Energy savings in dyeing fixing process of cotton fabrics by steaming

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Abstract: In this work, a traditional dyeing fixing process by steaming is studied, in order to improve both the coloration quality of the final obtained fabrics and the energy performances of the process. Starting from the factor's ranges currently in use by textile industrial operators, a full factorial design with two factors (steam flow rate and steaming time) and three levels is settled following the guidelines of Design of Experiments (DOE). A multiple response model characterized by different subset of regressors is proposed and the best one is chosen by means of Mallows' C_p, R^2 and R^2(adj) and S criterions. The analysis puts in evidence that the obtained predictive models point out the high influence of the time spent by the fabric in the steaming chamber, while the one of the steam flow rate can be considered negligible. This fact leads to the opportunity to produce the best quality fabrics at the lowest level of steam flow rate, maintaining the steaming time under the traditionally employed one of 14 minutes, actually 11.1 minutes, results which can be translated in consistent energy and, consequently, economical savings if applied to industrial size equipment.

Keywords: Cotton, steaming, DOE, process optimization.

1.Introduction

In the traditional textile printing fabric, the dye is mixed with the reagents in order to obtain a print paste. Normally, a steaming process is necessary to fix the dye, by means of the color's diffusion from the surface onto the fabric, until it reaches the inner part of the fibres.

The current world economic situation leads to the necessity to increase the performances and, consequently, the quality of the techniques employed to obtain the final products and improve their energy efficiency. In addition, nowadays is very difficult to propose to an industrial operator any modification to processes or to plant layouts that already reached a satisfying level of reliability. In general, innovation, even if promisingly successful, is usually hindered by the need of investments characterized by a long return time. In the present work, it is intention of the Authors to demonstrate that, in the specific case of dye fixing by steaming, a more rational use of the current and well-founded technologies gives the opportunity to increase the quality of the products and the energy efficiency of the process without any expenses whose break even point could be collocated in a long period of time.

2. Literature review

At the moment, the most employed process to fix pre-printed color on natural fibre fabrics is the one which makes use of saturated water steam, with pressures ranging from 3 to 4 bars (Corbani 1990). Several studies have been conducted in order to increase the efficiency of the same process and the quality of the resulting products. In particular, microwaves (Xu and Yang 2002; Kale and Bhat 2011; Öner at al. 2012) and high pressure steam (Ohshima at al. 2003a, 2003b) have demonstrated to be quite effective on the properties of final fabrics. In addition, some works have been carried out with the aim to develop a novel coloration technique that does not involve the use of electrolytes (Hashem 2006; Raslan at al. 2010). However, as it is known to the Authors, only sparse research (Yuen at al. 2010) has been conducted in order to investigate the opportunity to improve the current performances of steaming process and machines through process optimization by statistical methods.

3. Aims and objectives

Final product quality is one of the most important criteria employed to evaluate the Italian textile industry in the world. Competition is very strong in the global market and maintaining a high quality level requires a continuous update of manufacturing processes. Such level of innovation is not sustainable by small factories, which represent the majority of industrial operators in Italy.

In this context of extreme competition, color stiffness is clearly one of the key-aspects of the overall evaluation of the coloration process, being one of the most frequent reasons of complains about the product by the final customer. Its evaluation is normally given by visual inspection, but is evident that in this way is impossible to guarantee the same standards between the several producers which operate not only in the global market, but even in a small, local one. Obviously instrumental techniques exist, and have been translated into standard guidelines by International Organizations, but the normal industrial practices still make use of visual inspection.

For all this reasons, the first aim of this work is to purpose a standardized method to evaluate color stiffness. This method should be used to finally achieve a standardized level of quality of the final product. But the quality by itself, nowadays, does not make a competitive product, it is necessary in fact to reach a combination of high quality and low price. The final objective of this work is to demonstrate that a more rational use of well-established technologies can lead to a simultaneous increasing in both quality and economic competitiveness of the final products.

4. Experimental

4.1 Description of the process

In order to fix in a stable way pre-printed dyes into the chosen fabric, a steaming process is necessary to be performed as final step of production cycle. It's undeniable that, among all the stages that are required to ensure a good quality product, it's the most critical and an absolute attention is essential in this step.

The dyes and chemical reagents are mixed on a dry film that is applied on the surface of the fabric. In the case under investigation, which makes use of saturated steam at atmospheric pressure, the combination of the water condensed on the surface of the fabric and the heat of the controlled environment allows the penetration of the film into the core of the fibres where it is fixed. It is due to the physical and chemical reactions that are typical of each kind of fabric and colorant, but are not object of this work since they found their application in the printing stage of the production.

Now it's obvious that it is necessary to control several parameters in the process of color fixation and their optimal values will be different, as different are the fabrics and the specific kinds of colorants that they require. In general the parameters that have to be controlled are:

- Temperatures
- Steam Flow Rate
- Steam Pressure
- Steaming time

Since the temperature and pressure of the steam have been already optimized (142.9 °C and 4 bars) and the temperature inside the steaming chamber has to be in a range of 102-106 °C, the only parameters that can be manipulated to verify their influence in the final properties of the fabric are steam flow rate and steaming time. In figure 1 a simple representation of steaming process is given. As it is possible to notice the process works in continuous fabrics' feed whose rate has to be set in order to have the desired steaming time. In fact steaming time can be defined as the time spent by the fabric inside the steaming chamber, which means that is the time necessary to for the fabric to cover the distance between the entrance and exit of the steaming chamber. During this time the fibres undergo the action of steam, which arrives from a boiler and then is saturated and expanded in the steaming chamber until it reaches atmospheric pressure and, consequently, a temperature in the range previously stated.

An uniform distribution of steam and temperature is fundamental for the goodness of the process. In fact temperature gradients and excessive steam rate could lead to undesired condensed water on the surface of the fabric, which would result in the formation of water drops and the complete fail of the entire process, since it will ruin the pattern. A simple ad quick way to evaluate the amount of water that can be absorbed by fabrics available in the literature (Corbani 1990).

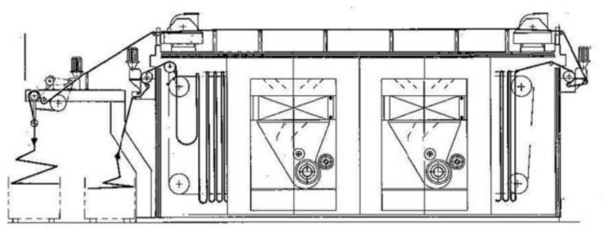

Figure 1: Steaming process – a simple representation of a steamer.

4.2 Equipment

All the experiments have been carried on a lab apparatus, which consisted of a lab-size steaming machine, called Labo2000. After the steaming process, from the resulting fabrics several samples (80 x 160 mm^2) were extracted and washed by using an Sdl Atlas Inc. Linitest Lab Dyeing System. An aqueous solution of a cationic detergent (Keopon CS) with a concentration of 2 g/l has been used as cleaning agent. The washing time and temperature were set at 10 min and 90 °C, respectively. After that, the resulting washing waters have been examined via spectrophotometric analysis by means of an Agilent Technologies Cary 5000 UV-Vis-Nir manual spectrophotometer in order to characterize color stiffness. A more detailed description of the experimental equipment is available elsewhere (Tronci 2017).

Figure 2: Pre-printed cotton fabric

4.3 Fabric

In this study, the efforts were focused on square 100% cotton fabrics (1000 mm x 1000 mm, 115 g/m^2), 90% pre-printed in black diamonds (Figure 2). The colorant is a commercial reactive based, as it is commonly suggested for this kind of fibres.

4.4 Design of experiments

A full factorial design, with 2 factors, or predictors, (steam flow rate SR [kg/h] and time spent by the fabric inside the steaming chamber t [min]) and 3 levels (low, medium and high) has been chosen in this experimental campaign. The factors' ranges were chosen between the ones that are commonly in use by the textile industrial operators. Three replications of the central point, for a total of 4 experiments characterized by the same parameters, were performed in order to evaluate the natural variation of the process. The total amount of experiments, in this way, was 12, which are reported in table 1 with the respective spectrophometric analysis results. It is important to point out that the execution order and the test number are different, in such a way that the importance of each factor in each experiment could not be affected by the time variable, which is implicitly included into the execution order. Each test is identified by a test ID, which allows to easily understand the parameters employed. In this case we have three fields, the first indicates the initial letter of the fabric type (Cotton), the second the steam rate and the time, the third the r number of replications of the central point.

Tab. 1 Experimental results.

Test	SR [kg/h]	t [min]	Absorbance [a.u.]	Test-ID
1	25	13	4.33	C25-13
2	55	13	4.44	C55-13
3	40	10	4.32	C40-10r1
4	40	13	4.40	C40-13
5	40	10	4.19	C40-10r2
6	40	10	4.19	C40-10r3
7	55	7	5.45	C55-7
8	25	7	5.85	C25-7
9	25	10	4.43	C25-10
10	55	10	4.18	C55-10
11	40	10	4.15	C40-10r4
12	40	7	5.01	C40-7

5. Results and discussion

In the present work a traditional color fixing process on proteic fibres fabric (Cotton) has been tested by means of Design of Experiments (DOE) (Montgomery 2001), in order to improve its efficiency and quality. The best subset regression analysis has been used to propose several regression models. To obtain the best fit of the data, non-linear polynomial models for the response, including second power of the predictor variables and their product (interaction of predictor variables) has been proposed. The best subset of independent variables (predictors) was chosen between contending subsets by using Mallows' C_p. The coefficient of determination, R^2, adjusted coefficient of determination, R^2(adj), and the standard error of the regression (the square root of the mean-square error or root-mean-square error), S, were adopted as a measure of the fitting quality of each model. The choice of these parameters can be explained quite easily. In fact, it is well-known that a model with large R^2 and small number of covariates could be a good choice by itself, since large R^2 implies the reliability of fitted values while, in general, a small number of predictors reduces the costs of obtaining information and, consequently, the costs of process monitoring. However, R^2 has an obvious weakness: it increases with the number of regressors added to the model, which often results in overfitting. On the contrary, C_p tends to be less dependent than R^2 on the number of regressors in the model, and hence, it tends to find the best subset that includes only the important predictors of the respective dependent variable (Hocking and Leslie 1967; Mallows 1973). The general procedure to find an adequate model by means of the C_p statistic is to calculate C_p for all possible combinations of variables and plot the C_p values against p (p is the number of regressors including the constant term) (Mallows 1991). The model with small C_p value and approximately equal to p is the most acceptable model. Another criterion for finding the best possible model is based on the standard error of the regression, S. According to this criterion, a model furnishing the lowest S and with the fewest predictors might be a sensible model. In this paper, all these statistics are synergically adopted for best subset evaluation.

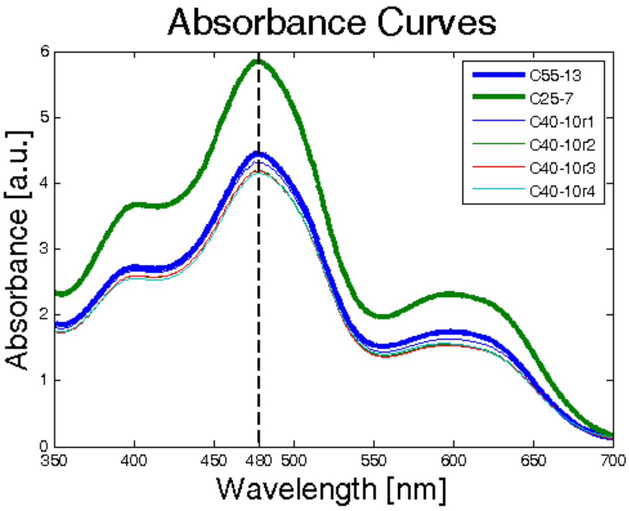

Figure 3: Absorbance curves

It has been chosen to show in figure 3 the absorbance curves of the samples obtained by the extreme steaming conditions, C25-7 and C55-13, which exhibit the lowest and the highest SR and t respectively, and the four central points of the experimental plan, C40-10 and its relative replications (thin lines).

In figure 4 a 3D representation of all measured peaks is shown in relation to the experimental plan, to have a general view of the process' behaviour. At first, is important to notice that the absorbance peaks were measured at a wavelength of 480 nm, which is the complementary, in the Ostwald circle, of the solution's colour obtained by washing the steamed samples. Now, it is well known that the absorbance's peak is proportional to the colour concentration in the solution so, the highest is the peak, the highest is the colour's concentration in the solution, which corresponds to the highest amount of colour released by the fabric after the washing procedure. In this way, a low absorbance value is equivalent to a good amount of colour fixed in the fabric.

Even from a quick observation, it is evident that there is an enormous difference between the peak measured for C25-7, the one for C55-13, while the ones corresponding to S40-10 and its replications exhibit a low natural, but anyway observable, variation of the process. At this point, the problem to determine which factor has the major influence in such a different behaviour of the absorbance curves arises. At a first sight, as C40-10’s curves are closer to S55-13’s one, as an inconsistent natural variation of the process can be observed and as the lowest absorbance’s peak was measured for C40-10r4 sample, it looks like the optimum value of t is located between 10 and 13 minutes, probably closer to the lowest time value (10 minutes).

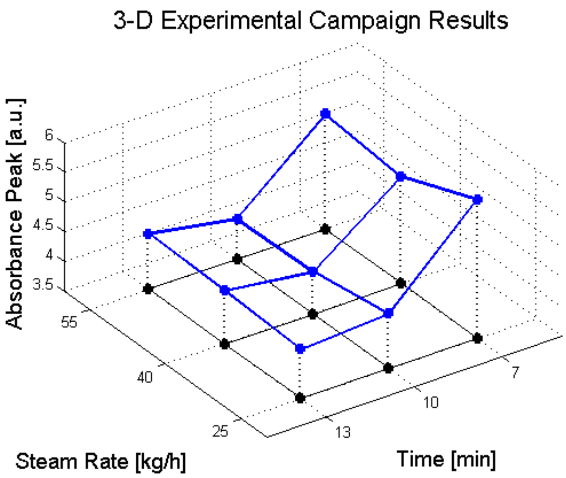

Figure 4: 3D representation of experimental results

In addition, is evident from figure 4 that the highest absorbance values are the ones corresponding to the 7 (C55-7), 8 (C25-7) and 12 (C40-7) tests, which are ones characterized by the lowest value of t (7 minutes). This could lead to the conclusion that 7 minutes could be not sufficient to guarantee a good color fixing. It is also evident as the influence of time decreases with the increasing of its value, being smoothed between 10 and 13 minutes, which confirms the previous hypothesis of the presence of the optimum in that range of exposure time. Another interesting behavior that can be observed is that, for each value of time employed in the experimental campaign (7,10 and 13 minutes) the influence of the steam rate appears to be negligible.

In order to confirm these hypotheses, all the data collected were analyzed via a statistical software tool called Minitab, to get a predictive model including the best subset of predictors that describes in the best way the relations between steam flow rate, time in the steaming chamber and the peak of color absorbance after spectrophotometric analysis. Because of the use of three levels for each factor in the experimental campaign, it is possible to appreciate until the second order of each one of them. So, the steam flow rate SR and its square SR^2, the time in the steaming chamber t and its square t^2 and their interaction, as product SRt, were adopted as variable predictors, in association to a constant one.

The best subset analysis gave nine different predictive models, whose characteristics are shown in table 2. In the first column is simply indicated an identification number for each model, in the second the number of variables which are present in it, in the third and the fourth its coefficients of determination, R^2 and R^2(adj), in the fifth and sixth its Mallows’ C_p and standard error of the regression, while the x in correspondence of the predictors' columns indicates only the presence of the respective predictor in the corresponding model. According to the criteria previously stated in the introduction, at first, the model number 9 and 7 have been chosen as the best, since they are characterized by the lowest C_p but, model 9 coefficients of determination are the greater ones (94.4 and 89.8 respectively) and its standard error S is the lowest above all the proposed models, which indicates a very good fitting properties if compared to the experimental data, as it is confirmed by its 3D representation shown in figure 4.

From the complete equation of model number 9 is possible to determine the optimal values of absorbance, steam rate and time, which are 4.08 a.u, 41.2 kg/h and 11.3 minutes respectively.

An analysis of the residuals, which are the difference between the calculated values of absorbance and the corresponding measured ones, enlightens once more the goodness of the chosen model. In figure 6 the adherence to the normal distribution is shown by the normal probability plot and the histogram, while the versus fits plot highlights the capability of the model to predict the lowest values of absorbance, which are the ones this study is interested to. Finally we can affirm that there is no evident influence of the execution order in the performance of the experimental campaign.

Moreover, the weaknesses of this model are related to high number of predictors that are present in it, which is contrast with the main objective of this work, and to the high precision in the data fitting of the points characterized by a exposure time of 7 minutes, which appears to be not so necessary, due to the experimental observations previously stated. In fact, the aim of this study is to get a reliable predictive model of the lowest values of absorbance peaks, which are located between 10 and 13 minutes.

Table 2: Best subsets analysis.

Model	Vars	R^2	R^2(adj)	C_p	S	PV	t	PV^2	t^2	PVt
1	1	48.2	43.0	47.2	0.420		X			
2	1	38.9	32.8	57.1	0.456				X	
3	2	87.7	85.0	7.1	0.215		X		X	
4	2	49.6	38.4	47.7	0.437	X	X			
5	3	89.2	85.1	7.5	0.215	X	X		X	
6	3	88.7	84.4	8.1	0.220		X	X	X	
7	4	92.5	88.2	6.0	0.191	X	X	X	X	
8	4	91.2	86.1	7.5	0.209	X	X		X	X
9	5	94.4	89.7	6.0	0.179	X	X	X	X	X

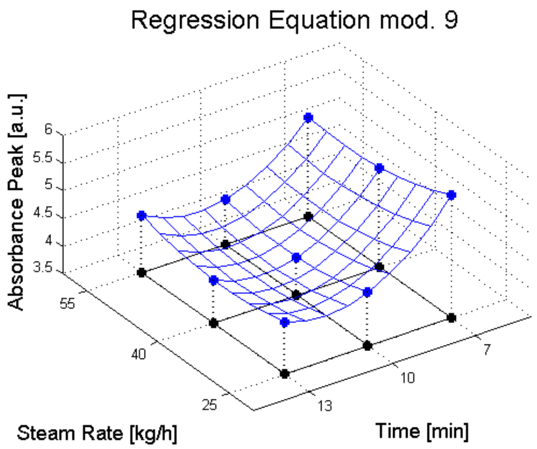

Figure 5: 3-D representation of model 9

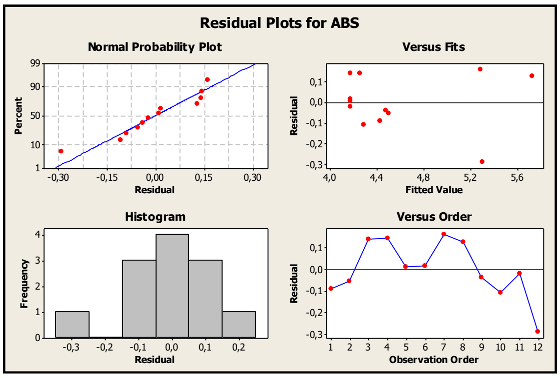

Figure 6: Analysis of the residuals of model 9.

For these reasons, at this pont, appears necessary to carry on a deeper analysis on each predictor to determine its real influence inside the model. The best way to fulfil this aim is to perform hypothesis tests by means of the calculation of the P-value of each regressor, as reported in table 3.

Table 3. Hypothesis test on model 9

Predictor	Coefficient	P-value
Constant	15.4	0.000
SR	-0.107	0.050
t	-1.62	0.001
SR^2	0.000913	0.110
t^2	0.0668	0.001
SRt	0.00282	0.206

Normally, it has been established that all the regressors whose P-value is over 0.05 have failed the hypothesis test, and their significance inside the proposed model is negligible. In this specific case, we can find two, SR^2 and SRt, which are over the desired significancy, and one, SR, which is just in the limit. These observations could lead to consider the steam flow rate not affective on the final performances of the dyeing fixing process but, to be completely sure of it, it is necessary to carry on by analysing another predictive model which does not contain SR^2 and SRt. In fact, the negligibility of the predictor SR is still not confirmed by this first analysis, even if the probability to incorrectly reject a true null hypothesis in the case of a p-value equal to the marginal value of 0.05 is typically 50% (Sellke et al. 2001).For these reasons, it has been decided to proceed with the analysis model number 5, characterized by the presence, as variables, of SR, t and t^2.. In this case the optimal values of absorbance, steam rate and time are, respectively, 4.12 a.u., 55 kg/h and 11.2 minutes. These results appear to be quite similar to the ones obtained from model 9, but the maximization of the optimum value of steam rate seems to be in contrast to what has been observed during the experimental campaign and could result in the formation of water drops on the surface of the fabric, which would lead to a quality decrease of the product. This fact and previous considerations about its marginal p-value, made the Authors doubt about the real effectiveness of the SR regressor and looks as if an additional hypothesis test needs to be performed on it (table 4).

Table 4. Hypothesis test on model 5

Predictor	Coefficient	P-value
Constant	13.7	0.000
SR	-0.00603	0.332
t	-1.66	0.000
t^2	0.0744	0.001

In this case, it has been found that the regressor SR has failed the hypothesis test, so its influence in the model is negligible. This fact confirms the initial hypothesis derived from the analysis of model number 9. On the contrary, the Constant, t and t^2 exhibit a strong influence so, it has been decided to perform a last analysis on model number 3, which contains only them in its equation. The results can be found in table 5.

Table 5. Hypothesis test on model 3

Predictor	Coefficient	P-value
Constant	13.4	0.000
t	-1.66	0.000
t^2	0.0745	0.001

In this circumstance, all the regressors show a high influence inside the predictive model, while their coefficients, being almost the same as the ones calculated for model number 5, confirm once more the complete negligibility of the steam flow rate in this process. Finally, we got a parabolic predictive model containing only t-dependent regressors, whose regression equation is:

$$Abs_3 = 13.4 - 1.66\,t + 0.0745\,t^2 \qquad (1)$$

From (1) is easy to determine the optimum values of absorbance and time which are, respectively, 4.15 a.u. and 11.1 minutes. These results point out how much the predictive model number 3, even with a low number of regressors, can be accurate. In fact, the calculated value of absorbance in correspondence of the optimum t value is exactly 4.15 a.u., the minimum one measured for the C40-10r4 test, while the optimum t value itself confirms the hypothesis pointed out from the observations on the absorbance curves in figure 2. In addition, the lack of influence of steam flow rate, allows to assume as correct the hypothesis to work at its low level of 25 kg/h, reaching at the same time a good performance and quality in the dyeing fixing process.

6. Energy Savings

In order to better understand the impact of these results on the economic performances of the steaming process is necessary to extend them to an industrial equipment. It has been chosen to evaluate the energy consumption of a small size steaming machine, i.e. the Vapo2015 produced by the same Arioli SpA, characterized by a mean required steam rate of 600 kg/h for cotton, the capacity to work on 200m of fabrics per cycle and a steaming time which ranges from 5 to 50 minutes. Technological constrains In this case, before steam production, the water is pre-heated at 60°C, due to the steam extraction from the steaming chamber. The calculations start from the well known equation of the power employed to produce steam:

$$Q_v = \dot{M}v \cdot \frac{r+(h_2-h_1)}{\eta_b \cdot \eta_d} \quad (2)$$

Where

- Q_v: power required for the steam production [kW];
- $\dot{M}v$: steam rate [kg/s];
- r: latent heat of water at 4 bars (2135.126 kJ/kg);
- h_2: specific enthalpy of water at 4 bars (T= 142.9°C; 601.57 kJ/kg);
- h_1: specific enthalpy of pre-heated water (T = 60°C ; 251.1 kJ/kg);
- η_b: efficiency of the boiler (0.85);
- η_d: efficiency of the steam distribution system.

The recommended steaming time (cycle) for cotton is usually 13 minutes (Corbani 1990), which is used in this study to determine the number of cycles per year. Assuming 250 working days and 16 hours per day, each year the machine will work for 18462 cycles. In this way we are setting our calculations to ensure the same level of production for both the traditional and the optimized process. Therefore the working time t_w will be $144 \cdot 10^5$ s and $123 \cdot 10^5$ s respectively. Assuming a steam consumption of 600 kg/h in both cases, we are able to calculate the primary energy necessary to maintain the same production level per year:

$$E_{tot} = Q_v \cdot t_w \quad (3)$$

and the consequent amount of consumed combustible (BTZ Oil, lower heating value LCV: 40166.4 kJ/kg):

$$M_{BTZ} = \frac{E_{tot}}{LCV} \quad (4)$$

The mean cost c of combustible in 2016 is given by the Italian association of combustibles producers (Unione Petrolifera) and is 0.333 €/kg, so the annual costs can be calculated as:

$$C_{tot} = M_{BTZ} \cdot c \quad (5)$$

The results of these calculations are resumed in table 6.

Table 6: Comparison between traditional and optimized process.

Predictor	t_w [s/year]	E_{tot} [kJ/year]	M_{BTZ} [kg]	c_{tot} [k€/year]
Traditional	$144 \cdot 10^5$	$77.8 \cdot 10^8$	$19.4 \cdot 10^4$	64.6
Optimized	$122 \cdot 10^5$	$66.6 \cdot 10^8$	$16.6 \cdot 10^4$	55.2

The decrease of the steaming time, respect to the traditional one, allows to achieve the aim of reducing the energy costs of 9.4 k€/year, but at this point the problem to evaluate the savings due to the consumption of steam rate arises. Considering the well known equation of power, it is possible to rewrite it introducing the difference of steam rate between traditional and optimized process, as it follows:

$$\Delta Q_v = \Delta \dot{M}v \cdot \frac{r+(h_2-h_1)}{\eta_b \cdot \eta_d} \quad (6)$$

Now it's unbearable to know how much it will be possible to decrease the steam rate in the industrial size equipment, but we can make some considerations about the potential savings proportionally to the reduction of steam consumption.

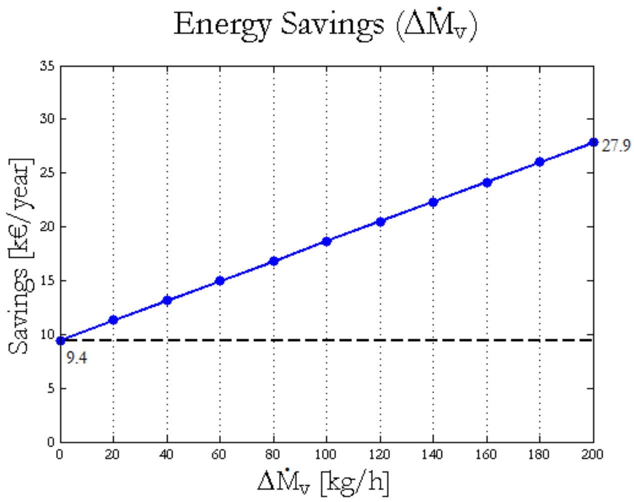

Figure 6: Energy savings as a function of steam rate variation.

In figure 6 is shown how hypothetically the optimized process could reduce the costs for primary energy up to 27.9 k€/year. This amount could appear quite significant by itself, but it reaches a greater importance if it's considered that it is associate to only a single, small-size machine. By increasing the number and the size of steamers, the savings increase proportionally and, since a normal industrial operator employs normally more than one steamer (3 or 4 in an average-size factory), the costs' reduction per year could be extremely relevant.

7. Conclusions

In this work, a traditional fixing color process of pre-printed cotton fabrics by steaming was studied through DOE technique. Two factors, the steam flow rate SR and the steaming time t in the steaming chamber, and three levels for each of them, have been chosen to perform a full factorial design plan of experiments. The squares of SR and t and their interaction, given by their product SRt have been added to the previous mentioned ones to complete the set of regressors to be used in the composition of the most adequate predictive model, in order to optimize the process. Absorbance peaks of released color in a washing solution of water and cleaning agent have been set as a measure of the quality of the process.

A critical approach to the best subset analysis, conducted by performing hypothesis tests on each predictor of the proposed models, has allowed to find the one containing only significant regressors and, consequently, to demonstrate the complete negligibility of the steam flow rate in the performance of the process. So, a simple parabolic regression equation dependent only on t, has been used to find the optimum steaming time of 11.1 min, corresponding to a minimum peak of absorbance of 4.15 a.u., which is exactly the one measured in the experimental campaign in correspondence of C40-10r4 test. These results confirm the adequacy of the chosen predictive model, even if it is characterized by a low amount of regressors and, consequently, the opportunity to operate the fixing cycles at the lowest value of steam rate and a lower value of steaming time.

The application of these results to an industrial steaming equipment has revealed to have the potential of reducing the energy costs per year up to 27.9 k€. This amount of money is subdivided into a part due to the decreasing of steaming time respect to the traditional employed one, evaluated into 9.4 k€, and a part due to the reduction of steam consumption per hour.

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References

Corbani, F. (1990). *La nobilitazione dei tessili*. Centro Tessile Cotoniero E Abbigliamento, Busto Arsizio, I.

Hashem, M. M. (2006). Development of a one-stage process for pretreatment and cationisation of cotton fabric. *Coloration Technology,* 122(3), 135–44.

Hocking, R. R. and Leslie R. N. (1967). Selection of the best subset in regression analysis. *Technometrics*, 9, 531–40.

Kale, M. J., and Bhat, N. V. (2011). Effect of microwave pretreatment on the dyeing behaviour of polyester fabric. *Coloration Technology*, 127 (6), 365–71.

Mallows, C. L. (1973). Some comments on Cp. *Technometrics*, 15, 661–75.

Mallows, C. L. (1991). More comments on Cp. *Technometrics*, 37, 362–72.

Montgomery, D. C. (2001). *Design and analysis of experiments*. John Wiley & Sons. New York, NY.

Ohshima, N., Itoh T., Shimohata I., Shigematsu M. and Tanahashi M. (2003a). Structural analysis for cellulose fibers treated by high pressure steam. *Sen'I Gakkaishi*. 59 (3), 83–86.Mallows, C. L. (1991).

Ohshima, N., Lee M., Jeong D. S., Hayashi A. and Wakida T. (2003b). Dyeing and mechanical properties of cellulosic fabrics processed by high pressure steaming. *Sen'I Gakkaishi*, 59 (2), 58–62.

Öner, E., Büyükakinci Y. and Sökmen N. (2012). Microwave-assisted dyeing of poly (butylene terephthalate) fabric with disperse dyes. *Coloration Technology*, 129, 125–30..

Raslan, W. M., Bendak A., Khalil E. M. and Fawzi T. (2010). Modification of polyester fabric by chemical/thermal treatment to improve dyeing ability. *Coloration Technology*, 126, 231–36.

Sellke, T., M. Bayarri J. A. and Berger J. O. (2001). Calibration of P-values for testing precise null hypotheses. *The American Statistician*, 55 (1), 62–71.

Tronci, A. (2017). Optimization of Dyeing Fixing Process on Silk Fabrics through DOE Analysis. *Journal of Natural Fibers*, Article in press, DOI: 10.1080/15440478.2017.1279099 .

Xu, W. and Yang C. (2002). Hydrolysis and dyeing of polyester Fabric using microwave irradiation. *Coloration Technology*, 118, 211–14.

Yuen, C. W. M., Kan C. W., Jiang S. Q., Ku K. A., Choi P. S. R. and Wong K. Y. 2010. Optimum condition of ink-jet printing in wool fabric. Fybers and Polymers, 11 (2):229–33.