Biotech Highlight

Biocatalytic pre-treatment processes of cotton: Industrial application of academic research

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Received 27 March 2011
Revised 5 May 2011
Accepted 11 July 2011

Keywords: Biocatalytic · Cost effective · Cotton · Industrial scale · Pre-treatment

1 Introduction

Much research effort is invested in developing enzymatic treatments of textiles by focusing on the performance of enzymes at the laboratory scale. Despite all of this work, upgrading these developments from the laboratory scale to an industrial scale has not been very successful. Nowadays, companies are confronted with rapid developments of markets, logistics, and social and environmental responsibilities. Moreover, these organizations have to supply an ever-increasing amount of information to the authorities, shareholders, lobbyists, and pressure groups. Companies have tried to fulfill all of these demands, but this has often led to the loss of focus on new products and process development. However, both theory and practices of breakthrough innovations have shown that those rightfully proud of previous successes are usually not the ones that led the introduction of new technology, as shown and excellently documented by Christensen [1]. The textile industry is no exception to this observation. With the lack of management impetus for new product and process developments, companies began to reduce investments in these activities. However, this results in a reduction of the size of the company or even closure. Besides the hesitation from the top management of textile companies to focus on new developments, middle management level is also reluctant to evaluate and implement developments in new products and processes. One of the reasons for this reluctance is that many processes in the textile industry are not fully explored or known. From this lack of knowledge, it is easy to explain that there is hesitation for change, since not all consequences of a change in processing or production can be predicted. Often new developments cannot be fully tested and evaluated on the laboratory- or pilot-scale level. This is caused by the impossibility of mimicking industrial-scale production in a laboratory. Additionally, pilot-scale equipment is very expensive and for many companies it is not realistic to invest in this type of equipment.

Fortunately an increasing number of textile companies have realized that they have to invest in new products and processes for their future survival and prosperity. New developments are decisive for future successes. If such companies decide to invest in new developments, it is clear that with the scarcity of capital for product and process developments, the chance of failure should be minimized. For successful process and product development, it is necessary to organize the development process with external partners because it is clear that it is almost impossible for individual textile companies to control the process from idea generation to academic research, implementation research, and development and industrial testing. These issues are especially characteristic for small- and medium-sized enterprises (SMEs).

Herein, the collaboration has been organized on two research levels. The first research level is

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Abbreviations: COD, chemical oxygen demand; **EFSM**, Engineering of Fibrous Smart Materials; **PU**, Pollution unit; **Nkj**, Nitrogen Kjeldahl value; $\mathbf{Q_d}$, average wastewater flow in m^3 per 24 h; $\mathbf{Q_y}$, average wastewater flow in m^3 per year

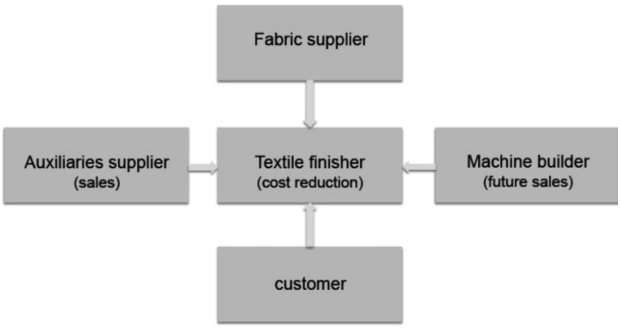

Figure 1. Simplified scheme of the position of the partners in the value chain.

knowledge and know-how based. The universities and chemical suppliers worked closely together to investigate the new process. The aim was to explore the influence of process conditions and interactions of chemicals in sub-process steps as a result of the treatment. The second level is that of the industrial implementation of the new process. The universities and chemical suppliers worked closely together with different industries to implement the newly developed process. The focus in this part of the research was the interaction between the chemistry of the new process, equipment, and fabrics.

A co-operation between the beneficiaries of the new process was established. The selection criterion for the co-operation was "who will earn something with the new process". To answer this question, the value chain has been drawn as the simplified scheme shown in Fig. 1 [2].

2 The importance of the value chain and the research funnel

The question of the beneficiaries of the new process is not difficult to judge by looking at Fig. 1. Here the value chain is drawn vertically and the suppliers of the finishing company are drawn horizontally. Based on this scheme the beneficiaries of the new process have been defined as follows:

1. Textile finishing company
   The benefits for the textile finishing company are cost savings on the consumption of water and energy and a reduction in wastewater costs.
2. Auxiliaries supplier
   It is expected that new chemicals, compared with those used in traditional pre-treatment processes, will be used. This gives sales opportunities for the auxiliary supplier.
3. Machine manufacturer
   It is likely that the new process can be applied to the currently installed base. Although no direct increase in sales will occur for the machine builder, it is likely that this newly developed process will lead to improvement of and innovations for the installed base for further optimization of this process, resulting in sales opportunities. This is, however, only expected for future optimization projects.

Based on this knowledge, it is clear that successful implementation of the new process depends on the co-operation of the finisher and the auxiliaries supplier. The role of the auxiliaries supplier is to explore and prepare new products for implementation in the textile industry. In this case, the development finds its origin in academic research. The newly developed knowledge has to be implemented by industry. Before implementation, it is often necessary to 'transform' academic knowledge into industrially applicable technologies, where the interaction between chemistry, the installed base, and process conditions, in particular, have to be explored. Only this total approach gives the best chance for successful implementation. Institutes that operate between universities and industry may facilitate the transition from academic knowledge into industrially applicable processes. Here a clear role can be defined for universities of applied science. The development process from academic knowledge to industrial implementation is schematically shown in Fig. 2. In this work, the following partners were involved:

- TanatexChemicals in Ede in the Netherlands. This partner is a supplier of textile auxiliaries;
- TenCate Advanced Textiles in Nijverdal in the Netherlands. This partner is a finishing company with continuous full-width pre-treatment equipment;
- Vlisco in Helmond in the Netherlands. This partner is also a finishing company. The pre-treatment process here is done in rope form.

These partners are all members of the Engineering of Fibrous Smart Materials (EFSM) Foundation, in which industrial partners are united and support the chair of the EFSM at the University of Twente.

From Fig. 2 it is clear that the development of academic knowledge involves long-term research. Research at universities for applied science is medium-term research that should deliver the required technology. Industry finally performs the short-term development work. In this research process from academic research to industrial ap-

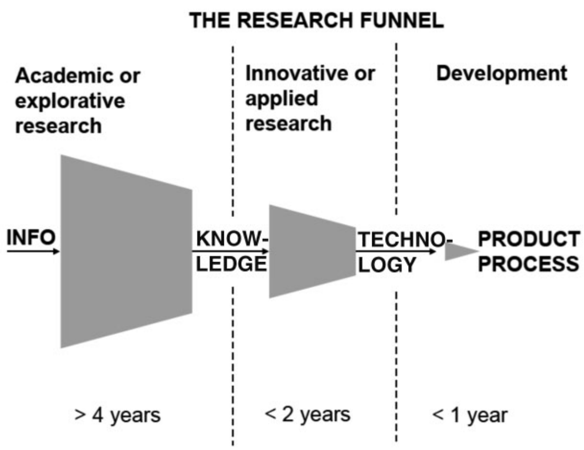

Figure 2. The research funnel (McKinsey).

plication, there are researchers involved with different focuses. To implement a new process, which is developed after academic research, it is necessary to 'translate' the academic knowledge before it can be applied in industry. Saxion University of Applied Science in Enschede in the Netherlands played a key role in the translation of this academic knowledge.

The new pre-treatment process for cotton is expected to be beneficial in terms of savings in the costs of water and energy, and a reduction in wastewater treatment costs [3]. These were important points to communicate and consider during this work to retain momentum.

3 Results

Based on lab-scale experiments, the new process has been tested at Satta e Bottelli in Italy in a hot pad batch bleach pre-treatment process for cotton. The changes and benefits of this process are shown in Table 1.

The industrial experiments are done with percale fabric. Percale is a woven, 100% cotton fabric with fine yarns and a high yarn count in the warp and weft directions. The weight of the fabric is approximately 130 g/m^2. In Table 2 the results of the industrial bio-catalytic pre-treatment experiment have been compared with a standard processed sample and generally accepted values. In the experiments, the performance of the enzymes amylase, cutinase, and pectinase have been reported in terms of desizing value, hydrophilicity, and pectin content. The whiteness of the fabric is reported in terms of degree of whiteness and the *S* factor, which is an indication of the damage to the fabric after bleaching. It is expected that the bleaching step can be improved by optimizing the concentrations of bleaching chemicals.

3.1 Financial evaluation of the reduction in water, energy, and wastewater costs

Much information on process costs in pre-treatment for various companies has been gathered. All of this information is incorporated into a 'model' pre-treatment company [4]. This hypothetical company produces, on a weekly basis, 500 000 running meters of a specific 100% cotton fabric. The production activity is pre-treatment and is concentrated on one single production line. In this work, we concluded that the savings in water, wastewater, and energy were possible and these were the drivers for the implementation of the new process.

3.1.1 Water consumption

Savings on water consumption during the rinsing process after bleaching are expected to be realized after optimization of the new process on an industrial scale. Bleaching in the new process is done with lower alkali concentrations than in the conventional pre-treatment. The reduction in alkali concentration is possible because the degradation

Table 1. An overview of the differences in the use of chemicals and process conditions for conventional and new processes after the first industrial experiment, May 2010, at Satta e Bottelli

	Conventional process	Newly designed process
Chemistry	amylase bleaching agents: high alkali quantities	amylase (1.7 g/L), cutinase (2.25 g/L), pectinase (1.5 g/L) bleaching agents: low alkali quantities
Process conditions		
Impregnation	60°C, pick-up 70%	60°C, pick-up 70%
Incubation	room temperature	room temperature
Rinsing	>95°C, 6 L/kg	<50°C, 6 L/kg
Impregnation bleach	room temperature	room temperature
Incubation in a steamer	100°C, 100% relative humidity (rh)	100°C, 100% rh
Rinsing	>95°C, 7 L/kg	<60°C, 6 L/kg

Table 2. Evaluation of greige, standard Satta e Bottelli pre-treatment, and generally accepted performance values with the bio-catalytic pre-treatment experiments on industrial scale[a)]

	Greige	Standard process	Generally accepted value	Bio-catalytic pre-treatment
Degree of whiteness (Cie/D65) basic	–	78	> 70	71
Indication of damage				
DP value	2371	1922	–	2033
S factor	–	0.26	< 0.50	0.19
Desizing				
TEGEWA violet scale value	starch	9	≥ 7	7
PVA presence	PVA	–	–	–
Hydrophilicity				
Time (s)	–	1	≤ 3	2
Warp (mm)	–	30	–	32
Weft (mm)	–	32	–	30
Pectin content (Ruthenium red test)				
Color strength (% vs. greige 100%)	100	37	< 50	44

a) *S* factor = damage factor (Schaedigungsfaktor); DP = degree of polymerization, TEGEWA = German Textile Research Association, PVA = polyvinyl alcohol.

of pectines in the primary wall of cotton is already realized in the bio-scouring step. This makes a substantial reduction of NaOH in the bleaching treatment possible, which allows less intensive rinsing after bleaching. To date, almost 10% water reduction was realized and this was used in the savings calculation. The expected ultimate water reduction in this process step was 20%. In the desizing and bio-scouring step, it was expected that the water consumption could increase because this effluent now also contains the degradation products of the wax and pectin. Industrial tests, however, showed that it was not necessary to increase the water flow in the rinsing compartments after this process step. In the financial evaluation, the cost of water is expressed in costs per m^3 of industrial water.

3.1.2 Energy consumption

The consumption of energy decreased substantially. Temperatures in the rinsing compartments decreased from 95 to 50°C and a further reduction of the rinsing temperature, even down to 30°C, was possible. No energy reduction was realized in the steamer. The conditions for the impregnated fabric in a steamer are 100% relative humidity at 100°C. Without additional capital investment, it is not possible to explore incubation conditions in a steamer below 100°C. Under these conditions, there is a high risk of condensation spots on the treated fabric, which can be seen only after dyeing. If the temperature in the steamer could be decreased, this would result in less cost savings than the cost savings realized by a reduction in the rinsing temperature. This was understood from the fact that the water content per kg fabric impregnated with the bleaching chemicals was only a fraction of the water quantity per kg fabric needed for rinsing. Therefore, the energy savings were only calculated for energy reduction realized in the rinsing processes. To calculate the energy cost reduction, the cost price of natural gas was used based on the reasoning given below.

Climate change and greenhouse effects are important subjects. Therefore, international conferences have been organized, where targets and regulations for Climate Change prevention were announced. The leading organization in these discussions and regulations is the United Nations organization. The ultimate objective of the United Nations Framework Convention on Climate Change (UNFCCC) is stabilization of greenhouse gas concentrations in the atmosphere at a level that would prevent dangerous anthropogenic interference with the climate system. The Kyoto protocol is an international agreement that is linked to the UNFCCC. The major feature of the Kyoto protocol is that it sets binding targets for 37 industrialized countries and the European Community for reducing greenhouse gas ($GHG=CO_2$) emissions. Over the five-year period from 2008 to 2012, an average reduction of 5% against the 1990 level is targeted (http://unfccc.int). Countries that have agreed the Kyoto treaty must meet their targets, primarily through national measures. However, the Kyoto protocol offers the countries additional means to meet their targets. The most well-known mechanism is "Emissions Trading". This system has been adopted in Europe since January 1st, 2005. In this system, organizations get greenhouse gas emission rights assigned. The emission rights have a direct relationship with their consumption of natural gas (www.nlenergieenklimaat.nl). To reduce the emission of greenhouse gas, a tax was introduced that was added to the price of natural gas at an amount of €0.029/m^3 (private communication with Kamphuis, 2010). The target of this emission right is to encourage the reduction of the consumption of natural gas. The greenhouse gas effect is separately calculated in the cost savings calculation.

3.1.3 Reduction of the pollution of wastewater

For the quantification of the pollution load of an effluent, the chemical oxygen demand (COD) value of that effluent is used. The COD value is usually taken as the summation parameter for the organic loading of the effluent and used as a limiting value for permitted discharge and also as a measurement for calculating effluent charges [5]. In this work, the COD value was used to calculate the savings of the new process. Samples of effluent were collected at the host company, Satta e Bottelli, after the rinse from the bio-scouring and bleaching step. These values were compared with samples taken during conventional treatment prior to the trials. The COD value decreased from 12.070 to 7.543 mg O_2/kg effluent. A decrease in the COD value of 4.427 mg O_2/kg effluent was realized. This value was used in the financial evaluation of the new process. To calculate the costs of wastewater, the term pollution unit (PU) is introduced here. PU is calculated with Eq. (1), which is also used in industry as a simplified calculation model [6]:

$$\mathrm{PU} = \frac{C_\mathrm{d}}{150}(\mathrm{COD} + 4.57\mathrm{Nkj}) \tag{1}$$

in which Q_d is the average wastewater flow in m^3 per 24 h, COD is measured in mg O_2/kg effluent, Nkj is the nitrogen Kjeldahl value. Nkj is expressed in g N/m^3 wastewater effluent [6]. To convert 1 g of N into NO_3, 4.57 g O_2 has to be used (private communication with Gooijer, 2010). In pre-treatment processes, the COD value dominates over the Nkj value, and therefore, the Nkj value is not used. To calculate the cost of wastewater, the average effluent per 24 h has to be calculated. The average effluent in the pre-treatment department is the daily production volume in kg multiplied by the water consumption per kg fabric, expressed in m^3, corrected for off-peak times for holidays and so forth, and finally the effluent volume, Q_d, is found. The Dutch Water Authorities, namely, Waterschap Regge en Dinkel, make use of a model to find the pollution unit where annual discharge forms the basis [Eq. (2)]:

$$\mathrm{PU} = \frac{Q_\mathrm{y}(\mathrm{COD} + 4.57\mathrm{Nkj})}{1000 \times 54.8} \tag{2}$$

The value Q_y is the annual discharge volume (effluent) and the values of COD and Nkj are identical to those values in Eq. (1). The constant 150 in the Eq. (1) is the approximation of the constants in Eq. (2) divided by 365, which is the number of days per year. The constant 1000 is used to convert the COD value from mg/L to g/L and the constant 54.8 is the consumption of oxygen for the purification of 1 PU of wastewater (private communication with Waterschap Regge en Dinkel in the Netherlands).

From the calculation model, it is clear that two options can be followed to reduce the costs of wastewater. The first option is to decrease the COD value of the effluent and the second option is to reduce the wastewater effluent. In the new process, the COD value of the wastewater was reduced, leading to substantial cost savings.

To calculate the cost savings, the assumption was made that there was an imaginary textile company with a weekly production volume of 500 000 running meters of full-width cotton fabric. The pretreatment process is called hot pad batch bleach. The process and production data from the imaginary finisher and the physical constants and prices are shown in the following survey:

In Table 3 the process settings used by Satta e Bottelli are given. Furthermore, the reduction of water and energy consumption, as well as the reduction of the wastewater pollution, is copied from the former company. The unit prices of gas, water, and PU were estimated after discussion with the partners.

Based on the former assumptions and realized reduction of the consumption of water and energy and the reduced wastewater pollution, the annual savings were calculated as follows:

Water	k€	4
Energy	k€	145
Greenhouse gas emission rights	k€	17
Wastewater	k€	449
Total annual savings	**k€**	**615**

3.2 Chemical costs

The chemicals used for conventional pre-treatment of cotton are amylase, detergents, NaOH, H_2O_2, sequestering agents, and peroxide stabilizers. The desizing step in the new process was transformed into a bio-scouring step, where besides the well-known amylase and detergent, the enzymes

Table 3. Survey of process and production data that have been used to calculate the cost reduction of the new process at an imaginary finisher. The process data have been collected from the Satta e Bottelli trials

Product information and quantities		
Composition	100% cotton	
Fabric weight	180 g/m^2	
Fabric width	1.75 m^1	
Annual production volume in m^1	23×10^6 m^1	
Annual production in kg	7.3×10^6 kg	
Process data	**Conventional**	**Bio-catalytic**
Impregnation temperature with enzymes	60°C	60°C
Incubation	room temperature	room temperature
Rinse after enzymatic incubation	6 kg water/kg fabric, 100°C	6 kg water/kg fabric, 50°C
Impregnation temperature with bleaching agent	room temperature	room temperature
Incubation in the steamer	100°C	100°C
Rinse after bleaching	6 kg water/kg fabric, 100°C	6 kg water/kg fabric, 50°C
Acidification	1 kg water/kg fabric	0 kg water/kg fabric
COD value in mg/L	12 070	7543
Data and costs		
Water		
Density	1000	kg/m^3
Heat content	4.18×10^3	J/kg/K
Cost in €	0.60	L/m^3
Natural gas		
Combustion value	31.65×10^6	J/m^3
Cost in €	0.25	L/m^3
CO_2 emission right	0.029	L/m^3
Cost of 1 PU in €	50	L/PU

cutinase and pectinase were also used. This process step was optimized, concerning the concentrations of chemicals and rinsing temperature. To date, the enzyme cutinase, used in the enzyme cocktail, has been produced only in relatively small quantities, and therefore, the commercial price is rather high. Therefore, in the beginning of the application of the new process part of the savings will be used to compensate for the increased chemical costs. However, it is expected that the cost price for cutinase will decrease with increasing turnover, and therefore, the cost per meter fabric will decrease.

In the bleaching step, it is expected that the quantity of NaOH will drastically be reduced, but a reduction in the concentration of H_2O_2 as well as the peroxide stabilizer are also expected. The sequestering agent and detergent are estimated to be constant. The extra chemical costs in the bleaching process are caused by the use of the manganese catalyst. The use of this catalyst might be necessary for the activation of the decomposition of H_2O_2 in low-alkaline bleaching conditions.

In conventional pre-treatment processes, the cost of chemicals is in the order of magnitude of €0.065 per kg fabric. Based on the savings calculation previously described, it is now possible to calculate the maximum cost increase for the chemicals, based on a breakeven situation for the finisher. In that case, all cost savings would be consumed by the increase in the chemical costs of the new process. The cost savings of the new process were calculated in the previous section at k€615 for a production volume of 7.3×10^6 kg. This means that the savings are in the order of magnitude of €0.084 per kg fabric. So, in a breakeven situation, the chemical costs per kg fabric of the conventional process can increase by 129%, which is unlikely. Based on the sales prices for experimental quantities of cutinase and the manganese catalyst, it has been calculated that the chemical costs will increase by €0.06/kg fabric. It is likely that the sales prices for the chemicals mentioned will decrease with increasing sales volumes. Therefore, it can be concluded that the new pre-treatment process is not only advantageous is terms of sustainability, but also in terms of cost reduction.

4 Conclusion

Based on the results of the industrial experiments, it is possible to calculate the benefits of biocatalytic pre-treatment in terms of cost reduction. The calculation is based on the realized process conditions at Satta e Bottelli. The cost reduction on water, energy, and wastewater reduction are substantial. It is also discussed that the total costs of chemicals will probably rise, but it is unlikely that the extra chemical costs will exceed the savings. This means that the textile finisher can realize major savings after implementing the new process.

It is clear that efficient production facilities are able to save up to €0.084 per kg fabric. This means that the theoretical global savings for 25 million metric tons of cotton are in the order of a magnitude of €2.1 billion. These savings confirm the savings postulated at Autex in 2007 [2]. By taking into account the fact that the majority of cotton pretreatment facilities are not efficient in the consumption of water and energy, it is likely that the global savings can be significantly higher. So, it can be concluded that the implementation of biocatalytic pre-treatment processes for cotton are not only environmentally friendly, but also improves the profit of textile companies. It is also shown that leadership in innovation enables the industrial implementation of a new innovative production process improvement.

We thank TanatexChemicals in Ede, the Netherlands, for supplying the chemicals for the industrial trials and Saxion University of Applied Science and University of Twente/Chair EFSM for their financial support.

The authors have declared no conflict of interest.

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