TECHNICAL UNIVERSITY OF NORTH

FACULTY OF APPLIED SCIENCE ENGINEERING

TECHNICAL REPORT

PHASE OPTIMIZATION SOAPING IN THE DYEING OF COTTON 100% WITH REACTIVE DYES BY EVALUATING AND SELECTING A FORMULA TECHNICALLY DEVELOPED

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PHASE OPTIMIZATION SOAPING IN THE DYEING OF COTTON 100% WITH REACTIVE DYES BY EVALUATING AND SELECTING A FORMULA TECHNICALLY DEVELOPED

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1. INTRODUCTION

The use of colourings reagents for the cotton dye 100 % at present is most used by the characteristics of solidity that acquire the textiles after the process of dye since the colouring reacts covalentemente with the fiber. The process of dye with colourings reagents develops in three phases:

1. Pretratamiento (Descrude and Semiwhite)
2. Dye
3. Elimination of the colouring hidrolizado

In the latter phase the colouring hidrolizado always exists in major or minor proportion, the above mentioned colouring hidrolizado is in two different situations. - Hidrolizado in the water - Hidrolizado in the fiber In the phase of elimination of the colouring hidrolizado languishes about 70 % of the water spent in a process of dye..

2. OBJETIVOS

2.1. OBJECTIVE GENERAL

Optimize the phase of soaping in 100% cotton with dyes dyeing reagents through of the evaluation and selection of a formula technically developed

2.2. EPESIFIC

- Determine objectives (time, temperature, pH, liquor ratio) variables that are involved in the realization of the process in the phase of tincture as the post-production phase is carried out in order to eliminate the dyestuff hydrolysate and reach the fastness to washing required in textile

- Describing systems that compose the team of Overflow dye used in the process

- Assess the characteristics and properties of the products that will be used in soaping and agreement to their technical sheets perform the respective tests to determine product and process more efficient.

- Analyze costs of products used in order to determine their ideal quantities to thus reduce production costs - establish the ideal formula and the process to eliminate the dyestuff hydrolysate.

3. ALCANCE

The work focuses in describing generalities of the cotton fiber, the colourings reagents used in the dye of cotton, the varieties of products used in the soaping, the machinery used for this process and in the practical part he analyzes in specific form from the neutralized one of the bath of dye with colourings reagents, the soaping and the later rinsings, with the purpose of obtaining improvements in time, water saving and energy, and optimization of products and used processes, obtaining at the end of this work an analysis of costs of different recipes and realized processes.

4. JUSTIFICACION

Today is relevant the importance of conserving natural resources and water being an essential element in the dye we must identify potential phases where this resource saving is significant and this leads us to determine that the process after the dye, soaping and rinsing phase where occurs the greater consumption of water since here it consumes up to 70% of the total water expended in the process of dyeing with dyes reagents.In order to optimize this phase is essential to understand the principle of how it works the process that makes it possible to eliminate the dyestuff hydrolysate, and through the use of appropriate products developed to fulfil this function contribute to the optimization of the process, thus reducing the time required to complete this phase, the cost of energy and the more important to reduce water

5. PARTE EXPERIMENTAL

5.1. MAQUINARIA EMPLEADA

5.1.1. AHIBA IR DATACOLOR

It is a machine that allows to realize dyes for depletion to laborator level, which combines infrared of heating and refrigeration of air forced with an electronic control.

5.1.2. OVERFLOW BRAZZOLI

To verify the results obtained in laboratory there realizes tests to industrial level in a machine Overflow de Brazzoli that allows to realize the dye for depletion. The relation of weight between weight of fiber and weight of solution of colouring is from 1/5 to 1/12

5.2. COLORANTES REACTIVOS

The colourings used in the dye are Corazol, Corafix and Coralite which are a range of vinilsulfónicos elaborated by COLOURTEX, proceeding from the India. Corazol and Corafix are colourings that allow the obtaining of average and dark colors, the Coralite use for low shades or colors of difficult reproducibilidad The process of dye with colourings reagents develops under the following mechanism:

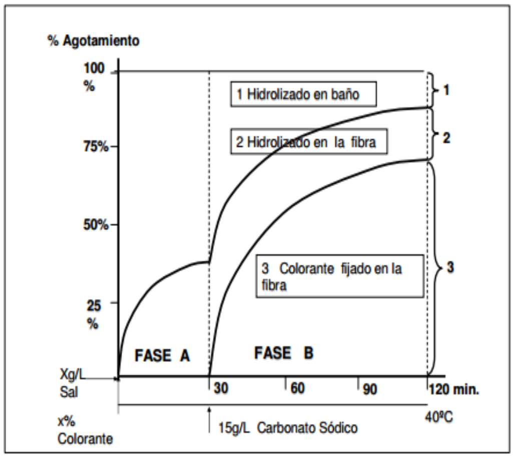


Fig. # 1 Mechanism of Dye figures * with colourings Reagents

F PHASE A. ABSORPTION in pH neutral and with salt, to exhaust the colouring

PHASE B. REACTION adds the alkali in order that the colouring should react with the fiber

PHASE C. ELIMINATION OF THE COLOURING HIDROLIZADO

1. Colouring hidrolizado in the bath. Drena on having emptied the machine after the dye.

2. Colouring hidrolizado in the fiber. It is extracted by rinsings later to the dye.

3. Colouring concentrated on the fiber

5.2.1. PROFILE TINTOREO

It allows to know the behavior that will have the colouring in the process of elimination of the colouring hidrolizadoermite

5.3. TINTURA'S PROCESS

COLORANTE	%	
	AGOTAMIENTO	FIJACION
YELLOW CORAZOL 3GL	90	63
TURQUISE CORAZOL 2GP	89	54
NAVY CORAZOL RFT	89	78
BLUE CORAZOL BB 133	80	60
BLUE CORAZOL R 160	88	72
BLACK CORAZOL B 150	90	79
CORAFIX JET BLACK GDR	83	78
YELLOW CORALITE 2R	92	80
BLUE CORALITE N	92	88
ROJO CORALITE B	88	80
BLUE CORALITE G	92	80

Tabla # 1 Profile Tintoreo Color Colourtex

5.3.1. DESCRUDE Y SEMIBLANCO

This phase of the dye is the first one in realize, allows to eliminate the impurities natural and acquired of the fibers and fabrics, as wax, pectin and alcohols, material of stuck, the dirt and the oil. Simultaneously with this process and by means of the use of Peroxide of Hydrogen (H2O2) there is realized the chemical whitening which does that the fibers see whiter and are more absorbent in the following stages. The correct accomplishment of this process guarantees the success of the dye in 50 %.

Absorbency	5 seconds
pH	6.5 – 7.0
Total Alkalinity	5%
Hardness	< 70 ppm $CaCO_3$ ó < 4^0dh Hardness German degrees

Table # 2 Parameters of control in the Descrude and Semiwhite

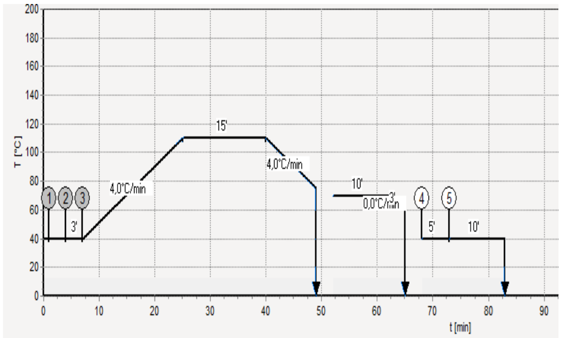


Fig # 2 Curve of the procesos of descrude and semiwhite

APPLIED RECIPE:

PRODUCT	g/l
Antiquiebre (TEBOLAN B-UF)	**2**
Secuestrante (DISPROSEK KG)	**1**
Detergente – estabilizador (PERESTABIL 3E)	**1**
Emulsionante (EMULSID S-OL)	**1**
Humectante (INVADINA LUN)	**1**
Sosa caustica en perlas	**2**
Agua oxigenada 50%	**3**
NEUTRALIZADO	
Acido (ACIDO CITRICO)	**0.7**
Katalaza (KILLETOX TX)	**0.15**

Tabla # 3 Receta de Descrude y Semiblanco

5.3.2 DYE

The phase of dye is realized applying a process of depletion I agree to the following curve.

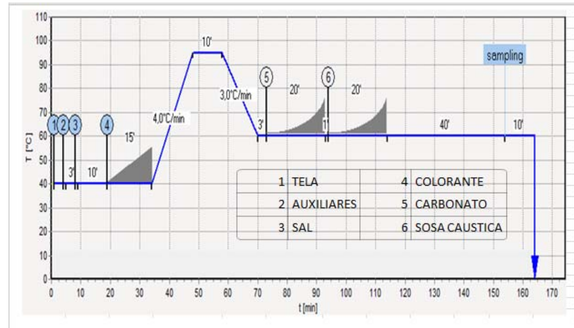


Fig # 3 Curve Cotton dyeing process with dyes Corazol strong tones - Corafix

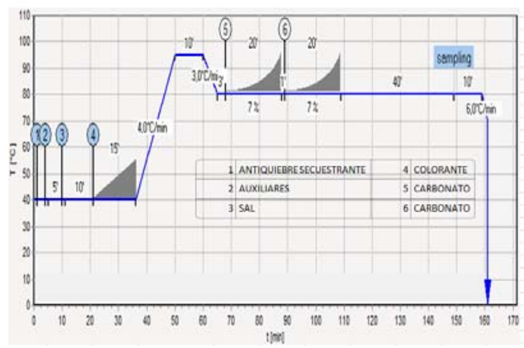


Figura # 4 Curva del proceso de Tintura de Algodón tonos fuertes Turquezas Corazol - Corafix

Control parameters:

TINTURA CO					
DUREZA TINTURA (ppm)	PH INICIAL	DENSIDAD TEORICA	DENSIDAD REAL	PH Na_2CO_3	PH Na(OH)
5	6 - 6.5	DENSIDAD		9,9-10,6	10,8-11,2

Table # 4 Control parameters in the dyeing

5.3.3 REMOVING DYE HYDROLYSATE

Achieves the required strength in the textile

PREVIOUS PROCESS - BASE COLORS<1

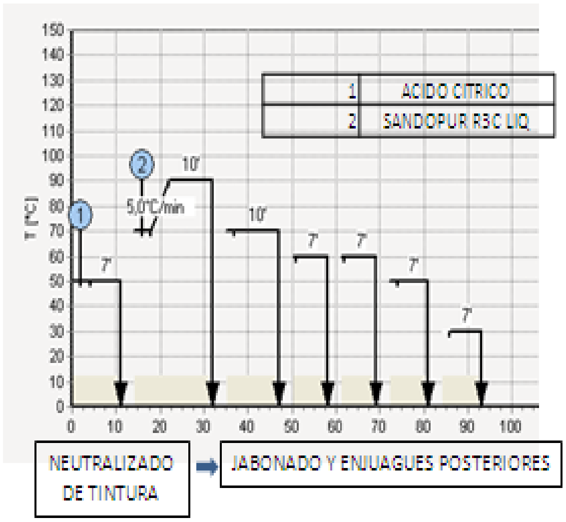


Fig # 5 Dye removal process hydrolyzate (above) base colors

PREVIOUS PROCESS - STRONG COLORS>1.5%

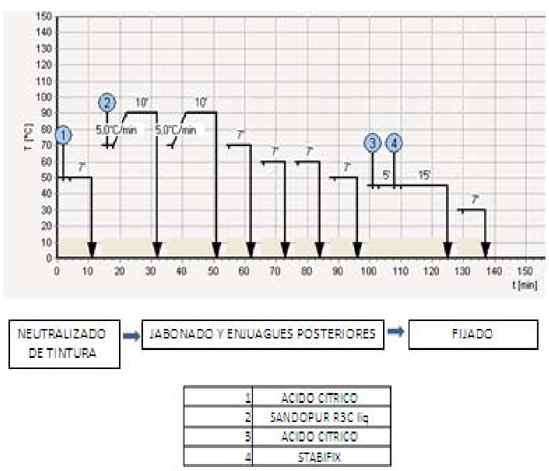


Fig # 6 Dye removal process hydrolyzate (previous) Strong colors

6. VARIABLES THAT INTERFERE IN THE DYEING PROCESS WITH REACTIVE DYES.

To proceed with the analysis and optimization of phase soaping is important to know the variables in the dyeing phase must be controlled to prevent the increase of dye hydrolyzate

6.1 PRETREATMENT

Conditioning the textile to ensure the success of a good dye in performance dye shade matching and obtaining required**.**

6.2 ELECTROLYTE

Electrolyte is used as Sodium Chloride (NaCl), which neutralises addition electronegative potential of the fiber, causes the water to expel the dye to the substrate

The amounts of salt to be used are a function of the concentration of dye and the liquor ratio, the greater the intensity of dye are required higher concentrations of electrolyte, and if the bath ratio decreases requires less amount of electrolyte.

The type of salt used is SALTEX a Brinsa marketed by NaCl, which has the following characteristics:

Sodium Chloride% m / m dry basis	99.0 min
Magnesium, mg / kg	2 máx.
Calcium, mg / kg	16 máx.
Humidity,% m / m of H2O	0.05 máx.
Other soluble in water, mg	460 máx.

/ kg	

Tabla # 5 Features of the electrolyte used: SALTEX

6.3 Ph

The absorption step in reactive dyes is performed at neutral pH since the elevation of the reaction pH of the dye with the fiber or with the water, and if the dye is not yet absorbed in the fiber is increased hydrolysis is important knowing that at the start of dyeing the pH should be equal to 6 to ensure that the dye does not begin to react with the fiber.

The alkaline pH activates the dye binding to the fiber, so that at this stage it is recommended that a pH value between 11 and 11.2, which is achieved with the dosage of alkali, using a simple method for setting the bass and complicated nuances, ie only with sodium carbonate (Na2CO3) and a mixed setting for moderate and high intensities using Sodium Carbonate (Na2CO3) + sodium hydroxide (NaOH)

6.3.1 FIXING TEMPERATURE

The optimum temperature setting is Corazol thereon 60oC, in the dyes as they are highly reactive and if Turquoise Blue colors formulated with 2GP Corazol to 80oC. dye being low reactivity.

6.3.2 WATER HARDNESS

The means of transport used to get products to the fiber is water, and must meet certain conditions to qualify for use in the textile process, from the controls to be carried out this hardness control.

Hardness. - Is produced by the presence of calcium and magnesium salts

PARÁMETROS	VALORES
hardness	0 - 8.5°A (alemanes)
Suspended solids (MES)	< 5 mg/l.
pH	cercano a 7
Solids	< 100 mg/l.
reducing material	Indetectables
iron	< 0,3 mg/l.
manganese	< 0,01 mg/l.
copper	< 0,01 mg/l.
D.B.O.5	cercano a 0

Tabla # 6 International standard of water quality in the textile industry

This work was performed with water that has passed through a softening system which allows to maintain hardness values below 15ppm, which is checked by a test to evaluate the content and the amount of hardness metals is within acceptable limits which is favorable because some dyes are very sensitive to these variations.

6.4 HYDROLYSISIDROLISIS

Literally means destruction, dissolution or change of a chemical substance by water.

The dyes can undergo alkaline hydrolysis or acid hydrolysis

6.4.1 ALKALINE HYDROLYSIS

- Exaggerated fixing conditions (excessive fixation time and / or pH and / or temperature)
- Wash at high temperatures (> 70 ° C), before all the alkali for fixation has been rinsed.
- Post-dyed mercerised genres.
- Repeated washings with conventional detergents at temperatures> 60 ° C.
- .

6.4.2 ACID HYDROLYSIS

- Neglect in the mercerizing neutralization.
- - Using too strong acids as catalysts in the finish.
- - Application of softening unusually acidic conditions.
- - Sweat acid.
- - Packing materials (PVC) that under the influence of heat and light, release acids during storage of genres.
- - Carelessness during rinsing, as is the use of slightly acidic water or demineralized water.

6.5 A PROCESS AFTER THE DYE "THE SOAPING”.

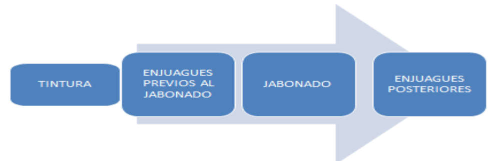


Fig # 7 Stages of the elimination phase of hydrolysed dye

his stage of the dyeing process comprising the following steps

6.5.1 Rinse to Reduce Salt

6.5.2 Diffusion, Desorption of dye hydrolyzate - soapingifusión

6.5.3 Elimination of dye hydrolyzatEliminación

6.6 VARIABLES PROCESS INTERFERING REMOVING DYE

6.6.1 LIQUOR RATIO

The bath ratio increased helps eliminate more rapidly the residual electrolyte, and the dye hydrolyzate.

6.6.2 TEMPERTATURE

An increase in temperature improves the solubility of the dye hydrolyzed without affecting the dye reacted

6.6.3 ELECTROLYTE

The presence of electrolytes in the wash water leads to increased substantivity of hydrolysed dye removal difficult.

	R/B	1:7			1:10			1:12		
	REQUIRED NaCl in dyeing according to the percentage DYE	RINSES			RINSES			RINSES		
		1	2	3	1	2	3	1	2	3
CONCENTRACION DE SAL g/l	50	13.3	3.56	0.95	10.00	2.00	0.40			
	60	16.00	4.27	1.14	12.00	2.40	0.48	9,6	1.54	0.25
	70	18.67	4.98	1.33	14.00	2.80	0.56	11.2	1.79	0.29
	80	21.33	5.69	1.52	16.00	3.20	0.64	12.8	2.05	0.33
	90				18.00	3.60	0.72	14.4	2.3	0.37
	100							1	2.56	0.41

Fig # 8 Influence of Liquor ratio in removing the electrolyte. Relación de baño en la eliminación del electrolito.

As shown in the table from the use of 50g / l of the dye salt is insufficient after a single rinse is discharged and the dye bath in this phase is also important working bath ratio as a greater amount of water used is greater than the amount of salt is eliminated, it is considered optimal work process with up to 3 g / l of residual salt.

6.6.4 Ph

An alkaline pH in the process of making the colorant rinses having affinity for the aqueous phase which makes its disposal, it is recommended based on testing the pH is 6 at this stage and 7 as colored Turquesas lower pH causes the shift of a yellowish hue

6.6.5 TIME

The mouthrinses made time should not be longer than 10min since no improvement is not observed while increasing

6.7 PRODUCTS USED IN SOAPING

6.7.1 DETERGENTS

-Foaming

- Resorption dye

ANIONIC ionize into two parts: (-) cleaning agent

Ionize nonionic: whole molecule acts as a cleaning agent

CATIONIC ionize into two parts: (+) cleaning agent

Amphoteric molecule centers (-) (+), assumes a function of pH or dissolution media

6.7.2 CHELATES

- Demetallised certain dyes
- Resorption dye

6.7.3 SEQUESTRANTS DISPERSANTS

- Cover multivalent metal ions
- - Facilitate particle dispersion

6.7.4 PROTECTIVE COLLOID

- Resorption inhibiting dye

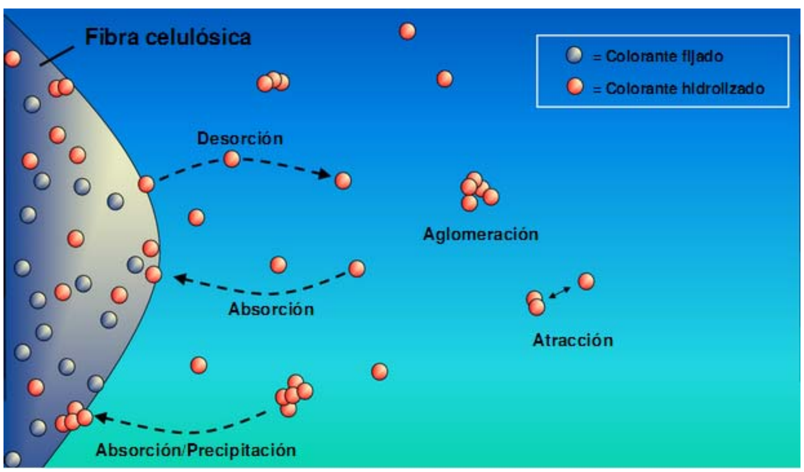


Fig # 9 Dynamic Balance After the dyeing

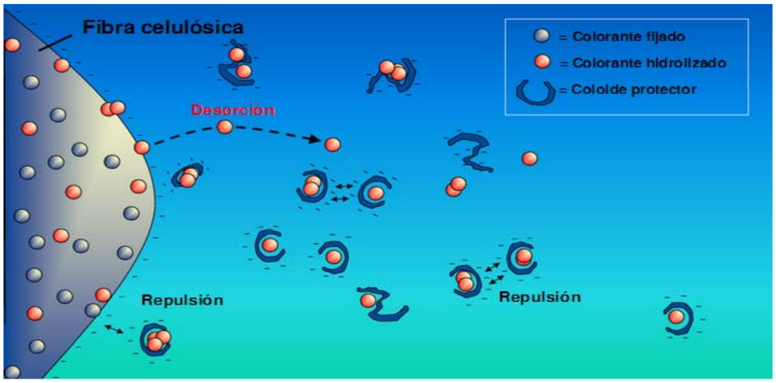


Figura # 10 Soaping with protective colloid

7.1 EVALUATION SOAPING PRODUCT

The products evaluated are recorded in the following table

7 RESULTS

PRODUCTO	FUNCION	CARACTER QUIMICO	CARACTER IONICO	PH	% BRIX
SANDOPUR R3C LIQ.	Agente secuestrante, dispersante y coloide protector	Acido policarboxilico	ANIONICO	5	18
ERIOPON WFE	Agente de jabonado funciona independiente de la dureza o la sal recidual	Polímero vinil acuoso	NO IONICO LIGERAMENTE CATIONICO	7 - 8	13
SERA FAST C-FRD	Agente secuestrante, dispersante y coloide protector	Mezcla de Surfactantes	CATIONICO	8±1	28

Tabla # 7 Evaluated Products soaping

ASSESSMENT METHOD

-. Dyeing with dye hydrolyzate (Jet Black color Corafix GDR 6%)

conditions:

R / B 1:6

Temperature 80 ° C

Time: 30 min

-. Soaping (Sandopur, Eriopon, Serafast)

- Collection of residual baths

-. Tincture 80 ° C - 10 min with residual rinses made.

-. Evaluation of the discoloration of the dyed fabric

Is important to have a product that does not foam to prevent tangling of tissue that can cause stains and dyeing inequalities

SANDOPUR R3C LIQ 2g/l ERIOPON WFE 2g/l SERA FAST C-FRD

Fig # 11 Foaming

Because in tests The R3C Sandopur liq, Eriopon WFE, Serafast CF-RD, behave similarly to make the process of soaping of the dyes produced by Colurtex under the specified circumstances, I consider only the influence of important costs these products in recipe

	KILOS	1						
	R/B	1:10						
	LITROS	10						
	clasificaion por intensidad		bajos	medios	fuertes	turquezas		
	Σ% de colorante		<5%	> 0.5 - 2%	>2-7%	> 0.5 - 2%	>2-4%	$/K PRODUCTO
g/l necesarios por proceso	SANDOPUR R3C liq	g/l	1	1	2	1	3	2.04
	ERIOPON WFE	g/l	1	1	2	1	3	4.87
	SERAFAST CF-RD	g/l	1	1	2	1	3	4.25
$ / proceso	SANDOPUR R3C liq	g/l	0.0204	0.0204	0.0408	0.0204	0.0612	
	ERIOPON WFE	g/l	0.0487	0.0487	0.0974	0.0487	0.1461	
	SERAFAST CF-RD	g/l	0.0425	0.0425	0.085	0.0425	0.1275	
	SANDOPUR R3C liq	58%	mas economico que Eriopon WFE					
	SANDOPUR R3C liq	52%	mas economico que Serafast CF-RD					

7.2 OPTIMIZATION OF ELIMINATION PHASE HYDROLYSED DYE

Based on the tests performed to determine in how they influence the different variables involved in this phase are detected the following problems from above:

- The sorting process is too limited, the use of a single process can not optimize resource consumption

- Ignorance of the effect the subsequent process steps to remove the dye hydrolyzate

- The lack of a method to evaluate the behavior.

- Not properly removes residual salt dyeing process and neutralizing the bathroom before the rinses is poor

    - In the strong colors performing a process set in pH 4.5, with cationic productsn

6.3 RATING OF DOWNSTREAM OF DY

CO-REACTIVOS	bajos	medios	fuertes	turquezas	
	<5%	> 0.5 - 2%	>2-7%	> 0.5 - 2%	>2-4%
Sal textil g/l	10 – 40	>40 - 60	>60	10 – 40	>40
Carbonato de Sodio g/l	5	5	5	14	20
Sosa Caustica g/l	0.7	1	>60		

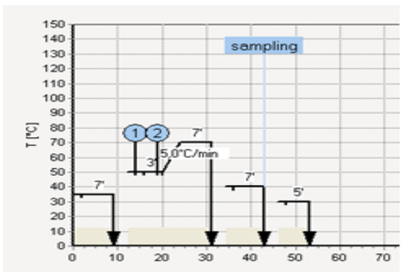
Turquezas <0.5% WORKING WITH THE PROCESS OF LOW

- selected product: R3C Sandopur liq 2g / l
- pH 6, pH 7 TQ
- R / B 1:10

Tabla # 8 Clasificación de la fase de eliminación de colorante hidrolizado en función de la concentración de colorante de la receta de tinte

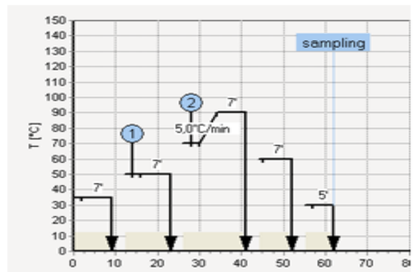
7.2.1 DISEÑO DE CUVAS DE PROCESO PARA OPTIMIZAR LA FASE DE ELIMINACION DEL COLORANTE HIDROLIZADO

PROCESO POSTERIOR - TONOS BAJOS <0.5%



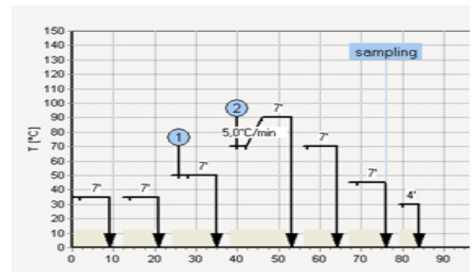
1	ACIDO CITRICO pH 6
2	SANDOPUR R3C Liq 1g/l

PROCESO POSTERIOR - TONOS MEDIOS > 0.5 - 2%



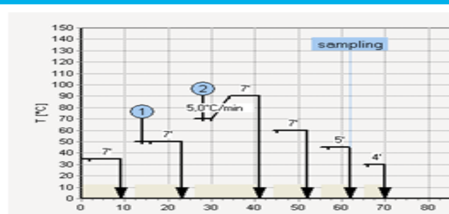
1	ACIDO CITRICO Ph6
2	SANDOPUR R3C Liq 1g/l

PROCESO POSTERIOR - TONOS FUERTES >2-7%



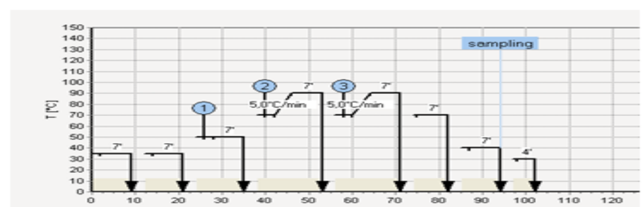
1	ACIDO CITRICO pH 6
2	SANDOPUR R3C Liq 2g/l

PROCESO POSTERIOR- TONOS BAJOS Y MEDIOS TURQUESAS Σ colorante >0.5 - 2%



1	ACIDO CITRICO pH 7
2	SANDOPUR R3C Liq 1g/l

PROCESO POSTERIOR- TONOS FUERTES TURQUESAS >2 - 4%



1	ACIDO CITRICO
2	SANDOPUR R3C Liq 2g/l
1	SANDOPUR R3C Liq 1g/l

8. CONCLUSIONS

- At the end of a 100% cotton dyeing with reactive dyes part of dye reacts with the fiber and some with water, but for the high potential hydrolyzed must be strictly controlled:

- The affinity of the reactive dyes

- The electrolyte used

- The pH

- Temperature and time setting

- Water Hardness

- Establishing controls to the successful completion of the process after the dyeing of cotton with reactive dyes is relevant and should be approached in three phases:
    1. Rinses after dyeing and neutralized.
    2. soaping
    3. Subsequent rinses.
- Rinses after dyeing must be performed no more than 40 ° C, and are intended to remove the dye hydrolyzed in water, the greater amount of alkali electrolyte.

- Complete removal of dye A hydrolyzate is only feasible after the electrolyte reduce washing system a minor amount of 3g / l as larger quantities increase the substantivity of the dye and its removal difficult. This is accomplished with a wash R / B 1:10 and color dye concentration by requiring the use of more than 50g / l NaCl two rinses needed to achieve this goal. The work requires lower liquor ratios of greater number of baths to achieve the elimination optimal amounts of electrolyte and therefore more time.

- The pH at the end of the dyeing is between 10.6 and 11.2 it is necessary that it is necessary the use of an acidulant product after downloading the dyebath. According to tests carried out it was observed that the process is more efficient when working at pH 6

- The use of adjuvants in the soaping stage allows the elimination of the dye in the fiber hydrolyzate, this phase should be made based on the amount of dye present in the dyeing recipe. The optimum working temperature is 90°C, this temperature rise decreases the affinity of the dye for the fiber, and increases the solubility of the dye colorant facilitating removal hydrolyzate.

- In this paper the product Sandopur R3C LIQ. Under the following conditions:

Optimal conditions in the soaping	
WATER HARDNESS	< 20ppm

Bath Ph	6
Residual electrolyte	< 3g/L
R / B	1:10
Temperature soaping	90°C

Tests show that the performance of Sandopur R3C LIQ. Fast will Eriopon C-DFR and WFE is similar and improves its work at pH 6. The optimal recipe is obtained with 2g/l

PRODUCTO	FORMACION DE ESPUMA	IONICIDAD	COSTO $/K producto	g/l requeridos	pH de trabajo
SANDOPUR R3C LIQ.	NO FORMA ESPUMA	ANIONICO	2.04	2	6
ERIOPON WFE	LEVE FORMACION DE ESPUMA	NO IONICO - LEVEMENTE CATIONICO	4.87	2	6
SERA FAST C-FRD	FORMA ESPUMA	CATIONICO	4.25	2	6

- In turquoise shades strong due to its low setting value can perform two processes soaping at 90 ° C and the pH is neutralized optimum pH is 7 as lower causes variation in hue.

- After soaping process takes two rinses trying to allow the second end to an optimum temperature to proceed to download tissue without loading a new cooling bath.

- ith the completion of this investigation was obtained a reduction of process time, reduced water consumption and thus a better utilization of which is recorded in the following comparative table:

	litros	costo productos	min	% litros totales	%min respecto a min p. ant.
PROCESO ANTERIOR	64	0,04	92	62%	
	82	0,11	137	68%	
OPTIMIZACION	36	0,04	52	47%	57%
	46	0,04	62	52%	67%
	66	0,06	84	62%	61%
	56	0,04	70	58%	76%
	76	0,07	102	66%	74%

- Due to the time savings achieved, you can increase the number of items per month so stained that increases productivity, reduces energy costs and therefore the cost of the process.

9. RECOMMENDATIONS.

- Tests performed for evaluation of products can be applied soaping article the methods used are practical and can appreciate how they influence the different steps that are performed to remove the dye.
- Consider selecting a new range of reactive dyes that the higher the percentage value in fixing have better washability, require less water to be removed and therefore less processing time.
- Is needed solides tests to verify the efficiency of the process. The slightly turbid water does not mean they need to make a new rinse.
- Must perform several washing processes which must be considered, the% dye recipe of the dyeing, the advantages of the machine on which the process is carried.
- When the pH was not controlled process is a clear need to increase rinses, but you should continue to perform correct before rinsing.
- It is important to know the composition of the products used, the pH, and the ionicity
- It must control the water quality used throughout the dyeing process to ensure that this variable does not affect the desired results.
- There must be a hydrometer to determine the value of salt and a pH meter to monitor the pH of the process
- It is recommended to design tank or cistern to retrieve the baths used in the process after dyeing, which could be reused in the same process

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