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Green Solution for Cotton Scouring

Abstract
The classical procedure for cotton scouring generates pollution due to the chemical reagents used for pectin destruction. Up to now a number of solutions using enzymes as catalysts for pectin degradation have been proposed. The use of chemicals like ethylene diamine tetra acetate (EDTA), which are known as toxic, makes processes of bio-scouring unecological. We studied the possibility of replacing EDTA with a biodegradable compound, namely sodium citrate. The use of ultrasounds for improving the bio-scouring process is also illustrated.

Key words: *cotton, bio-scouring, complexing agents, ultrasound.*

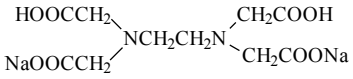
Introduction

Our interest in the application of new green (technology) technologies in textile material processing has been shown in previous publications [1 - 7]. The use of enzymes instead of polluting reagents has been studied for textile waste recovery, cotton and wool finishing, and waste water treatments.

Cotton fibre has a multilayered structure that has been studied and characterised for nearly a century. The structure of the primary cell wall of cotton fibre, particularly the outermost surface layer, has a determinant influence on textile processes [8].

Cotton mostly consists of cellulose, but there are also other components mainly located in the outermost layer - the cuticle. These components are usually wax, protein and pectin [9, 10]. The process of removing existing impurities or those attached during spinning is termed scouring. The classical scouring operation is performed using alkali for pectine degradation by hydrolysis.

A large number of experiments involving bio-scouring with *Esterases* and *Polygalacturonases* or *Pectinlyases* were performed [11 - 17]. The solutions proposed gave high yields in pectin elimination, leading to materials with good properties, comparable with those obtained by chemical procedures. The results obtained were promising enough, but the need for an auxiliary compound like EDTA (**1** - sodium salt of ethylene diamine tetra acetate), which is known as toxic [18], makes the process unecological.



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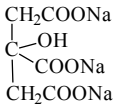

(2)

Our paper presents experimental results from a bio-scouring process using a non-toxic compound, sodium citrate (**2**), instead of EDTA. To improve the results ultrasound was also used while performing the bio-scouring process.

Experimental

Materials and methods

A commercial product, namely SERA ZYME C-PE (Roglyr Eco 183), based on (Pectinlyase) Pectate Lyase (E.C. 4.2.2.2), was used, supplied by Rotta, Germany. The commercial product contains around 5% enzyme.

The activity of the enzyme was determined by the method described in literature [19], spectrophotometrically monitoring the absorbance at 235 nm. The experimental activity obtained was 0.039 U/mg of the commercial product.

The textile material used was 215 g/mL of plain woven 100% cotton fabric. Samples of 10 × 10 cm were prepared. The mass of the dried samples was determined with a Sartorium MA 100 thermobalance of high precision.

As auxiliaries the following compounds were used: a weakly anionic wetting agent: Sulfolen 148 (S-148, alkyl polyglicol ether), alkali stable, used for the pre-treatment and bleaching of cellulose fibres, as well as EDTA (sodium salt of ethylene diamine-tetra acetic acid, $C_{10}H_{14}N_2Na_2O_8 \cdot 2\ H_2O$) and sodium citrate ($C_6H_5O_7Na_3 \cdot 5.5\ H_2O$) as complexing agents. The treatments were performed in a buffered solution of pH 7.5 prepared from Na_2HPO_4 and NaH_2PO_4.

Surface changes were highlighted using a photonic brightfield microscope (Motic B1 series) connected to a 2.0 M pixel camera (Moticam 2000) inserted into a photo tube. The samples were observed through 40× objective lenses and 10× ocular lenses so that the microscope's total magnification power was 400×. Each 1 cm^2 square sample analysed was mounted on a slide in a drop of distilled water.

Ultrasound treatments were performed in an ELMA ultrasonic bath - the TI-H-10 model with a capacity of 8 litres and two frequencies: 35 kHz and 130 kHz. The frequency used was low (35 kHz).

Method of bioscouring

Enzymatic treatment was performed in a solution of pH 7.5 (Na_2HPO_4 and

NaH_2PO_4) containing the quantities of enzyme and auxiliaries listed in ***Tables 1 - 4***. The experimental conditions were designed based on a mathematical model [19 - 21]. The experiments were developed at a 15/1 in mL/g liquor-to-fabric ratio over different times (1/3 h or 1 h) at 55 °C, with or without ultrasound. Alternatively, EDTA and sodium citrate were used as complexing agents. After the treatments the fabric was washed with water at 90 °C to eliminate all the reagents and products. All the experiments were done in triplicate, the data presented being average values.

Method for hydrophilicity determination

Circular samples of 2 cm were cut from each bio-scoured cotton material, and the time of submersion in 7 cm of water in a 600 mL beaker was measured with high precision [21, 22].

Results and discussion

All the samples were treated according to the experimental part. The effects of the enzyme and the auxiliaries on the textile material were evidenced by the following properties of the cotton: the mass loss and hydrophilic behaviour.

The sample's mass loss was calculated according the following formula:

$$\%\Delta M = (M_i - M_f)/M_i \times 100$$

where:
M_i - initial mass,
M_f - final mass.

Measurement of hydrophilic properties was performed according to the procedure described in the experimental part. The reagent ratios and presence or not of ultrasound treatment are specified in ***Tables 1 - 4***.

We compared the results obtained by enzymatic treatment with those resulting from classical treatment performed with a solution of NaOH (10 g/L), Na_2CO_3 (5 g/L) $Na_2S_2O_4$ (2 g/L), Sulfolen 148 (2 g/L) and sodium $NaHSO_3$ (1 g/L) for 1 h at 98 °C. For this type of scouring, the average data are as follows: a mass loss of 3.21% and a hydrophilic characteristic of 2.5 s.

Surface changes were evidenced using high performance microscopy (see ***Figures 1*** & ***2***).

Table 1. *Reagents and results of bio-scouring with sodium citrate without ultrasound.*

No	Sample, g	Enzyme, % o.w.f.	S-148, mL/L	Sodium citrate, g/L	Weight loss, %	Hydrophilicity, s
0	1.22	0.00	0.0	0.0	0.58	6.3
1	1.21	0.10	5	1.25	1.15	3.5
2	1.22	0.685	5	2.0	1.56	3.5
3	1.24	0.685	5	0.5	1.45	4.8
4	1.23	1.55	5	0.0	1.47	3.2
5	1.20	1.55	1.75	2.5	1.59	5.0
6	1.22	1.55	10	1.25	1.97	2.5
7	1.20	1.55	5	1.25	1.66	2.3
8	1.24	2.41	2	2.0	1.69	3.3
9	1.28	2.41	8	0.5	1.25	2.8
10	1.24	2.41	8	2.0	2.58	2.8
11	1.20	3.00	5	1.25	1.78	2.9

Table 2. *Reagents and results of bio-scouring with sodium citrate and ultrasound.*

No	Sample, g		Enzyme, % o.w.f.	S-148, mL/L	Sodium citrate, g/L	Weight loss, %		Hydrophilicity, s	
	60 min	20 min				60 min	20 min	60 min	20 min
0	1.28	1.25	0.00	0.0	0.0	1.45	1.12	17	5.0
1	1.25	1.20	0.10	5	1.25	2.17	1.33	2.5	2.5
2	1.23	1.20	0.685	5	2.0	2.20	1.50	3.0	2.7
3	1.26	1.21	0.685	5	0.5	1.83	1.24	3.0	2.9
4	1.25	1.21	1.55	5	0.0	1.84	0.99	2.5	2.7
5	1.26	1.22	1.55	1.75	2.5	2.14	1.31	3.0	4.6
6	1.24	1.21	1.55	10	1.25	2.49	1.66	2.0	2.8
7	1.21	1.20	1.55	5	1.25	2.23	1.33	2.5	3.0
8	1.26	1.23	2.41	2	2.0	2.06	1.14	2.5	2.7
9	1.27	1.22	2.41	8	0.5	1.96	1.72	3.0	3.0
10	1.22	1.20	2.41	8	2.0	1.81	1.09	2.3	2.9
11	1.21	1.18	3.00	5	1.25	1.90	1.28	2.6	3.2

Table 3. *Reagents and results of bio-scouring with EDTA without ultrasound.*

No	Sample, g	Enzyme, % o.w.f.	S-148, mL/L	Sodium citrate, g/L	Weight loss, %	Hydrophilicity, s
0	1.25	0.0	0.0	0.0	0.58	17
1	1.24	0.10	5	1.25	2.01	2.5
2	1.21	0.685	5	2.0	2.31	2.0
3	1.27	0.685	5	0.5	1.57	3.0
4	1.20	1.55	5	0.0	1.58	2.6
5	1.21	1.55	1.75	2.5	1.74	2.6
6	1.28	1.55	10	1.25	2.27	2.0
7	1.20	1.55	5	1.25	1.84	2.5
8	1.22	2.41	2	2.0	1.56	3.0
9	1.22	2.41	8	0.5	2.69	3.0
10	1.22	2.41	8	2.0	1.89	2.2
11	1.24	3.00	5	1.25	2.50	2.5

Table 4. *Reagents and results of bio-scouring with EDTA and ultrasound.*

No	Sample, g		Enzyme, % o.w.f.	S-148, mL/L	Sodium citrate, g/L	Weight loss, %		Hydrophilicity, s	
	60 min	20 min				60 min	20 min	60 min	20 min
0	1.23	1.22	0.0	0.0	0.0	1.45	1.12	7.3	5.0
1	1.25	1.18	0.1.0	5	1.25	1.93	1.95	2.7	2.3
2	1.23	1.25	0.685	5	2.0	2.35	2.01	3.2	2.1
3	1.26	1.26	0.685	5	0.5	2.38	2.15	2.5	2.3
4	1.22	1.20	1.55	5	0.0	1.96	1.26	2.7	3.3
5	1.21	1.21	1.55	1.75	2.5	1.58	1.57	3.0	3.5
6	1.24	1.23	1.55	10	1.25	2.27	2.19	2.5	2.4
7	1.27	1.21	1.55	5	1.25	2.61	2.24	2.3	2.8
8	1.27	1.21	2.41	2	2.0	2.74	2.24	2.5	3.5
9	1.29	1.20	2.41	8	0.5	1.70	2.58	2.8	2.7
10	1.20	1.20	2.41	8	2.0	2.67	1.25	2.3	2.6
11	1.18	1.23	3.00	5	1.25	3.14	2.43	2.2	2.8

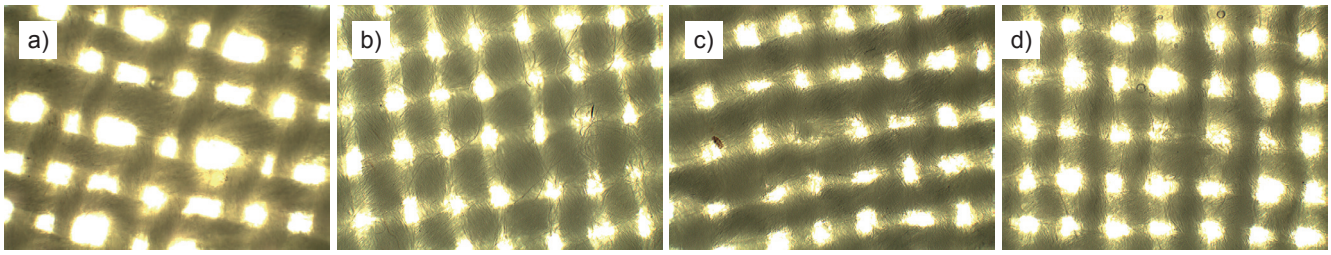

Figure 1. *Microscopic images of samples treated without complexing agents; a) Grey cotton, b) Classical treatment, c) Buffer, d) Buffer with US.*

From the experimental results, the following data were evidenced:

- The classical and bio-scouring processes yielded similar results; the enzymatic procedure may replace the chemical one.
- The samples obtained using sodium citrate have comparable properties with those obtained with EDTA with respect to the surface aspect and hidrophylicity values;
- In all the experiments, except for the blanc samples (no. 0), the hydrophilicity has acceptable values (less then 10 s);
- For all the samples, enhancement of the enzyme ratio to 2.41% o.w.f. gave, in similar conditions, a higher weight loss and lower values for hydrophilicity;
- The presence of wetting agent S-148 is absolutely necessary in citrate experiments, otherwise the material has a low wet capacity (hydrophilicity over 60 s);
- In the experiments with higher quantities of complexing agents (sodium citrate and EDTA) better results were obtained;
- Ultrasounds improved the bio-scouring process, with a time of 20 minutes giving better results;
- Longer time under ultra sound (60 minutes) fragmented the fibres (see ***Figure 2***)

Conclusions

A number of experiments on the bio-scouring of 100% cotton using EDTA and sodium citrate as calcium complexing agents were performed. From the experimental results we can state that the replacement of EDTA with sodium citrate leads to materials with comparable properties. The results of the bio-scouring are not very different from those of classical treatment with strong alkali solution. The use of sodium citrate made the bio-scouring treatment greener than that with EDTA.

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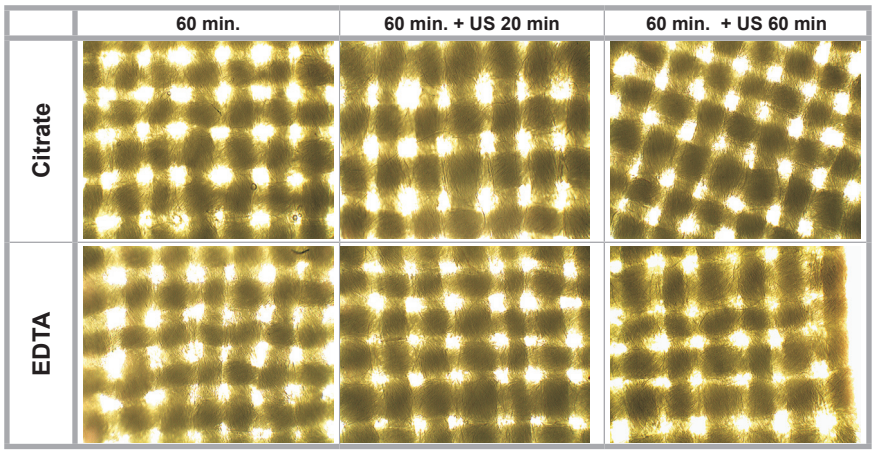

Figure 2. *Microscopic images of samples treated with complexing agents (US = ultrasound treatment).*

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