Degumming process of flax at industrial pilot scale

Mihaela Dochia, Cecilia Sîrghie, Lucian Copolovici,

Institute of Technical and Natural Sciences Research-Development of Aurel Vlaicu University, Romania, 2 Elena Dragoi St., 310330, Arad, Romania

Abstract

The enzymatic/ultrasounds degumming of flax fibers becomes an important alternative to conventional procedures. The present study shows the benefits of the ultrasounds in laccase degumming process at pilot scale. A pilot experiment using 1 % laccase and less than 30 minutes ultrasounds was performed, and high-quality degummed flax fibers were produced from flax tows using processes which have not been exploited commercially. We tested the influence of the ultrasounds/laccase system on the physical-mechanical and chemical properties of the flax fibers. The properties of the fibers are compared with those of the cotton fibers. Those properties allow us to obtain fine blend yarns (Tex 25) when the flax percentage is 50 %.

Keywords: flax, cotton, laccase enzyme, degumming, ultrasounds

Introduction

For flax degumming the conventional method is done using alkali solutions and high temperature with or without applying pressure (Hu et al., 2010; Aydin et al., 2012; Kozlowski et al., 2012). The conventional treatment reduces the quality of the fiber and produces waste waters highly polluting for the environment (Ranganathan et al., 2007). Consumption of large amount of chemicals and energy, it becomes economically not feasible. In the last years, the interest for using other methods instead of the conventional non-biological methods for flax degumming has increased.(Ko et al., 2011; Fu et al., 2012; Karaca et al., 2012).

Pectinase, cellulase and laccases had mostly used for degumming of flax tow in the industrial processes (Alix et al., 2012; Karaca, et al., 2012). There are many studies about Laccases (benzenediol:oxygen oxidoreductases; EC 1.10.3.2) intermediated degumming processes on different bast fibers (Kozlowski, et al., 2012). Laccases show low substrate specificity and high level stability in the extracellular environment in addition it is not necessary the usage of co-subtrate (Couto and Herrera, 2006). Low molecular mass mediators such as *N*-heterocylic bearing N–OH groups (e.g. 2,2-azinobis-3-ethylbenzothiazoline-6-sulfonic acid, violuric acid, hydroxyl benzotryazole) should be used in the degumming process of the flax with laccase, due to the non-specificity of the enzymes (Bao et al., 1993; Kozlowski et al., 2006; Batog et al., 2008; Tauber et al., 2008).

Due to the fact that enzymes have low diffusion rates and the effect is concentrated on the outer fibers there are some strategies to improve these drawbacks. For example it can be used the ultrasound energy (Basto et al., 2007), microwaves (Sgriccia and Hawley, 2007), steam explosion (Kessler et al., 1998) could be a way to improve the diffusion of the enzymes to the interior of the technical fibers in order to release elementary fibers. Among them the ultrasonic energy has been used in different processes such as: mercerization, desizing, bleaching, scouring, and dyeing of natural fibers (Yachmenev et al., 2002; Yachmenev et al., 2004; Stanescu et al., 2010). The use of ultrasounds in textile wet processes reduce energy and chemicals consumption (Moholkar and Warmoeskerken, 2004; Moholkar et al., 2004; Sirghie et al., 2008). The combined ultrasound/hydrolytic enzymes processes conduct to less fibers damage and better results (Yachmenev, et al., 2002; Yachmenev et al., 2007) which consist in cottonised flax fibers with physical-mechanical properties similar to cotton.

Akin et al (2001) has reported one of the first results on pilot scale studies of enzyme/ultrasounds applied on retted flax fibers obtaining fine materials.

In the present study we show the benefits of the ultrasounds in laccase degumming process at pilot scale. We tested the influence of the ultrasounds/laccase system on the physical-mechanical and chemical properties of the flax fibers.

Material and methods

All chemicals were supplied by Sigma-Aldrich unless otherwise stated.

Enzyme concentration used in the present work was 1 % regarding to fiber mass. In every experiment 10 kg flax tow fibers were treated in two steps in an ultrasound thermostatic bath (Bandelin RM 210 UH, Badelin electronic Gmbh., Berlin, Germany), with a capacity of 300 liters. In the first step were used 1 % o.w.f laccase and 0.01 % HOBT (respect to enzyme concentration) as mediator, at 55 ^{0}C and ultrasound frequency of 35 kHz for 25 min in a liquid to fiber ratio 1:15. In the second step the fibers were rinse for 10 min in the same liquid ratio conditions (1:15).

After drying the cottonized flax fibers were evaluated from physical-mechanical point of view using High volume instrument (HVI) System for cotton in order to appreciate the degumming degree.

200 kg of cottonised flax fibers obtained from 20 independent experiments of 10 kg each were mixed with cotton fibers (140 kg) in two ways (50 % + 50 %; 70 % + 30 %) in order to obtain the blended yarns. The blends were spun on cotton type equipments as follows: 3C carding machine (Unirea SA, Cluj, Romania), LB drawing frame equipments (Unirea SA, Cluj, Romania), BD-200 S spinning equipments (Elitex OE, Avanco, Praha, Czech Republic).

Results and discussion

In a previous study (Sirghie et al.), we have been sown that the optimal concentration of enzyme for degumming of flax fibers had been 1 %. Industrial pilot scale experiments were conducted in order to prove the technical efficiency for this concentration of enzyme (o.w.f) in presence of ultrasounds. This value (in correlation with ultrasounds) is a compromise by economical point of view which can be used at industrial scale due to the high cost of pure enzymes.

A higher concentration of enzyme without ultrasounds gives the same results while an increasing amount of enzyme in ultrasound treatment conditions would offer better results but is more expensive. Since “degumming” effect was obtained at 1 % enzyme concentration a higher percent could determine better fibers quality but with higher costs.

The composition of flax fibers includes besides cellulose, hemicellulose, lignin and pectin, etc. (Kozlowski, et al., 2012). Pectin and lignin act mainly as bonding agents. The chemical compositions of cottonised flax fibers compared with flax tow are shown in Table 1.

Table 1. Chemical composition of row material (flax tow) and degummed flax fibers

Chemical composition	Flax tow ± SEM	Degummed flax fibers ± SEM
Hemicellulose (%)	16.0 ± 0.1	14.7 ± 0.7
Lignin (%)	4.5 ± 0.1	2.44 ± 0.06
Pectin (%)	1.5 ± 0.1	0.95 ± 0.12
Degree of polymerization	3510 ± 13	3229 ± 59

The hemicellulose, lignin and pectin percentages are decreasing significantly for degummed flax fiber compared with flax tow. Similar composition of flax fibers is described in other papers (see for review (Baley, 2002)). The same decreasing in pectin, lignin and hemicellulose amount in flax fibers was shown in many studies and should be desirable in order to avoid poor absorbency and wettability (Bismarck et al., 2002).

As it can be seen from Table 2 the physical-mechanical characteristics of degummed flax fibers are comparable with those of the cotton fibers, in terms of length, tenacity and elongation.

Table 2. The physical-mechanical characteristics of degummed flax fibers and cotton fibers

	Length (mm)	Tenacity (cN/Tex)	Micronaire	Elongation (%)	Impurities (%)
Cottonised flax fibers	32.6-35.4	26.7–29.4	6-7.6	4.1-5.06	0.7-3.9
Cotton fibers	28.96-31.01	27.4-31.6	3.7-4.6	5.8-6.1	0.05-0.36

Fineness is one of the most important factors affecting fiber quality for spinning into yarns.(Akin et al., 1997) Fineness of degummed flax (6-7.6 micronaire) obtained using our procedure is 150 % higher than one which used pure cotton (3.7- 4.6 micronaire). Fiber strength, which has been related to fineness in enzyme-degummed flax, and length are other important fibers parameters that, along with fineness, influence quality and production efficiency of blended yarns.

From the physical-mechanical properties of blended yarns presented in Table 3 it can be seen that these yarns fall in 100 % cotton yarn quality parameters especially for strength and elongation.

Table 3. The physical-mechanical characteristics of different blended yarns

Characteristics/ composition		Yarn 29 Tex 50 % Degumming flax + 50 % Cotton	Yarn 25 Tex 50 % Degumming flax + 50 % cotton	Yarn 31 Tex 70 % Degumming flax + 30 % cotton
Fineness	Tex	30.3	24.5	31.8
	CV%	9.6	8.4	3.4
Strength	CN	163	133	208
	CV%	17.4	21.0	17.1
Breaking length	Km	5.4	5.4	6.5
Elongation	%	5.1	5.7	6.7
	CV%	20.3	20.2	12.4
Torsion (OE)	Torsions/m	1264	1164	1062
	CV%	4.5	2.9	3.8
Uster irregularity	CV%	12.9	10.43	11.43
Uster/1000 M	Neps	1477	1595	978

The torsion, even if it is slightly higher than the yarns of 100 % cotton, is in the acceptable range. From the irregular values, it can be seen that the blended yarns are as uniform as 100 % cotton yarns, a fact which indicates a very good degree of homogenization of fibrous mixture, thus confirming the results presented in Table 2 for degummed fibers.

The cost of our process of ultrasonic – enzymatic degumming of flax implies two different activities. The degumming process including water, energy, chemicals, drying of the fibers lead to a price of 0.88 EUR/kg. The price of cotton and flax is 2 EUR/kg. The second process included in the price is the spinning with the total costs of 1.33 EUR/kg. Taking in account all costs, the specific consumption for a blend yarn of 29 or 25 Tex is 1.4 EUR/kg while for a blend yarn of 31 Tex is 1.6 EUR/kg. The above mentioned costs were calculated for 300 kg mixtures in a pilot experiment. It is expected that the production of yarns in large amounts (over 1000 kg) may improve the specific consumption.

Conclusions

We showed that by using 1 % enzymes in ultrasound conditions for 25 min were obtained at industrial scale, degumming flax fibers of very good quality (similar to cotton fibers from physical-mechanical point of view). This procedure allowed us to obtain a fine blended yarn (Tex 25) when the flax percentage was 50 %. Such yarns has never been obtained at industrial scale except in one of our earlier report (Sirghie, et al., 2008).

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