ENZIMES IN COTTON BIO-SCOURING

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Legislaţia strictă privind mediul înconjurător a dus, în ultima perioadă, la creşterea cererii de tehnologii noi. Procesele din domeniul textil sunt caracterizate de o poluare avansată generată de cantitatea mare de ape reziduale cu pH acid sau bazic. De aceea, aplicarea de procedee de prelucrare enzimatică se dovedeşte de mare interes. În lucrare se prezintă încercarea de a înlocui dezincrustarea alcalină a bumbacului cu un procedeu enzimatic. Se subliniază importanţa auxiliarilor pentru a obţine rezultate corespunzătoare. Proprietăţile bumbacului astfel tratat sunt discutate în comparaţie cu cele ale materialului tratat prin procedeul clasic.

The demand for new ecological technology lately enhanced, due to the strict environmental regulations introduced. Textile processes are characterized by high pollution, generated by the large amount of waste waters with acid or alkaline pH. Thus, the application of enzymatic work-up procedures seems of great interest. Attempt to replace the alkaline scouring of cotton with an enzymatic procedure is described. The importance of auxiliaries for obtaining good results is underlined. The properties of the treated cotton are discussed in comparison with the classical treated material.

Keywords: cotton, pectinase, bio-scouring, surfactant, complexing agent, clean technology

1. Introduction

The sustainable development concept together with the climate change phenomenon enforced the necessity to solve pollution problems in all industrial branches. The elimination of the waste waters produced by different processes is a type of approach. Some results in this respect are published in recent issues of this journal [1,2]. Another approach consists in changing the polluting technology

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with a green procedure. The textile industry is a source of pollution, generating a large amount of waste waters with high chemical content.

De-pollution with modern methods is imposed primarily due to the new regulations concerning environment [3, 4]. A more accurate procedure is the replacement of the polluting technology with a biotechnology [5]. Such an example is the enzymatic scouring of cotton.

The cotton fibres have a multilayer structure. The main part of it has a cellulose structure, but there are also non-cellulose components, mainly located in the outermost layer, the cuticle. Beside cellulose, the components of the cuticle are: wax, proteins and pectins [6]. The average composition of raw cotton is presented in Table 1 [7, 8]. Hardin and Kim [9] mentioned a similar composition.

Table 1

Cotton fibre composition

Component	Total amount (% wt)	Amount in the primary wall (% wt)
Cellulose	86-93	52
Pectins, proteins, dyes	5-6	24
Oils, fats, waxes	0,1-1	7
Minerals	1	
Moisture	8	

These non-cellulose components generate limited water absorbency and whiteness of cotton, as well as an uneven dye fixation. Thus, procedures for eliminating these impurities have been elaborated. Scouring is the process for removing of existing impurities or those attached during the spinning process. The main component to remove is pectin. Pectin has as main structure a polymer chain of α-(1-4) poly-D-galacturonic acid (Fig.1), as a free acid or its methyl ester, randomly distributed.

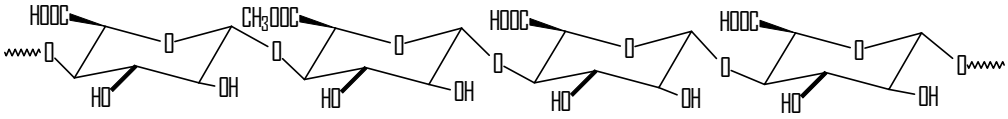


Fig. 1. Pectin structure

The carboxyl groups (COOH) are usually as calcium salts. This insures a stability of the primary wall composite. For breaking this complex structure the elimination of calcium ions is absolutely necessary.

At an industrial scale, scouring is carried out using high alkaline solutions which hydrolyse the ester and the semi-acetal bonds. The method gives good results in term of products but there are problems in connection with the energy consumption and environment protection.

One alternative procedure, lately investigated is bio-scouring. The process uses enzymes as reagents for impurities elimination and has a lower energy consuming. The enzymes used in cotton scouring belong to the class of *Hydrolyses* (class 3) or *Lyases* (class 4) [10]. In the first case, two types of enzymes are involved: *Pectinesterases* catalysing the methyl ester hydrolysis and *exo-* and *endo- Polygalacturonases* breaking the 1-4 carbohydrate bonds. In the second case the fragmentation of the pectin polymer chain may be performed through elimination reactions giving smaller fragment of poly-D-galacturonic moieties. The second solution is more economic needing only one type of enzyme.

There are a number of examples with cotton bio-scouring, using mixtures of enzymes as reagents [11-17].

The present work describes the results obtained by using a single *Pectate Lyase* (class 4), a commercial product, BioPrep 3000 L, in order to obtain a less expensive bio-scouring technology.

2. Experimental

Materials and Methods

Knitted 100% cotton jersey structure, with a weight of 120g/m^2 was pre-treated for degreasing with a water solution of Na_2CO_3 (5g/L) and Felosan RG-N as surfactant (5m L/L). The degrease incubation time was 1h. After the treatment, the material was washed and dried.

A commercial product, namely BioPrep 3000 L, supplied by Novozym, having an activity of 3000 APSU/g (Alkaline Pectinase Standard Units), has been used. According the classification (E.C. 4.2.2.2), the contained enzyme is a Pectate Lyase.

As auxiliaries the following compounds have been used: a non-ionic wetting agent, Sandozin Nit (a poly-etoxylated alcohol, biodegradable, stable in alkaline conditions) and EDTA (sodium salt of ethylenediamine-tetraacetic acid) as complexing agent for the elimination of the calcium ions.

The treatments have been performed in a buffered solution of pH 8.8 prepared from H_3BO_3, KCl and NaOH.

The enzymatic treatments have been done in the solution containing the quantities of enzyme and auxiliaries listed in Table 2, at 15/1 (mL/g) liquor-to-fabric ratio and kept 1h at 55^{0}C. After the treatment, the fabric has been washed at 90^{0}C for eliminating the enzyme, the surfactant and the complexing agent.

3. Results and discussion

A number of attempts have been done for finding an environmental friendly way of scouring the cotton, in order to establish the best conditions for the enzymatic treatments. Table 2 presents different composition of the reaction mixtures studied in the experiments:

Table 2

Enzymes and auxiliaries

Sample	Cotton	Enzyme		Wetting agent		EDTA		Buffer
	(mg)	o.w.f. (%)	(mL)	(mL)	(mL/L)	(mg)	(g/L)	(mL)
1	764	0.1	0.0008	0.057	5	14.3	1.25	11.46
2	688	0.685	0.0047	0.083	5	20.8	2.0	10.32
3	716	0.685	0.0049	0.086	5	5.2	0.5	10.74
4	765	1.55	0.0119	0,0574	5	0	0	11.47
5	605	1.55	0.0094	0.0159	1.75	22.7	2.5	9.075
6	738	1.55	0.0114	0	0	13.8	1.25	11.07
7	729	1.55	0.0113	0.1094	10	13.7	1.25	10.93
8	798	1.55	0.0124	0.059	5	15	1.25	11.97
9	685	2.41	0.0165	0.0198	2	20.7	2	10.28
10	750	2.41	0.0181	0.0908	8	5.4	0.5	11.25
11	752	2.41	0.0181	0.0910	8	22.8	2	11.28
12	714	3.0	0.0214	0.0536	5	13.4	1.25	10.71

The enzyme reacts on the pectin substrate by an acid-base catalytic mechanism, generating a mixture of saturated and unsaturated (mainly α-β-unsaturated acids) fragmented chains [18]:

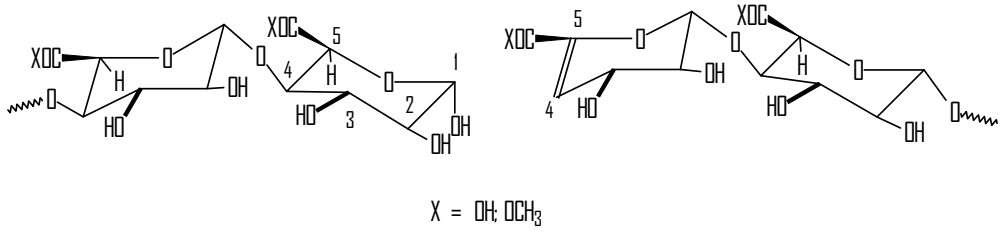


Fig. 2. Pectin fragments after treatment with a *Pectate-Lyase*

The reaction is a trans–elimination process favoured by the axial *trans* configuration of the H and O from the 4 and 5 positions of the monosaccharide unit (see Figure 2).

All the samples have been treated according to the experimental part. The effect of the enzyme on textile material was evidenced by the following properties

of the cotton: the weight loss, the hydrophilic behaviour and the value of the light remission after the usual [19] whitening treatment. All the values are discussed in comparison with those of the ***Sample 13*** treated, according the classical procedure, with a solution obtained from NaOH (5 g/L), Felosan RG-N (2 g/L), EDTA (1.25 g/L), $Na_2S_2O_4$ (2 g/L), for 1h at 98 ^{0}C.

Table 3

Properties of the cotton material after different treatments

Sample	Weight loss (%)	Hydrophilicity (s)	Light remission (%)
1	1.60	2.5	83.5
2	2.03	2.0	82.5
3	1.52	2.5	83.8
4	1.05	3.0	84.3
5	1.16	2.0	83.6
6	1.22	2.5	82.6
7	1.51	3.0	80.1
8	1.50	<2.0	84.5
9	1.75	2.0	80.5
10	2.00	2.5	84.5
11	1.86	2.0	80.9
12	2.24	3.0	78.5
13	5.02	2.0	83.0

Better results are obtained in a number of enzymatic treatments in comparison with the classical alkaline scouring. In the classical procedure an advanced destruction of the material is observed, the weight loss being ~ 5 %, meanwhile in the enzymatic procedure the weight loss is situated between 1% and 2.24%. The hydrophilicity and the light remission of the sample treated by the classical procedure are comparable with some of those from the enzymatic experiments.

For establishing the optimum conditions for the enzymatic treatment the reaction parameters are discussed. The influence of the enzyme quantity (o.w.f. = over weight fibre) on the cotton jersey properties are presented in Table 4.

Table 4

The influence of the enzyme

Properties/Sample	Sample 1	Sample 3	Sample 8	Sample 9	Sample 12
Enzyme o.w.f (%)	0.1	0.685	1.55	2.41	3.0
Weight loss (g)	1.60	1.52	1.50	1.75	2.24
Hydrophilicity (s)	2.5	2.5	< 2.0	2.0	3.0
Remission (%)	83.5	83.8	84.5	80.5	78.5

Based on the experimental data a 1.55 % enzyme o.w.f. is recommended due to the properties of the treated cotton: enhanced hydrophilicity and a good value for the light remission.

The influence of the wetting agent is established by taking in consideration the experiments where the enzyme and EDTA are constant (see Table 5).

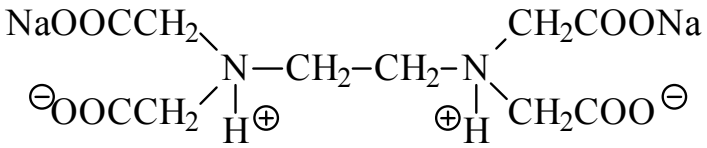
Table 5

The influence of the wetting agent

Properties/Sample	Sample 4	Sample 8	Sample 7
Sandozin (g/L)	0	5	10
Weight loss (g)	1.22	1.50	1.51
Hydrophilicity (s)	3.0	<2.0	3.0
Remission (%)	84.3	84.5	80.1

A concentration of 5 g/L wetting agent seems appropriate for obtaining a good material after the enzymatic treatment.

In a similar way, the effect of the added quantity of EDTA (**1**) is evidenced in Table 6.



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(1)

Table 6

The influence of the complexing agent

Properties/Sample	Sample 4	Sample 8	Sample 5
EDTA (g/L)	0	1.25	2,5
Weight loss (g)	1.22	1.5	1.16
Hydrophilicity (s)	3	< 2	2
Remission (%)	84.3	84.5	83.6

The quantity of 1.25 g/L EDTA had given the best results.

Based on the experimental data the following reaction conditions seemed the most appropriate: commercial enzyme o.w.f. 1.55 %, Sandozin 5g/L, EDTA 1.25 g/L. The weight loss is reasonable (1.5 %) and the material properties are very good (hydrophilicity less than 2 s, light remission 84.5 %).

4. Conclusions

The experimental works done with the commercial *Pectate Lyase* BioPrep 3000 L have confirmed the possibility to make the scouring process with only one enzyme. The conditions for obtaining the best results with this enzyme product have been established. The material obtained has better properties compared with the classical scoured cotton. This makes possible the replacement of a polluting process (alkaline treatment) with an environmental friendly one (enzymatic treatment).

Acknowledgements

This work has been supported by The Ministry of Education and Research through the BIOTECH national research programme, contract: 02-3-PED-443. Thanks are due to Novozym for supplying the BioPrep 3000 L and to Bezema-CHT Tubingen which kindly supplied the surfactant used in the experiments

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