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## Study On The Optimization Of The Textile Coloristic Performance Of The Bleaching Process Using Pad-Steam

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### Abstract

Information concerning the critical variables of the bleaching process (transformation of a raw fabric into bleached) by pad-steam is scarce, since it is considered a recent process in the textile industry. One hundred percent fine cotton fabrics present great difficulties in the standardization of bleaching across different production batches, which is even more complex when the fabric composition provided by the supplier is unknown. Thus, one carried out an evaluation of the variables that influence color yield in the bleaching process by pad-steam. The conditions used consisted of the reintroduction of the process of desizing by pad-batch, as well as washing and bleaching by pad-steam. The other variable changed was the chemical recipes (desizing and bleaching). As result of this research, the variables which most influenced color performance were the degree of whiteness (Berger) and the pH of extraction. Hence, a change in the production process for this type of 100% fine cotton is required. In this work, a statistical control was performed on the characteristics of the product obtained, which compared results before and during the study. The suggestions for improvement, some of which have already been implemented, are also presented. The results were then compared, enabling one to observe a significant improvement.

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*Keywords:* Bleaching; Pad-steam; Color yield; Cotton; Woven fabric.

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### 1. Introduction

Cotton fibers can be classified according to their different sizes, namely 2 cm to 2.5 cm, 3 cm to 3.5 cm and 4 cm to 4.5 cm [1]. When these are cut transversally, they may be bean or kidney-shaped (as can be seen in Fig. 1. A).

Their structure is composed of an outer layer - the cuticle - which is followed by a primary wall, a transition layer, and a secondary wall, with the lumen located in the core (see Fig. 1. B) [1, 2, 3].

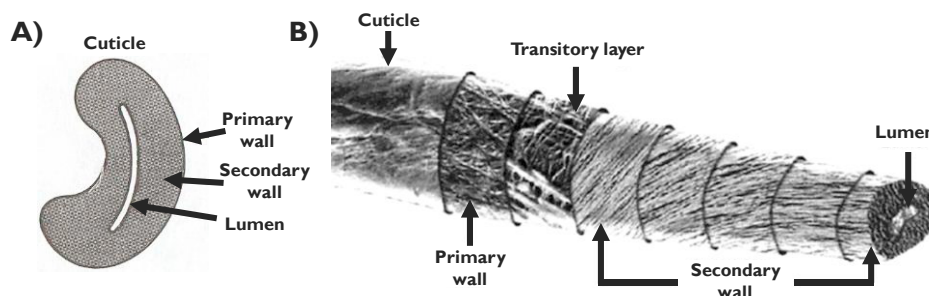


Fig. 1. A) Cross-section of a cotton fibre, in the form of a bean or kidney. From the exterior to the interior, one observes the outer part or cuticle, which is followed by the primary wall, the secondary wall, and the lumen located in the core. B) The structure of a cotton fibre, from the exterior to the interior: the cuticle, the primary wall, the transitional layer, the secondary wall, and the lumen [2, 4, 5].

Cotton is predominantly composed of cellulose, a natural fiber [4 - 9]. As can be observed in the study undertaken by Mather and Wardman [5], approximately 90% of cotton is composed of cellulose, while the remaining 10% consists of impurities introduced during the textile process [1, 4, 5, 10]. Table 1 presents the manner in which they are grouped. In order to create a fabric, the fibre is subjected to various industrial processes: first, the fibers are carded [4], then spun [11], and finally joined together by the weaving process, thus creating the woven fabric. During the spinning and weaving processes, sizing agents such as starch, polyvinyl alcohol [12, 13] and cellulose carboxymethyl [12, 14, 15, 16], are introduced to ensure the stability, resistance and manageability of the fibers [11].

Table 1. Characterization of the chemical composition of cotton [1, 5, 10].

Cotton composition	Types of substances	Percentage by weight (%)
Cellulose		88.0 – 96.0
Impurities		4.0 – 12.0
Waxes	Fatty acids, wax alcohol, esters, glycerine and hydrocarbons.	0.4 – 1.2
Ashes	Carbonates, sulphates, phosphates and oxides of: K, Mg, Ca, Fe, Na, Al.	0.7 – 1.2
Pectin	Salts and acids: Mg, Ca and Fe.	0.4 – 1.2
Proteins	Polyamide salts from dead plants.	1.0 – 1.9
Others	Organic acids, lignin, pigments, hemicellulose and sugars.	0.5 – 8.0

In order to transform the yellow color of raw material into white, the fabric must be subjected to a preparation process. The first step is known as desizing, namely the process of removing sizing agents, which is carried out by using enzymes (amylases for starch) [17] and chemical products. Afterwards, the fabric must be bleached removing impurities, thus uniformising the entire fabric and making it white [18, 19, 20].

The bleaching process can be undertaken through exhaustion, where the article is impregnated in a chemical bath consisting of hydrogen peroxide ( $H_2O_2$ ), caustic soda (NaOH) [21], a hydrogen peroxide stabilizing agent [22], a metal scavenging agent and wax removers, at a temperature close to that of boiling, and with a pH stabilizer. As this solution has an alkaline pH, citric acid [23] is used to lower the pH, thus neutralizing the pH within the fibers. If the pH is higher or lower than these neutral pH values (close to 7.0), this may lead to dyeing or staining problems, since dyes may react differently with each article. The oxidative bleaching of hydrogen peroxide requires an alkaline activator (NaOH) for the reaction to take place, and oxygen is released to oxidize the natural color of the raw tissue [24].

Bleaching can be carried out through various processes: by jet (discontinuous), pad-batch (semi-continuous) [25] or pad-steam (continuous). The industry increasingly prefers to work with continuous processes such as pad-steam, since the steam potentiates the bleaching effect and requires lower production times to do so, in comparison to the traditional pad-batch or jet processes. The products listed in table 2 are essential when undertaking traditional

bleaching [21 - 23]. This process was carried out for 40 – 60 minutes, at 98°C (Celsius) in a bath ratio of 1:10, followed by cooling and washing with a neutralized pH (with acid and enzyme catalysis).

Table 2. Chemical recipe for traditional oxidative bleaching [22, 26, 27].

Concentration (ml/l)	Product
1.0 – 2.0	Wetting agent.
1.0 – 2.5	Alkali agent (caustic soda or sodium carbonate).
0.5 – 1.5	Peroxide stabilizer agent.
2.0 – 10.0	Hydrogen peroxide.

The consistency of results in the same kinds of woven fabric was difficult to achieve. This was due to the amount of sizing agents, or the definition of the sizing agents, to be added to the weaving process, which were not described by the supplier. An additional problem was keeping the results constant in similar fabrics over time, as well as in the same fabric batch. Thus, the aim of the present study was to identify and measure the critical variables in the bleaching process by pad-steam. This study was undertaken at *Estamparia Adalberto Pinto da Silva, S.A.*, which is a textile industry dedicated to fashion and home fabrics. The article is divided into five sections: the first of these consists of the introduction, where one describes the aim of the work and presents the literature review; the second provides a description of the methodology used; the third defines the practical parameters, more specifically, the chemical and mechanical; the fourth presents the results and discussion; and, lastly, the fifth deals with the final conclusions.

## 2. Methodology

One hundred percent woven cotton fabric was adopted for this study, which is predominantly used in fashion manufacture. The laboratory trials of the chemical recipes and the temperature gradient were performed in a cooking machine - Bimby, model TM 31 - before testing in production. The variables measured to evaluate the bleaching effect are presented in Fig. 2.

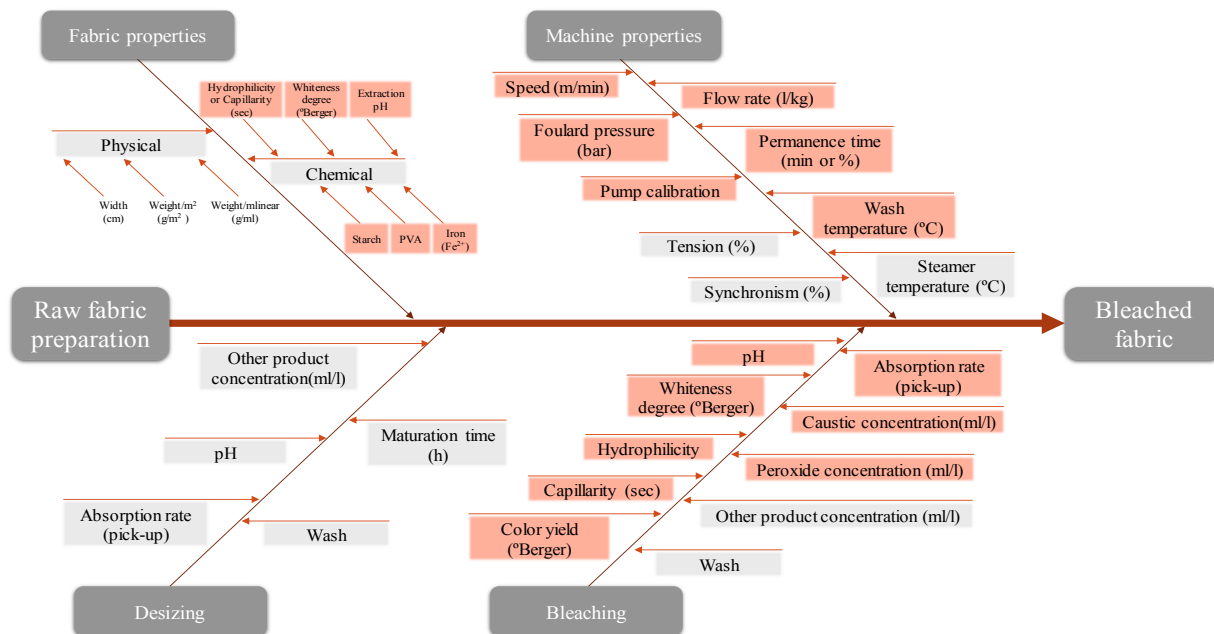


Fig. 2. Ishikawa diagram for the different variables identified in each bleaching phase. Highlighted in red are those which constitute the potential causes of variability in the bleaching process and affect color yield.

The forms of measuring the different parameters of the woven fabric, enumerated in Fig. 2, are listed below [28]:

- Starch concentration after desizing: place a drop of 0.1% iodine solution on the fabric and compare the reaction with the violet TEGEWA scale;
- Whiteness degree (Berger scale) allows one to read the fabric samples by using the spectrophotometer colorimeter Datacolor SF 600 Plus, in the wavelength between 360 – 700 nm (nanometers);
- Extraction of fabric pH, with the Morapex® A machine (5 milliliters of extraction solution and 5 drops of pH Merck indicator); when this is compared to the Merck pH scale, the pH value must be between 6.5 and 7, after bleaching;
- Detection of the presence of iron (associated with pinholes), using the reaction of four drops of hydrochloric acid with 0.05 grams of ammonium thiocyanate, on tissue previously wet with distilled water; if there is iron, the reaction turns red in color;
- Evaluation of fabric absorption through hydrophilicity with a 0.2% blue patent solution V (depositing a drop of solution from an approximate height of 4 centimeters, on the fabric) and evaluating the shape that the drop acquires. If this is round and about 4 centimeters in diameter, it is considered appropriate; if it acquires an irregular shape, it is not. In the case of capillarity, the method consists of counting the time required (approximately 120 seconds) for the fabric sample to absorb 1 cm of the previous solution;
- Color yield through whiteness degree (Berger scale) of the navy (424) printed bar. The reading is carried out by means of the spectrophotometer colorimeter Xrite, in the wavelength between 360 - 700 nm.

### 3. Practical parameters

To better understand how this work was performed, one characterized the different parameters considered relevant in order to explain the results presented.

#### 3.1. Chemical products

A production method was developed in an attempt to solve these problems. Initially, the woven fabrics were treated by desizing and bleaching in the same pad-steam machine. However, one observed that the enzyme used in desizing did not have enough time to act. Thus, the maturation time of desizing was improved, so that desizing was carried out by pad-batch, followed by washing and bleaching operations on pad-steam. The bleaching results were thus improved. After changing the preparation of the 100% woven cotton fabric manufacturing process, two different chemical recipes were tested. These were provided by different suppliers and can be observed in Table 3.

Table 3. Characterization of the different products used in the tests under study, according to name, supplier and specifications.

Product name	Supplier	Specifications
Avco-Blanc HB-LF	AVCO Chemicals	Wetting agent
Avco-Stabilizer HSF	AVCO Chemicals	Peroxide stabilizer without silicates
Avcotex CAN	AVCO Chemicals	Wetting and de-aerating agent
Avco-Tryl FCE	AVCO Chemicals	Complex and sequestering agent
Avcozim POR	AVCO Chemicals	$\alpha$ -amilase
Beisol T2090	CHT Group	Amilase
Beixon Q	CHT Group	Sequestering and dispersing agent
Bioluze MAX GC-NF	AVCO Chemicals	Emulsifying agent
Caustic soda 48° Bé*	Quimitécnica	Caustic soda (alkali agent)
Cerriostar RN	Farbotex Fast Solutions, SpA	Peroxide stabilizer and metal sequestering agent
Citric acid 35%	Quimitécnica	Alkaline bath neutralizing agent
Contavan CIL	CHT Group	Peroxide stabilizer
Felosan FOX	CHT Group	Detergent
Hydrogen peroxide 50%	Ângelo Coimbra & CA, S.A.	Hydrogen peroxide
Neutraacid NCS	CHT Group	Alkaline bath neutralizing agent
Poliquet 1096E	AVCO Chemicals	Sequestering agent
Subitol	AVCO Chemicals	Wetting agent

\* Bé, Baumé

With the information listed in Table 3, two different products from diverse suppliers were tested after the manufacturing process has been changed: from desizing and bleaching by pad-steam to desizing by pad-batch (Brückner), and washing and bleaching by pad-steam (Erbatech machine). The recipes used are presented in Table 4.

Table 4. Chemical recipes used before and in the two trials performed, for each manufacturing process.

Product name	Prior concentration	Manufacturing process	Concentration in trial 1	Concentration in trial 2	Manufacturing process
Felosan FOX	2 ml/l*		4 ml/l*	-	
Beisol T2090	4 ml/l*		5 ml/l*	-	
Avcozim POR	-		-	4 g/l	
Beixon Q	3 ml/l*		3 ml/l*	-	Desizing by pad-batch.
Subitol	-		1.5 ml/l*	-	
Avcotex CAN	-		-	2 g/l	
Poliquest 1096E	-		-	5 g/l	
Bioluze MAX GC-NF	-	Desizing and bleaching by pad-steam	-	5 g/l	
Bioluze MAX GC-NF	-		-	2.5 ml/l*	
Avcoblanc HB-LF	-		-	15 ml/l*	
Avco-Stabilizer HSF	-		-	15 ml/l*	
Avcotryl FCE	-		-	5 ml/l*	Washing and bleaching by pad-steam
Felosan FOX	-		1 ml/l*	-	
Contavan CIL	15 ml/l*		30 ml/l*	-	
Cerriostar RN	25 ml/l*		-	-	
H <sub>2</sub> O <sub>2</sub> 50%	140 ml/l*		95 ml/l*	130 ml/l*	
NaOH 48°Bé**	40 ml/l*		30 ml/l*	40 ml/l*	

\* ml/l, millilitre/litre

\*\* Bé, Baumé

### 3.2. Machine parameters

Desizing was performed by means of a Brückner pad-batch machine. One used a speed of 100 meters/minute, a bath temperature of 80°C, and desizing maturation was carried out for 6 hours at room temperature (RT). Washing and bleaching were performed on an Erbatech pad-steam machine, using a speed of 50 meters/minute. The strain was 50%, synchronism was 5%, and pressure in the NIP was 2.3 bar. Additional conditions are listed in Table 5, in which the washing compartments are VG1, VG2 (jbox compartment) and VTR1, VTR2, VTR3, VTR4 and VTR5 (washing drum compartments). The NIP was added to the bleaching bath zone.

Table 5. Characterization of the washing and bleaching process by pad-steam machine (Erbatech), common to the performed trials 1 and 2.

Control Parameters	VG1	VTR1	VTR2	NIP	Steamer	VG2	VTR3	VTR4	VTR5
Flow rate (l/kg)	6.0	-	-	-	-	-	5.0		4.0
Temperatures (°C)	80	80	60	RT	102	90	80	70	60
Remaining time (min)	3	-	-	-	10	3	-	-	-
Foulard pressure (bar)	3.5	3.5	5.5	2.3	3.5	3.5	3.3	3.5	5 - 6

#### 4. Results and discussion

The initial production plan for the woven fabrics did not allow the starch to be completely removed at the end of the bleaching process. Hence, the process for enzymatic desizing by pad-batch, washing and bleaching by pad-steam had to be changed. In this way, one ensured that the enzyme (amylase) would have more time to act on the degradation of the starch and thus, after a maturation time of 6 hours, the woven fabric was washed in the pad-steam machine, guaranteeing that most of the starch and washes were retained in the wash water, and the remainder removed through the bleach constituents.

The optimal activity of the enzyme can be influenced both by the pH as well as the temperature of the bath where it is applied. If these two parameters are not controlled adequately, the starch degradation activity is affected [13, 29]. This could constitute a disadvantage in the application of dyestuff by means of dyeing or stamping, since the absorption capacity might be different if there are still traces of sizing agents in the fibers.

The use of amylases in pre-treatment may increase the whiteness degree by about 6%, according to Ali et al., [20]. It is therefore important to control the variables which directly affect the enzymatic activity of the amylase.

The implementation of silver nanoparticles in cotton fabrics, to improve the impregnation of bacteria within the cotton fibers, could enhance the activity of the enzymes, thus enhancing the removal of sticks from the starch family [30].

The optimization of the method to evaluate the absorbability of the woven fabric was also altered. Through the expedient method of placing a drop of a blue patent solution V onto the fabric, and verifying whether the shape created was adequate or not, constituted a rather subjective aspect of process validation. The change implemented by this work was to use the same solution, and measure the time (in seconds) required for 1 centimeter of the solution to rise in the fabric, using the capillary test [31].

Prior to changing the production schedule of 100% woven cotton fabrics, another test was carried out to validate the mechanical parameters of the pad-steam machine, as well as to optimize the bleaching process. In this type of machine, the NIP is used for the impregnation of the bleaching bath. By using a container in which the bath may have to be heated to a temperature near boiling point, one is thus able to reduce the amount of bath required (chemicals and water) and minimize wasted energy [32]. The first validation was to evaluate the dispersion of the NIP foulard and check if the fabric was being evenly squeezed. Thus, observation was carried out by means of the talcum powder test, in which talcum powder is applied to the foulards. These were then squeezed and submitted to a certain amount of pressure, with a subsequent validation of whether the powder thickness was uniform across the length of the foulard. From the various pressures analyzed, 2.3 bar was the one that obtained better uniform dispersion values along the pad width.

In this study one also evaluated whether the pick-up used to calculate the delta pick-up, and which is obtained instantly by the machine's software, was correct. In order to do so, one verified if the pick-up was affected by speed and, consecutively, if speed influenced whiteness degree, color yield, pH and starch at the end of the bleaching process.

The validation of the uniformity of the bleached weft (width) led to a reduction in the collection of samples to validate the bleaching process. These were initially collected at the beginning, middle and end of the production batch, and evaluated at the left, middle, and right sides. In this way, one was able to perceive what was happening on the machine and detect any disruption. Subsequently, one only collected center samples at the beginning and end of production, which are representative of full fabric width. The velocity was also changed, from 25 meters/minute to 50 meters/minute because, in terms of color yield, one observed that speed did not influence whiteness degree, color yield or pH.

In brief, Table 6 summarizes the data of the trials carried out and suggested by chemical suppliers, where: 1, the data were analyzed before changing the production method; 2, Farbotex's chemical recipe test; 3, CHT's chemical trial; and, 4, Quiterma's chemical recipe trial. The different tests of the critical variables in the process led to the implementation of a new process of bleaching 100% cotton fabrics by desizing in pad-batch and bleaching in pad-steam.

Table 6. Summary of test data. The means ( $\bar{x}$ ) and standard deviation ( $\sigma$ ) of the variables: whiteness degree, color yield degree, pH, hydrophilicity or capillarity and starch.

Trials	Type of fabric	Whiteness degree (°Berger)		Color yield (°Berger)		pH		Hydrophilicity or capillarity	Starch	
		$\bar{x}$	$\sigma$	$\bar{x}$	$\sigma$	$\bar{x}$	$\sigma$		$\bar{x}$	$\sigma$
1	642	81.5	2.7	14.0	1.7	4.1	0.6	Good	8.0	1.0
2		81.6	n/a*	13.5	n/a*	6.5	n/a*	Good	9.0	n/a
3		78.8	n/a*	14.7	n/a*	6.5	n/a*	Good	9.0	n/a
4		72.8	1.0	12.2	0.8	6.2	0.0	Good / 38.0 sec	9.0	0.0
1	901	81.5	2.8	13.9	1.6	4.1	0.6	Good	8.0	0.8
2		84.0	n/a*	13.4	n/a	6.5	n/a*	Good	Not evaluated	
3		81.6	n/a*	15.1	n/a	7.0	n/a*	Good	Not evaluated	
1	906	81.5	2.8	14.0	1.6	4.1	0.6	Good	7.9	0.8
2		81.6	n/a*	15.1	n/a*	6.5	n/a*	Good	Not evaluated	
3		77.5	n/a*	15.4	n/a*	5.5	n/a*	Good	Not evaluated	
4		73.0	1.6	14.5	0.9	8.5	0.0	Good / 24.6 sec	Not evaluated	
1	1070	79.4	2.7	14.5	1.8	4.3	0.8	Good	8.2	0.7
2		78.2	n/a*	12.9	n/a*	6.5	n/a*	Good	Not evaluated	
3		74.9	n/a*	13.6	n/a*	10.0	n/a*	Good	Not evaluated	
1	1538	82.1	2.5	12.0	n/a*	3.7	0.6	Good	8.5	0.7
2		78.8	n/a*	13.7	n/a*	6.5	n/a*	Good	Not evaluated	
3		72.2	n/a*	13.9	n/a*	5.8	n/a*	Good	Not evaluated	
4		71.4	6.2	10.6	0.4	6.2	0.6	Good / 39.9 sec	9.0	0.0

\* n/a, not applicable

## 5. Conclusions

The bleaching of 100% woven cotton fabrics was initially carried out in a single pad-steam process, which did not allow for the efficient washing of the fabrics. This was due to the fact that the time required for the starch-degrading enzymes to act and degrade was insufficient and did not facilitate impregnation in the bleaching bath.

The aspects improved by this study were: a weekly calibration of the chemical dosing pumps, and the change of the NIP foulards pressure to 2.3 bar. A problem which must still be addressed is how to measure, in real time, the concentration of products in the bath impregnated in NIP. It will be rather difficult to find a solution since caustic soda and hydrogen peroxide react simultaneously. The changes carried out in the two-stage bleaching process, pad-batch desizing and washing and bleaching by pad-steam, for 100% woven cotton fabrics, ensured whiteness degree and color yield values in the same types of fabric. However, it should be pointed out that the lack of autonomy in the isolation of variables in each critical process did not facilitate the study.

The implementation of the new production roadmap for 100% cotton fabrics has increased the productive capacity of the pad-steam machine in the case of bleached knitted fabrics. This results from the fact that while fabrics are subjected to the maturing process, other orders can be produced.

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