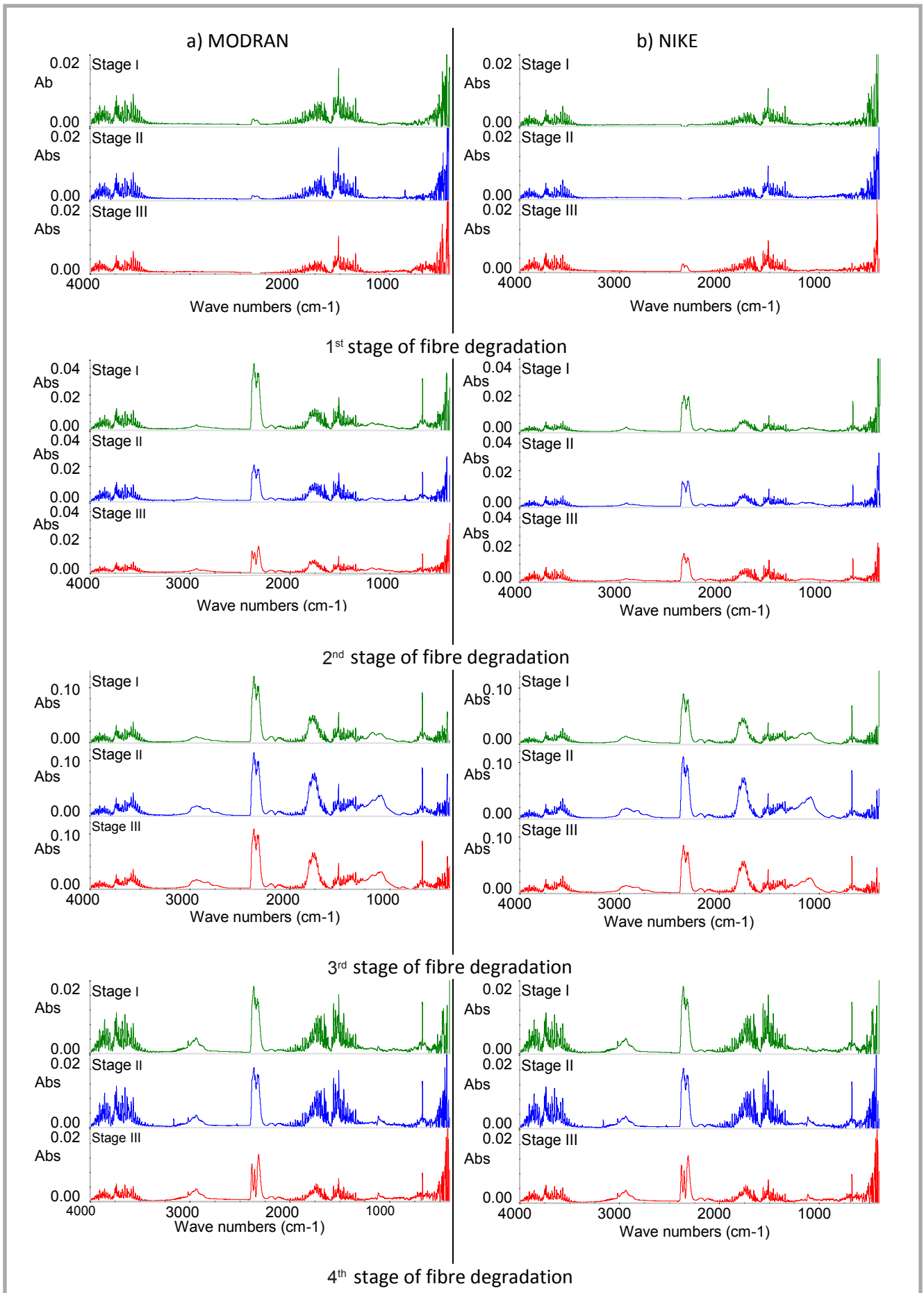


Figure 15. FTIR spectra for thermal degradation of test fibres after each stage of processing: a) MODRAN, b) NIKE.

thermal degradation analysis, the gases released were tested with the use of the Fourier transmission technique (FTIR).

Detailed analysis of FTIR spectra based on DTG analysis (**Figures 13-14**) allowed the determination of degradation products for all four stages, see **Figure 15**.

The studies in infrared, presented in **Figure 14** and **Table 2**, showed that in the structure of the gases released, the following bands can be distinguished representing vibrations for such functional groups as:

- O-H – the water molecule and hydroxyl group,
- CH<sub>3</sub> – the alkyl group, specific for aliphatic compounds,
- CH<sub>2</sub> and CH – methylene groups, specific for aliphatic compounds,
- CHO – the aldehyde group,
- C=O – the carbonyl group, specific for aldehydes, acids or esters,
- COO – the acid carboxyl group,
- CO-O-C – the ester group,
- CO<sub>2</sub> – for carbon dioxide,
- CO – for carbon oxide.

The presence of the same absorption bands in a few compounds leads to their overlap, which makes it impossible to identify separate compounds with certainty. FTIR analysis with OMNIC software showed that during thermal degradation the following compounds were released: water, carbon dioxide, carbon monoxide, acetic acid, formic acid, formaldehyde, methanol and methane (**Table 2**).

Moreover analysis of the band intensity of the spectra from specific stages of the decomposition showed that flax fibre of the Modran variety was characterized by a higher intensity of the gases released than Nike fibre (**Figure 15**). The highest intensity of degradation for specific stages was for cellulose in the fibre, decreasing in the following sequence: cellulose → hemicellulose → lignin → water. In the case of the decorticated fibres for the 3<sup>rd</sup> stage of thermal degradation of fibres, the lowest intensity of all organic gases released was also observed as compared with the fibre after wet processing or after mechanical cottonisation.

## Conclusions

- The technology of cottonization/elementarization of decorticated fibers developed with the application of

the degumming process in a closed degumming device with controlled changing pressure and with the use of ultrasound caused a considerable reduction in the linear mass, length and impurity content in flax fibres of the Modran and Nike varieties.

- Flax fibers obtained thanks to the use of the technology developed reached parameters comparable with those of cotton fibers in terms of the length and linear mass. The most efficient linear mass reduction of flax fibers was observed after the degumming process.
- Analysis of the chemical composition of the fibre indicated a significant reduction in hemicellulose content in all fibre types after subsequent processing steps; in the case of other components such considerable changes were not observed.
- Infrared analysis showed that the fibre from Modran is characterised by a higher intensity of the gases released as compared with the Nike fibre.
- Thermal analysis showed that the use of the process developed affected positively the thermal stability of the degummed flax fibres in comparison with the decorticated fibres.
- The results of this study proved that it was possible to obtain high quality flax fibers from low quality decorticated fibers by application of the technology developed, which allows to eliminate the retting process. This enables to shorten the technology of fiber extraction and to make it independent from weather conditions, which results in the production of more uniform fibers.

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