ORGANIZATION OF INDUSTRIAL APPLICATION OF ACADEMIC RESEARCH ON BIO-CATALYTIC PRE-TREATMENT PROCESSES OF COTTON

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Introduction

A lot of research effort is put in developing enzymatic treatment of textiles by focusing on the performance of enzymes on lab-scale. Despite all this work upgrading of these developments from lab-scale to industrial scale has not been really successful. Companies are nowadays confronted with rapid developments of markets, logistics and social and environmental responsibilities. Moreover these organizations have to supply an even increasing amount of information to the authorities, shareholders, lobbyists and pressure groups. Companies have tried to fulfill all these demands, but this led often to the loss of focus on new product and process development. However, both theory and practices of breakthrough innovations has shown that those rightfully proud on previous successes in the past, are usually not the ones that lead the introduction of new technology, as was shown and excellently documented by Harvard professor Clayton Christensen [Christenson, 2003]. The textile industry is no exception in this observation. With the lack of management impulses on new product and process developments companies began to reduce the investments in these activities. Finally, however, this will result in a reduction of the size of the company or even closing down. Besides the hesitation from the top management of textile companies to focus on new developments it is also seen that the middle management level is reluctant to evaluate and implement developments in new products and processes. One of the reasons for this reluctance is that many processes in textile industry are not fully explored and known yet. From this lack of knowledge it is easy to explain that there is hesitation for changes, since not all consequences of a change in processing or production can be overseen. Often new developments cannot be fully tested and evaluated on lab- or pilot scale level. This is caused by the impossibility to mimic industrial scale production in a lab. Besides of that, 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 realize 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 obvious that with the scarcity of capital for product- and process developments, the chance of failures should be minimized. For successful process- and product development it is necessary to organize the development process with external partners, as it is clear that it is almost not possible for individual textile companies to control the process from idea generation, academic research, implementation research and development and industrial testing. These issues are specially characteristic for small and medium sized enterprises (SME's).

In the present work the collaboration has been organized on two research levels. The first research level is knowledge and know-how based. Here the universities and the chemical supplier worked closely together to investigate the new process. The aim was to explore the influence of process conditions and interaction of the chemicals in the sub process steps on the result of the treatment. The second level is that of the industrial implementation of the new process. Here universities and chemical supplier 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 has been 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 in a simplified scheme in figure 1 [Bouwhuis et al. 2007].

The importance of the value chain and the research funnel

The question of the beneficiaries of the new process is not difficult looking at figure 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:

1. Textile finishing company
   The benefits for the textile finishing company are cost savings on the consumption of water and energy and the reduction of the wastewater costs.
2. Auxiliaries supplier
   It is expected that new chemicals, compared to the traditional pre-treatment process, will be used. This gives sales opportunities to the auxiliary supplier.
3. Machine manufacturer

It is likely that the new process can be applied on present installed base. Although no direct increase of sales will occur for the machine builder it is likely that this new developed process will lead to improvement of and innovations on installed base for further optimization of this process, resulting in sales opportunities. This is however only expected for future optimization projects.

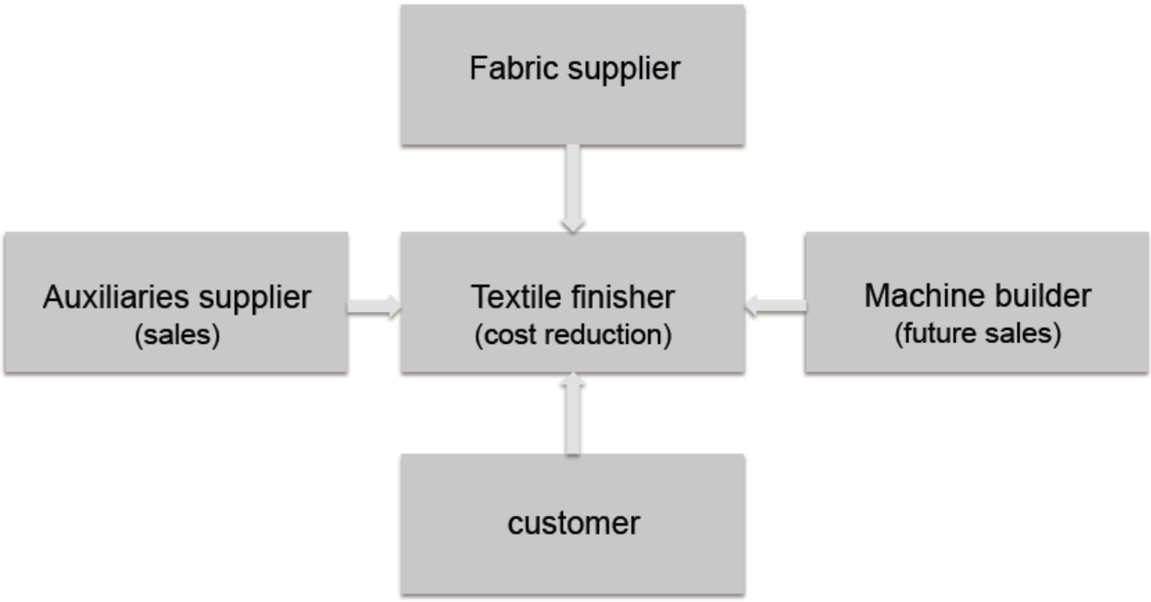

Figure 1: Simplified scheme of the position of the partners in the value chain

Based on this knowledge it is clear that successful implementation of the new process depends on co-operation of the finisher and the auxiliaries supplier. The role of the auxiliaries supplier is to explore and prepare the new products for implementation in textile companies i.e. 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 in industrially applicable technologies, where especially the interaction between chemistry, installed base and process conditions has to be explored. Only this total approach gives better chances for successful implementation. Institutes that operate between universities and industry may do the transition of the academic knowledge into industrial 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 drawn in figure 2: the research funnel. 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. Also this partner is a finishing company. The pre-treatment process here is done in rope form.

These partners are all member of the Foundation EFSM, in which organization the industrial partners are united, who support the chair EFSM at the University of Twente.

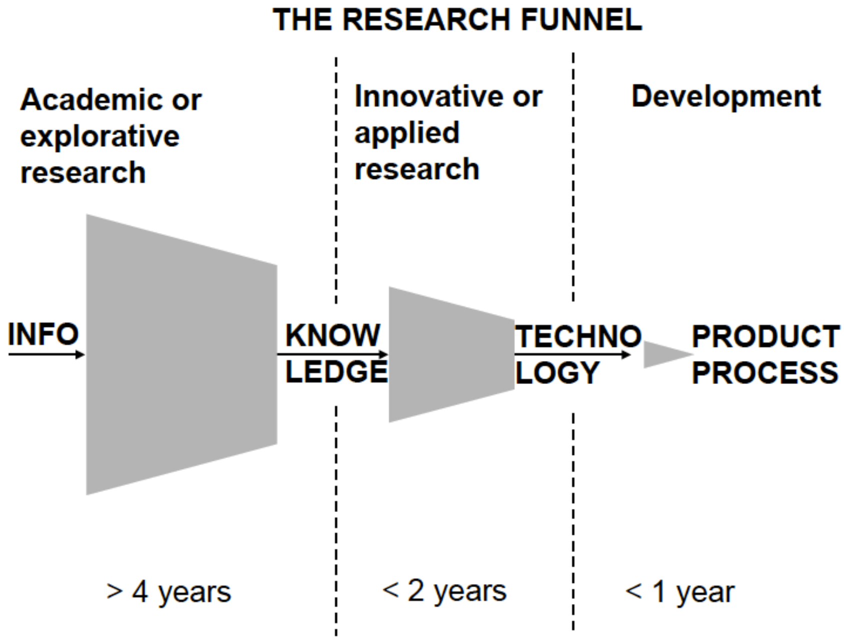

Figure 2: The research funnel (McKinsey)

From figure 2 it is clear that the development of academic knowledge is long-term research. The research at universities for applied science is medium term research that should deliver the required technology. The industry finally does the short-term development work. In this research process from academic research to industrial application there are researchers involved with different focus. 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 on the costs of water and energy and reduction of the wastewater treatment costs [Bouwhuis et al. 2009]. This was an important item for communication during this work to retain the momentum.

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

Satta e Bottelli Case		
Fabric: 100% cotton, percale	Conventional process	New designed process
Chemistry	amylase	amylase (1.7 g/l), cutinase (2.25 g/l), pectinase (1.5 g/l)
	bleaching agents: ***high*** alkali quantities	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 % rh	100°C, 100 % rh
Rinsing	>95°C, 7 l/kg	<60°C, 6 l/kg

Table 1. Overview of Satta e Bottelli Case, performance after 1st industrial experiment, Mai 2010.
The performance of the treated fabric is comparable with conventional treated fabric; except whiteness, which stays behind. It is expected that further optimization will lead to an improved whiteness.

Financial evaluation on cost reduction of water, energy and wastewater
A lot of information on process cost in pre-treatment in various companies has been gained. All this information is incorporated in a 'model' pre-treatment company [Bouwhuis, 2011]. This hypothetical company produces on a weekly basis 500,000 running meter in a specific 100% cotton fabric. The production activity is pre-treatment

and is concentrated on one single production line. In this work we have concluded that the savings on water, wastewater and energy are possible and these are the drivers for the implementation of the new process.

Water consumption

Savings on water consumption during the rinsing process after bleaching are expected, to be realized after optimization of the new process on industrial scale. Bleaching is done in the new process with low alkali concentration, compared to the conventional pre-treatment. The reduction of alkali is possible, because the degradation 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. Until now almost 10% water reduction has been realized and this has been used in the savings calculation. The expected ultimate water reduction in this process step is 20%. In the desizing and bio-scouring step it has been expected that the water consumption could increase because in this effluent now contains also 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 are expressed in costs per m^3 industrial water.

Energy consumption

The consumption of energy is 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, seems to be possible. No energy reduction is 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 smaller cost savings compared to the cost savings realized by reduction of the rinsing temperature. This can be understood from the fact that the water content per kg fabric that is impregnated with the bleaching chemicals is only a fraction of the water quantity per kg fabric that is needed for rinsing. Therefore the energy savings are only calculated for energy reduction that is realized in the rinsing processes. To calculate the energy cost reduction the cost price of natural gas has been used based on the following reasoning.
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 2008 – 2012 an average reduction of 5% against the 1990 level is targeted [UNFCCC 2010]. 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 is adopted in Europe since January 1st, 2005. In this system organizations get greenhouse gas emission rights assigned. The emission rights have a direct relation to their consumption of natural gas [Agentschap.nl, the former Senter Noven, 2010]. To reduce the emission of Greenhouse Gas a tax has been introduced that is added to the price of natural gas at the amount of € 0.029/m^3 [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.

Reduction of the pollution of wastewater

For the quantification of the pollution load of an effluent the COD-value (Chemical Oxygen Demand) 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 [Rouette (Ed.), 2001]. In this work the COD-value has been used to calculate the savings of the new process. Samples of effluent are collected at the hosting company Satta e Bottelli after the rinse of the bio-scouring and bleaching step. These values are compared with samples that have been taken during conventional treatment prior to the trials. The COD-value decreased from 12.070 to 7.543 mg O_2/kg effluent. So here a decrease of COD-value is realized of 4.427 mg O_2/kg effluent. This value will be used in the financial evaluation of the new process. To calculate the costs of wastewater the term pollution unit is introduced here. This pollution unit (PU) is calculated with formula [1], which is also used in industry as a simplified calculation model [Stoop, 2003; Gooijer, 2010]:

$$PU = \frac{Q_d}{150}\left(COD + 4.57Nkj\right) \qquad [1]$$

in which Q_d is the average wastewater flow in m^3 per 24 hours, COD is the chemical oxygen demand in mgr O_2/kg effluent, Nkj is the Nitrogen Kjeldahl value. Nkj is expressed in gram N/m^3 wastewater effluent [Stoop, 2003]. To convert 1 g N into NO_3, 4.57 g O_2 has to be used [Gooijer, 2010]. In pre-treatment processes the COD-value is dominating over the Nkj-value and therefore the Nkj-value is not used. To calculate the cost of wastewater the average effluent per twenty-four hours has to be calculated. The average effluent in the pre-treatment department is the daily production volume in kg multiplied with the water consumption per kg fabric, expressed in m^3, corrected for off time for holidays etc. and finally the effluent volume Q_d has been found. The Dutch Water Authorities, i.e. Waterschap Regge en Dinkel, make use of a model to find the pollution unit where annually discharge forms the basis. This formula reads:

$$PU = \frac{Q_y\left(COD + Nkj\right)}{1000 \times 54{,}8} \qquad [2]$$

The value Q_y is the annual discharge volume (effluent) and the values COD and Nkj are identical to these values in formula [1]. The constant 150 in the equation [1] is the approximation of the constants in [2] divided by 365, being 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 the 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 for reduction of the costs of wastewater 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 has been reduced, leading to substantial cost savings.

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

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 mgr/l	12,070	7,543

Data and costs		
- **water**		
density	1000	kg/m^3
heat content	4.18×10^3	J/kg/K
cost in €	0.60	1/m^3
- natural gas		
combustion value	$31{,}65\times10^6$	J/m^3 *
cost in €	0.25	1/m^3
CO_2 emission right	0.029	1/m^3 *
- cost of 1 pollution unit (PU) in €	50	1/PU

Table 1 Survey of process and production data of the imaginary finisher

- at ambient conditions of 20°C and 1 bar

In table 1 the process settings of Satta e Bottelli have been used. Further 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 are 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 following annual savings have been calculated:

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

Chemical costs

The chemicals that are 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 has been transformed in a bio-scouring step, where besides the well-known amylase and detergent also the enzymes cutinase and pectinase have been used. This process-step is now optimized concerning the concentrations chemicals and rinsing temperature. Until now the enzyme cutinase that was used in the enzyme cocktail has been produced only in relatively small quantities and therefore the commercial price is rather high. So in the beginning of the application of the new process a part of the savings will be used to compensate the increased chemical costs. However it is expected that the cost price for cutinase will decrease with increasing turnover and therewith the cost per meter fabric will decrease.

In the bleaching step it is expected that the quantity NaOH will drastically be reduced but also a reduction of the concentration H_2O_2 as well as the peroxide stabilizer are 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 are in the order of magnitude of € 0.065 per kg fabric. Based on the savings calculation as previously described it is now possible to calculate the maximum cost increase for the chemicals, based on a break-even situation for the finisher. In that case all cost savings would be consumed by the increase of the chemical-costs of the new process. The cost savings of the new process have been calculated in the previous section at k€ 615 for a production volume of $7{,}3 \times 10^6$ kg. This means that the savings are in an order of magnitude of € 0.084 per kg fabric. So in a break-even situation the chemical costs per kg fabric of the conventional process can increase with 129%, which is unlikely.

Conclusions

Based on the results of the industrial experiments it is possible to calculate the benefits of bio-catalytic 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 ton cotton are in an order of magnitude of € 2.1 bln. These savings confirm the savings as it has been postulated at Autex in 2007 [Bouwhuis et al., 2007]. Taking into account that the major part of cotton pre-treatment 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 bio-catalytic pre-treatment processes for cotton are not only environmentally friendly but also improves the profit of textile companies. It was also shown that leadership in innovation enables the industrial implementation of a new innovative production process improvement.

References

Agentschap NL (in dutch), the former Senter Novem, www.nlenergieenklimaat.nl last time visited October 4th, **2010**

Bouwhuis, G.H., V.A. Nierstrasz and M.M.C.G. Warmoeskerken, "The use of (bio)catalysts and ultrasound in the pretreatment of cotton: Application on industrial scale as a sustainable process", Proceedings of the 7th AUTEX Conference, Tampere, Finland, 26-28 June **2007**

Bouwhuis G.H., B. Dorgelo and M.M.C.G. Warmoeskerken, "Textile pretreatment process based on enzymes, catalyst and ultrasound", Melliand International, Volume 4, p150 – p151, **2009**

Bouwhuis, G.H., "The design of a novel, environmentally improved, industrial cotton pre-treatment process", ISBN 987-90-365-3153-5, Thesis University of Twente, the Netherlands, **2011**

Christenson, C., "The innovator's dilemma", Harper Business Essentials, ISBN 0-06-052199-6, p 193-202 **2003**

Gooijer, H., private communication, October 1st, **2010**

Kamphuis, B., private communication, October 4th, **2010**

Stoop, M.L.M., "Water management of production systems optimized by environmentally oriented integral chain management: case study of leather manufacturing in developing countries", Technovation 23, p 265 - 278, **2003**

UNFCC, home page, http://unfccc.int ,last time visited October 4th, **2010**

Rouette, H.K., "Encyclopedia of Textile Finishing", ISBN 3-540-65031-8, Springer Verlag Berlin/Germany, **2001**