

## Combination of enzymatic methods for a more continuous cotton processing

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### ABSTRACT

The urgent requirement of environmental pollution, the energy and water consumption is nowadays a fact and the textile industry has still many changes to accomplish in this concern. Aside the environmental aspect, the use of toxic compounds and the necessity of recycling enormous amounts of residual products and wastewater means an economical effort to a company that is reflected by higher prices on their products. Therefore, possible alternatives given to this sector have to be eco-friendly, but also sustainable.

The aim of our studies was to substitute conventional textile wet processes of the cotton industry for alternative methods that are (1) less harmful, (2) less aggressive towards the environment, but also the fibre, (3) made in mild conditions, and (4) cost-effective. Therefore, other objectives derived from the main one involve the saving of time, water, energy and products. The alternative ways of cotton processing we suggest are based on the use of enzymes as biocatalytical agents. This enzymatic application to textile wet processing is long known, but a broad industrial implementation has not been successful so far. Our principal innovation consists in the combination of two or more processes in a single step by improving the conditions and the products used.

The presented work consist in the study of several combinations of bioprocesses (desizing, scouring, removal of bleach, polishing) and the required improvements in order to achieve a partial or total continuous processing of cotton.

### KEYWORDS

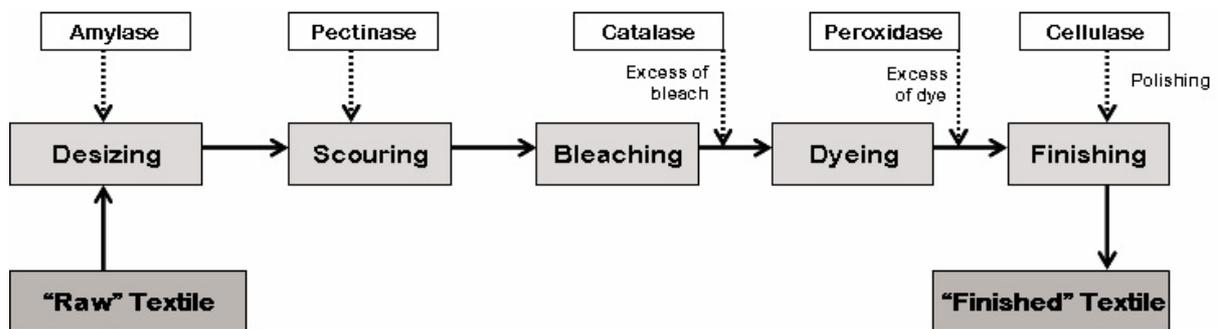
Enzyme catalysis, cotton bioprocessing, environmentally friendly.

## 1. INTRODUCTION

The actual situation of the textile market has promoted a new tendency, meaning that consumers are aiming for goods that have perfect finishes, are of high quality and, on top of that, are manufactured in an environmental friendly way<sup>1</sup>.

The so-called “White Technology” refers to the biotechnology applied to industry and it contains several activities among which we can find bioprocesses, like the use of enzymes in textile processing. The textile industry, and concretely the cotton sector, offers a great deal of possibilities where biotechnology could be applied in order to achieve (1) costs reduction, (2) a better environmental protection, (3) to give answers to security and health problems, an (4) to improve the quality and functionality of products. The use of enzymes allows a reduction or, in the best case, an eradication of the use of harmful chemicals<sup>2,3</sup>.

The interest in enzyme use is because they are natural catalysators with high substrate selectivity and reaction specificity. In the case of cotton fibres there are a large amount of possible enzymatic applications along its wet processing. As an example, amylases could act on starch desizing, cellulases on the fibre’s cellulose content, and pectinases could specifically reduce the pectine content without influencing cellulose<sup>4</sup>.



Cotton is the most commonly used natural textile fibre in the world. In the wet processing of cotton the use of size (natural or synthetic) is necessary to ensure that yarns are well protected from the weaving process abrasion. After weaving it is indispensable to remove the size before the following steps, like scouring, where natural impurities are separated from the fibres to assure a good conditioning for subsequent steps as bleaching and dyeing. The conventional processing needs the use of acid and basic solutions, detergents and big quantities of water to rinse the excess of products after their use. The alternative method is the use of enzymes: amylase in the case that sizing was done with starch and pectinase for the scouring process<sup>5,6</sup>. So far, these two enzymes have been successfully used and the idea now is to make possible the combination of these two processes in one step to achieve a resource reduction (water, energy, products). In order that two or more enzymes can work together is necessary that their optimal working conditions coincide and that their activities do not interfere.

After desizing and scouring have been completed the following processes can also integrate enzymatic catalysis. After bleaching, an excess of hydrogen peroxide can

compromise the dyeing step. To facilitate its elimination from fabrics and water one can use a catalase. Similarly, to assist the excess of dye elimination the answer is to use a peroxidase. Another very powerful enzyme is cellulase, which is used to reduce fuzziness and consequently soften fabrics. The catalysis takes places on the surface and the weak extending cellulosic fibres are finally detached by the use of mechanical agitation<sup>7</sup>.

The challenge for us was to combine all those processes where enzymatic catalysis could be used in a semi-continuous wet processing of cotton.

The possibility to use enzymatic catalysis for textile processing offers companies of the textile sector a much friendly way to treat cotton and the environment at the same time. Moreover, combination of two or more processes by combining several enzymes offers an extra way of resources saving.

## 2. EXPERIMENTAL

### 2.1 Materials

The textiles used are 100% popelin cotton and starch-sized.

The enzymes used are all commercially available. Aquazim SDL (amylase), Scourzim L (pectinase), Terminox Ultra 10L (catalase) and Cellusoft CR (cellulase) were purchased to Color-Center SA., a distributor of Novozymes in Spain.

The textile auxiliaries used were Cotemoll DNS (wettener) and Amplex T-ECO (chelator), also from Color-Center SA. For the bleaching process 35% hydrogen peroxide, sodium silicate, magnesium sulfate and 85% sodium tripolyphosphate were purchased from Scharlab.

### 2.2 Textile treatments

#### *Enzymatic desizing and scouring*

Starch-sized cotton fabrics were treated with Aquazim SDL and Scourzim L in a concentration of 1 g/l and 5 g/l, respectively, and in the presence of 4.5 g/l wettener and 1 g/l chelator in 0.1 M phosphate buffer pH 7.2 at 60°C during 3h.

#### *Standard scouring process*

Alkaline scouring was performed in a sodium hydroxide solution (0.2 M) with 4.5 g/l of wettener and 1 g/l of chelator for 1h at 90°C.

#### *Standard bleaching process*

Fabrics were bleached following the recipe: 35% hydrogen peroxide (15 g/l), sodium silicate (1.5 ml/l), magnesium sulphate (0.1 g/l) and 85% sodium tripolyphosphate (2 g/l), for 1h at 90°C.

#### *Enzymatic removal of bleaching excess*

Catalase is known by its efficiency converting hydrogen peroxide onto water and oxygen. In this way an excess of this compound can be removed from the solution and fabrics to avoid interferences in further processes. For this purpose, Terminox Ultra 10L was added in a concentration of 5 g/l for 20 minutes at 40°C.

#### *Global enzymatic wet processing of cotton*

A semi-continuous processing of cotton was achieved. Desizing and scouring steps were performed as previously described. Following, samples were embedded in the bleaching recipe for 1h at 90°C. After this time, temperature was decreased to 40°C and catalase was added directly to the solution for 20 minutes. Bleaching water was discarded and the solution used for desizing and scouring was here reused after having thermally inactivated the remaining enzymes. To this recycled solution Cellusoft CR was added in a concentration of 1 g/l at 60°C for 1 h to achieve polishing of cotton previous dyeing.

All processes were done in a Redkrome machine generally used for dyeing samples, but very helpful for controlling exact temperatures for our purpose.

### 2.3 Testing methods

#### *Weight loss evaluation*

At the end of the bioprocessing, fabrics were removed from the solution, boiled to inactivate the enzyme, washed and dried under a humidity controlled atmosphere followed by weight loss determination.

$$\% \text{ Weight loss} = 100 \times (\text{initial weight} - \text{final weight}) / \text{initial weight}$$

#### *Desizing evaluation*

Starch removal can be easily tested by using the iodine staining test. A small fabric sample is placed for 1 minute in a iodine solution (potassium iodide/iodine) and rinsed for 20 seconds with cold water. After drying the sample, the degree of desizing can be assessed with the Tegewa Violet scale, being dark blue/violet a presence of non-degraded starch and yellow a complete starch free sample.

#### *Bleach removal evaluation*

Fabrics and water hydrogen peroxide removal was evaluated by a iodometric titration procedure. Peroxide oxidizes iodide to iodine in the presence of acid. The iodine

formed can be then titrated with thiosulfate solution incorporating a starch indicator, which forms a dark blue-grey complex with the triiodide ion. At the end point, the reduction of triiodide ion turned transparent the solution.

#### *Determination of the recovery from creasing (UNE EN 22313:2002)*

With this test it is possible to measure the capacity of a bent fabric to recover after being exposed to a 1 kg weight for 5 minutes. The diagnostic is done by measuring the angle of recovering.

#### *Tensile properties of fabrics (EN ISO 13934/1:1999)*

Samples are submitted to a constant strength to determine the maximum load that can be achieved without breaking.

#### *Fibre evaluation by Scanning Electron Microscopy*

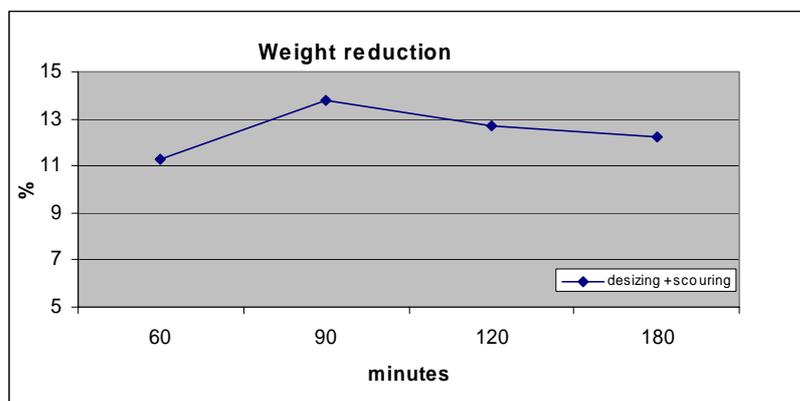
The fibre topology after treatments was studied by Scanning Electron Microscope (JEOL JSM 5610) imaging. Samples were coated with metal and analysed to search for damages at the fibre level.

### 3. RESULTS AND DISCUSSION

#### 3.1 Enzymatic desizing and scouring

A study to search for optimal conditions was done. Temperature, pH and bath ratio was always kept constant and chosen following indications by the manufacturer. A time course was performed and best results were obtained by treating fabrics for 3 h. After the best timing for treatment was fixed, the next variables to test were amounts of enzymes and auxiliaries. In summary, the best working conditions are as following: Aquazim SDL and Scourzim L in a concentration of 1 g/l and 5 g/l, respectively, and in the presence of 4.5 g/l wettener and 1 g/l chelator in 0.1 M phosphate buffer pH 7.2 at 60°C during 3h.

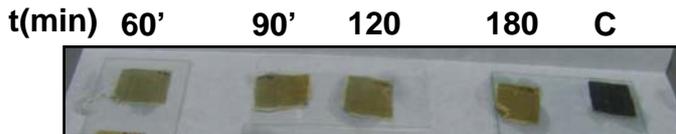
After starch and impurities elimination there is a weight reduction of treated fabrics as shown in Figure 1.



**Figure 1.** Time course and weight reduction after desizing and scouring treatment.

### 3.2 Evaluation of the desizing result

The iodine staining test was used for starch removal evaluation. Samples with dark blue/violet colour indicate presence of non-degraded starch and coloured yellow means a complete starch free sample. On the picture (Figure 2) one can appreciate that the sample treated for 1 h still have blue shades that completely disappear after 3 h treatment. The control sample was treated for 3 h in the same liquor, but without the presence of enzymes.



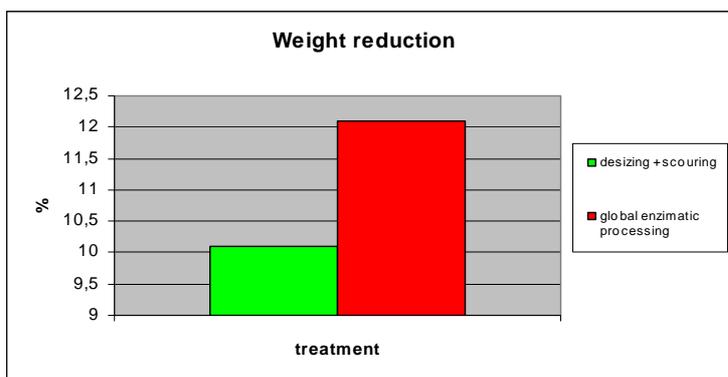
**Figure 2.** Iodine staining test for desized + scoured samples at different time points.

### 3.3 Enzymatic removal of bleaching excess

Catalase catalyses the conversion of hydrogen peroxide onto water and oxygen. In this way an excess of this compound can be removed from the solution and fabrics to avoid interferences in further processes. In previous works the main concern was to eliminate the peroxide excess adhered onto fabrics, but with this treatment we have completely eliminated any peroxide presence from fabrics and the reaction bath. The treatment of the water bath allows it to be recycled for next steps. The iodimetric titration of peroxide presence showed that no rest were found neither in fabrics nor in the reaction bath.

### 3.4 Global enzymatic wet processing of cotton

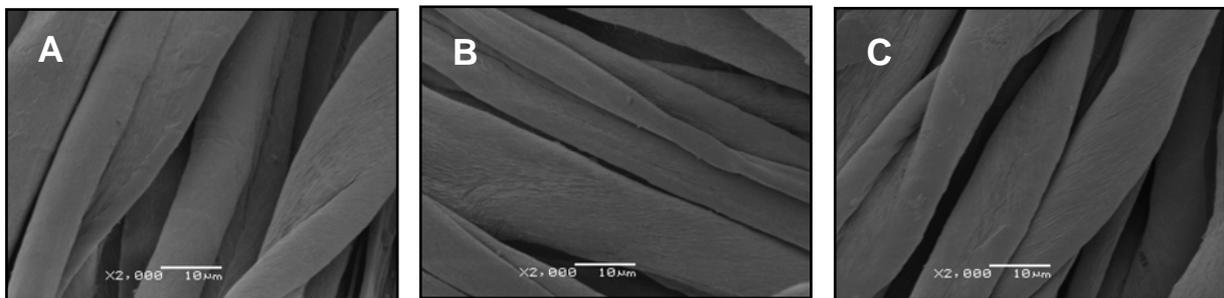
After a successful enzymatic desizing and scouring was achieved the aim was to further add to the combinatorial processing of cotton the bleaching and polishing steps. A series of tests were carried out to study the effect of the different treatments onto fibres and fabrics. A simple following up test is to measure the weight loss. After desizing and scouring fabrics have a considerable weight reduction because of starch and impurities elimination. After bleaching and polishing is performed, a further reduction in weight is achieved due to superficial cellulose fibrils detachment (Figure 3).



**Figure 3.** Comparison of weight reduction after desizing and scouring treatment and global enzymatic processing.

The results from the determination of the recovery from creasing (UNE EN 22313:2002) indicate that the angle of recovering is similar for all tested treatments: conventional desizing and scouring, enzymatic desizing and scouring and the global enzymatic treatment. This means that there are no differences in modification at the superficial level. The tensile properties of fabrics test (EN ISO 13934/1:1999) indicates changes at the fibre level structure that evolves to a less resistant fabric. In this case there are no differences between untreated cotton and the desized and scoured one, but on the other hand, adding further processes (bleaching and polishing) do have a negative effect in the structure.

Fibre evaluation by Scanning Electron Microscopy showed no difference between enzymatically treated cotton and alkaline desizing and scouring (Figure 4).



**Figure 4.** Scanning Electron Microscopy pictures of (A) Conventional desizing and scouring (B) Desized and scoured fibres with the use of enzymes (C) Global enzymatic processing.

#### 4. CONCLUSIONS

From the achieved results we can conclude that cotton wet processing taking advantage of enzymatic catalysis is successfully feasible. The longer time consuming procedure for desizing and scouring in comparison with the conventional method is waded by the time that is not lost between process. On the other hand, savings in water are also important. The reaction bath from the desizing and scouring processes can be used later on for the polishing. Similarly, the bleaching liquor can be recycled after its peroxide-free status.

This is our first attempt to combine several cotton processing steps in a semi-continuous way. The obtained results need of further investigations and development, but it is a very good starting stage for future improvements.

#### 5. ACKNOWLEDGEMENTS

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