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# Study of the Water and Energy Consumptions in the Dyeing of Cotton with *Curcuma Longa* by Pad-Batch Process Using Response Surface Methodology

Mônica A. Faloppa<sup>a</sup>, Joselene B. F. Correia<sup>a</sup>, Thaís S. Silva<sup>a</sup>, Bruna R. Daniel<sup>b</sup>, Raquel S. R. Almeida<sup>b</sup>, Marta H. F. Spoto<sup>b</sup>, Jorge M. Rosa <sup>a,c,d</sup>, and Sueli I. Borrely <sup>d</sup>

<sup>a</sup>Department of Textile Chemistry, College of Technology SENAI “Antoine Skaf”, São Paulo, Brazil; <sup>b</sup>College of Agriculture “Luiz De Queiroz”, University of São Paulo – USP, Piracicaba, Brazil; <sup>c</sup>College of Chemical Engineering, State University of Campinas – UNICAMP, Campinas, Brazil; <sup>d</sup>Radiation Technology Center, Nuclear and Energy Research Institut, IPEN-CNEN/SPe, São Paulo, Brazil

## ABSTRACT

Mathematical modeling was employed in order to optimize pad-batch process using *C. longa* natural dyestuff applied in dyeing of cotton, against the conventional dyeing by exhaustion with the same dyestuff under best applications parameters recommended by dyestuff supplier. Ecological costs, consumption of water, electrical, and thermal energy, were assessed. The application of the model in the studied process versus the conventional process demonstrated that is possible to obtain an economy of  $1.418 \times 10^6$  J kg<sup>-1</sup> of energy, in addition to an economy of 95% in water consumption, without significant detriment in the color fastness assessed.

## 摘要

采用数学建模的方法,用C.Longa龙卡天然染料应用于棉织物的染色,在染料供应商推荐的最佳应用参数下,与传统的同一种染料上染进行对比.评估了水、电和热能的生态成本.将该模型应用于所研究的工艺与传统工艺进行比较,结果表明,该模型可获得1.418的经济性×能量为106 Jkg<sup>-1</sup>,耗水量经济性为95%,且不会对评估的色牢度造成重大损害.

## KEYWORDS

Natural dyestuff; textile dyeing; ecological costs; consumption of energy; consumption of water; response surface methodology

## 关键词

天然染料; 纺织品染色; 生态成本; 能源消耗; 用水量; 响应面法

## Introduction

Natural dyestuffs are gaining interest due their expected low risk to human health and the environment and biodegradability. Until the middle of the 19th century, only natural colorants were used for textile coloration and after the advent of synthetic dyes, the use of natural colorants declined drastically and today only a small fraction of textiles that are commercially traded are colored with the natural dyes (Silva et al. 2020b; Zerín et al. 2020).

In the textiles dyeings, these kind of dyestuff are commonly applied by exhaustion process, consuming a considerable amount of water and energy. However, there are ever-growing potential new sources of natural dyestuff in the form of production waste products that merit consideration for coloration of textile materials as an alternative of synthetic dyestuffs (Rossi et al. 2017). Henna, for example, is a red-orange pigment that has long been used for the coloration of skin and hair as well as textile materials (Bhuiyan et al. 2017) and *Thespesia populnea* is another plant that was studied in order to separate natural dyestuff to obtain dyeing properties on different textile fabrics (Mohini, Tejashree, and Vijay 2018).

Nowadays, natural dyestuffs are strongly researched in the textile area applications, such blueberry waste for dyeing cotton with biomordants (Phan et al. 2020); *Lawsonia inermis* on cellulosic, protein, and synthetic fibers (Bhuiyan et al. 2017); waste from eucalyptus wood steaming (Rossi et al. 2017); natural dyestuff obtained from the fruits of *Terminalia arjuna* and *Thespesia populnea* (Karuppuchamy, Grace Annapoorani, and Narayanasamy 2019); plasma treatment employed to improve the dyeability of wool fibers with cochineal (Sajed et al. 2018); printing of viscose rayon (Patel and Kanade 2019) or even on diverse textile materials (Fröse et al. 2019).

It is also have being applied on other areas, such in solar cells (Mensah-Darkwa et al. 2020; Özbay Karakuş et al. 2017; Kabir et al. 2019b, 2019a; Hosseinneshad et al. 2020; Ruhane et al. 2017; Djibrilla Alio et al. 2021), dyeing of plastics via the sol-gel process (Velho et al. 2017), bioapplication as a fungicide (De Lima et al. 2019) and in the human spermatozoa for morphology assessment (Chomean et al. 2019).

The natural dyestuff *Curcuma longa* also have been researched in textile area in order to obtain applications in hospital patients and even medical workers to prevent the microbial infections (Maghimaa and Alharbi 2020). The textile application of *C. longa* also was studied by was studied by Naveed et al. (2020), in which the authors observed that microwaves increased the color strength as well as color fastness properties of irradiated cotton using aqueous solubilized mixed extract of irradiated pomegranate rind and turmeric rhizome powder.

Those facts demonstrate the importance of the various natural dyestuffs, all of them obtained through renewable sources. Besides, there is also the fact that textile effluents with synthetic dyestuffs are extremely polluting, as demonstrated by Iqbal, Abbas, and Nazir (2019) that described about bioassays based on *Allium cepa* as dosimeters for ecotoxicity monitoring; *Vibrio fischeri* was applied by Abbas et al. (2018) for the monitoring of toxicity, applicable to many types of matrices such textile effluents and Iqbal (2016) described about the utilization of *Vicia faba* bioassay for environmental toxicity monitoring.

## Materials and methods

Two dyeing process on cotton fabrics were compared, one by exhaustion and other by pad-batch process (Figure 1).

In both process, aluminum potassium sulfate PA 98%  $[KAl(SO_4)_2]$  was used as mordent salt supplied by Labsynth, Brazil; *C. longa* as dyestuff supplied by Quimica Inteligente, Brazil, being all chemicals used with no previous purification.

### Substrate

The experiments were executed in a 100% cotton woven fabric with gramature equal to  $180 \text{ g m}^{-2}$ , 1.60 m of width, with 26 20/1 Ne yarn counts per cm in the weft and 24 30/1 Ne yarn counts per cm in the warp, yield equal to  $0.288 \text{ m kg}^{-1}$ , bleached according to process described by Rosa et al. (2019).

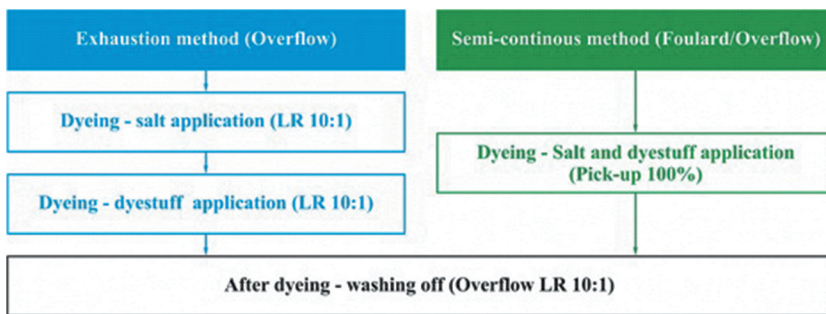


Figure 1. Comparison between exhaustion and Pad-batch processes

The width (m), gramature ( $180 \text{ g m}^{-2}$ ), and yield ( $0.288 \text{ m kg}^{-1}$ ), were used in order to calculate the ecological costs.

**Water consumption**

The 10:1 Liquor Ratio (LR) was used in order to calculate the amount of water spent, being 10 L of water for each dyed kilogram in the salt and in the dyestuff application, applying the equation 1.

$$Lkg^1 = LR_1 + LR_2 + LR_n \tag{1}$$

Where  $L \text{ kg}^{-1}$  = total L per kg;  $LR_1 = 10 \text{ L per kg for the salt application}$ ;  $LR_2 = 10 \text{ L per kg for the dyestuff application}$ .

The formulation applied on this process had 5% on weight of fabric (owf) of dyestuff and  $5 \text{ g L}^{-1}$  of salt (Mathis Alt-1), with liquor ratio (LR) equal to 10:1, according to dyestuff supplier recommendation. The process already studied by dyestuff supplier is described graphically in the Figure 2.

**Pad-batch process**

The scarcity of water, along with the amount of pollution created by dyers and its treatment cost, has led to a rethink about the conservation of water used in textile processes (Gopalakrishnan, Punitha, and Saravanan 2019). The pad-batch process promotes a high economy of water in the dyeing process of cellulosic fibers with many kind of dyestuffs, alone or in combination with many others aggregated process (Babar et al. 2017; Shu et al. 2018; Tavares et al. 2018; Yu et al. 2020).

The pick-up used in the experiments was 100% (equation 2), being 1 L of water per each kilogram, dyed in a bath containing dyestuff and salt.

$$Pick - up = - \left[ 1 - \left( \frac{WW}{DW} \right) \right] \times 100 \tag{2}$$

Where *Pick-up* in %; *WW* = wet weight; *DW* = dry weight

Dyestuff and salt were applied simultaneously (Foulard Mathis), with the same amount of dyestuff applied by exhaustion. The proportionally amount of salt was used in the center point of the  $2^2$  planning factor ( $50 \text{ g L}^{-1}$ ). The process is described in the Figure 3.

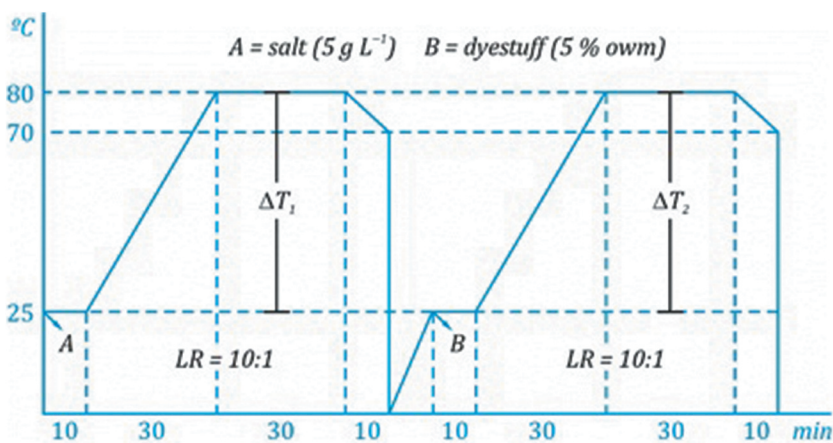


Figure 2. Exhaustion process

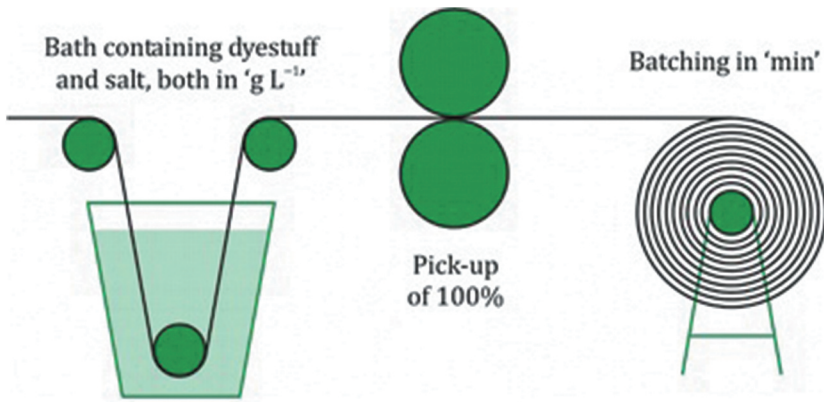


Figure 3. Pad-batch process

**Modeling**

Response Surface Methodology (RSM) has been widely used in various processes optimization, promoting significant less spend of energy and water consumption, in addition to chemical among other inputs (Abdulgader et al. 2020; Bahrami, Amiri, and Bagheri 2019; Safari et al. 2019; Samarbaf et al. 2019; Siddiqua et al. 2020, 2021; Umer et al. 2019).

In this paper, the experiments were assisted by 2<sup>2</sup> planning factor, accomplished with a Central Composite Rotatable Design (CCRD), two levels and alphas, analyzed by ANOVA model and by RSM (Academic Statistica 13<sup>®</sup>).

The independent variables studied were the amount of salt and the time of batching (Table 1). The dependent variable was the coloristic strength (K S<sup>-1</sup>), analyzed by visible spectrophotometry under illuminant D<sub>65</sub>, 10° (Konica-Minolta CM-3600d).

The response surface model was used for the preliminary regression fits, using equation 3 (Jaafari and Yaghmaeian 2019; Jeffrey et al. 2019; Rosa et al. 2019).

$$Y = \beta_0 + \sum_{j=1}^k \beta_j x_{ij} + \sum_{j=1}^k \beta_{ij} x_{ij}^2 + \sum_{j < m}^{k-1} \sum_m^k \beta_{jm} x_{ij} x_{im} \tag{3}$$

Where ‘Y’ is the response,  $\beta_0$ ,  $\beta_j$ ,  $\beta_{ij}$ , and  $\beta_{jm}$  are the regression coefficients for the intercept (linear, quadratic, and interaction parameters), and  $x_{ij}$  and  $x_{im}$  are the intercept variables.

The ANOVA was employed for the determination of significant variables, analyzing the F-value as the ratio of the mean square of regression, representing the significance of each controlled variable on the tested model (Santana et al. 2018; Sathish and Vivekanandan 2016). For an adjusted model, the calculated F is  $\geq$  than tabulated F, with R<sup>2</sup> value near to 1 (Chitichotpanya, Pisitsak, and Chitichotpanya 2018; Das and Mishra 2017).

**Ecological costs**

**Electrical energy**

The equipment Jet HT Riviera Eco Metalwork parameters was used in order to calculate the electrical energy applied in the exhaustion process, considering the process capacity of 50 kg and installed

Table 1. Independent and dependent variables studied

Independent variable	Variable code	Levels				
		$-\sqrt{2}$	-1	0	1	$\sqrt{2}$
Salt (g L <sup>-1</sup> )	x1	14.6	25	50	75	85.4
Time (min)	x2	18	30	60	90	102

potency of 7.4 kW. The theoretical consumption for each kilogram of processed substrate was determined by the time of the process, in minutes, applying equation 4 (Rosa et al. 2019, 2014).

$$Q_{E1} = \frac{t \times I_p \times 6.00 \times 10^4}{E_C} \tag{4}$$

$Q_{E1} = J \text{ kg}^{-1}$ ;  $t = \text{process time in min}$ ;  $I_p = \text{installed potency}$ ;  $E_C = \text{equipment capacity}$

The equipment Padding Foulard Crispim was used in order to calculate the electrical energy applied in the pad-batch process, considering an operation with a speed of 20 m min<sup>-1</sup> and installed potency of 4.7 kW. The theoretical consumption for each kilogram of processed substrate was determined applying equation 5.

$$Q_{E2} = I_p \times \frac{Y}{F_S} \times 6.00 \times 10^4 \tag{5}$$

$Q_{E2} = J \text{ kg}^{-1}$ ;  $I_p = \text{installed potency in 'kW'}$ ;  $Y = \text{yield in 'm kg}^{-1}$ ';  $F_S = \text{foulard speed in 'm min}^{-1}$ ';  $t = \text{process time in 'min'}$

In order to calculate the amount of thermal energy spent in the processes, the equation 6 was applied.

$$Q_T = \sum_n^1 \Delta T \times C_{p_{H_2O}} \times m_{H_2O} \times 10^{-3} \tag{6}$$

$Q_T = J \text{ kg}^{-1}$ ;  $T^o$  in Kelvin used in all steps;  $C_p$  in  $J \text{ kg}^{-1}K^{-1}$ ;  $m$  in grams, adopting specific mass of water = 1.0 g cm<sup>-3</sup>;  $LR = 10:1$ .

### Color fastness

The color fastness to water (ABNT 2013) and rubbing (ABNT 2013) of the colors obtained by the two process were assessed by visible spectrophotometry under illuminant D<sub>65</sub>, 10° (Konica-Minolta CM-3600d).

## Results

### Dyeings

The K S<sup>-1</sup> value obtained in the dyeing by exhaustion process, executed according to orientation of dyestuff supplier, was 5.94.

### Modeling

The K S<sup>-1</sup> values of the dyeings executed by pad-batch process assisted by CCDR experimental planning and applied during the assays are described in the Table 2.

This data was used to obtain the model by the least squares' method. It can be observed similarities between experimental K S<sup>-1</sup> values and calculated K S<sup>-1</sup> values, obtained by the model. It is also observed graphically in the Figure 4, where low data fluctuation is presented.

The equation of the model predicted, only with the significant interaction ( $p \leq 0.05$ ), is shown in Equation 7.

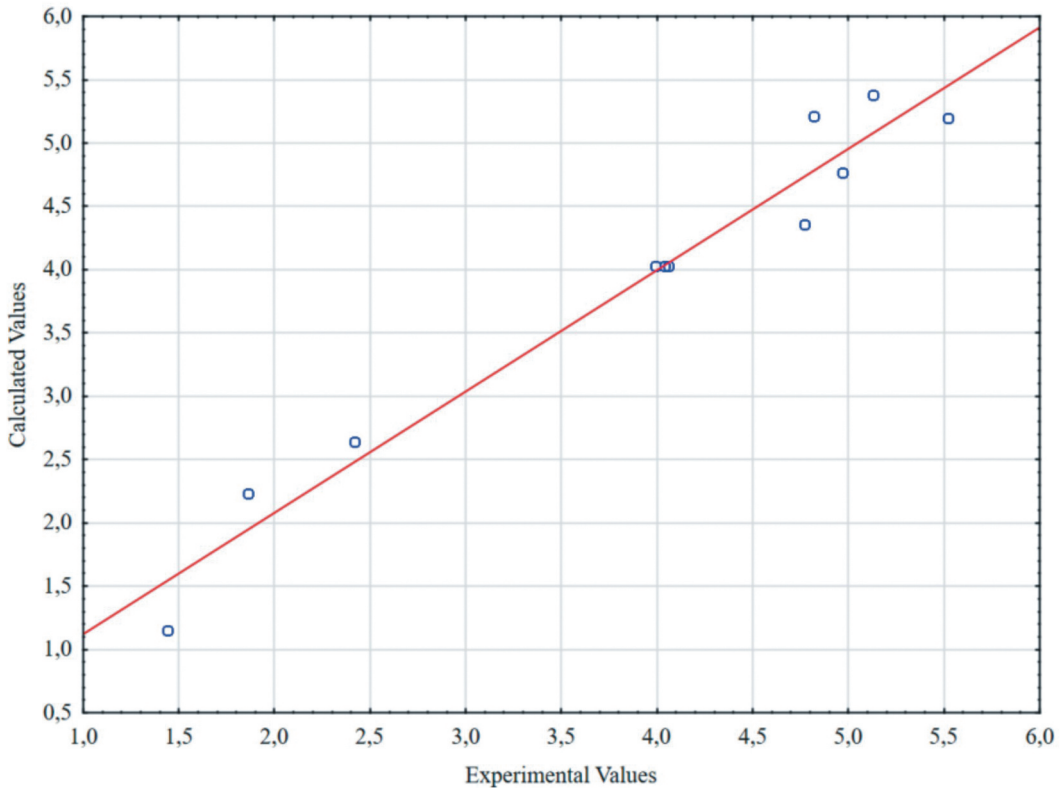
$$KS^{-1} = -0.0232 + 1.3089x_1 - 0.0686x_1^2 \tag{7}$$

Table 3 presents the ANOVA analysis to the model, demonstrating the adjusted to the experimental data.

The multiple regression value was close enough to the unit and the variance values were near 86%. Those value obtained indicates that the model is able to predict 81.80% with at a 95% of confidence

**Table 2.** K S<sup>-1</sup> values of pad-batch process

x1	Salt (g L <sup>-1</sup> )	x2	Time (min)	K S <sup>-1</sup> (exp)	K S <sup>-1</sup> (calc)
-1	25.0	-1	30	1.86	1.95
-1	25.0	1	90	2.42	2.20
1	75.0	-1	30	4.82	5.28
1	75.0	1	90	5.13	5.05
$-\sqrt{2}$	14.6	0	60	1.44	1.53
$\sqrt{2}$	85.4	0	60	5.52	5.04
0	50.0	$-\sqrt{2}$	18	4.77	5.15
0	50.0	$\sqrt{2}$	102	4.97	4.83
0	50.0	0	60	4.04	3.83
0	50.0	0	60	3.99	3.84
0	50.0	0	60	4.06	4.21

**Figure 4.** Experimental versus calculated K S<sup>-1</sup> values

level.  $F_{\text{Calc}}$  value was 8.35-fold higher than  $F_{\text{Tab}}$ , showing the significance of the model in the prediction of K S<sup>-1</sup> values under the studied conditions.

### RSM

Mathematical models have been studied to contribute into predictions on the amount of dyestuffs present in textile coloration processes (Siddiqua et al. 2021) among many other process such in the extraction of natural dyestuff to industrial applications such Jabeen et al. (2019); in the increasing pigment masses in ceramic glazes (Schabbach et al. 2018) or even in the use of calcium carbonate as a paper pigment (Bunkholt and Kleiv 2014). The studied factors coexist from the analysis of each surface if an optimal condition for each factor is obtained (Silva Filho et al. 2018).

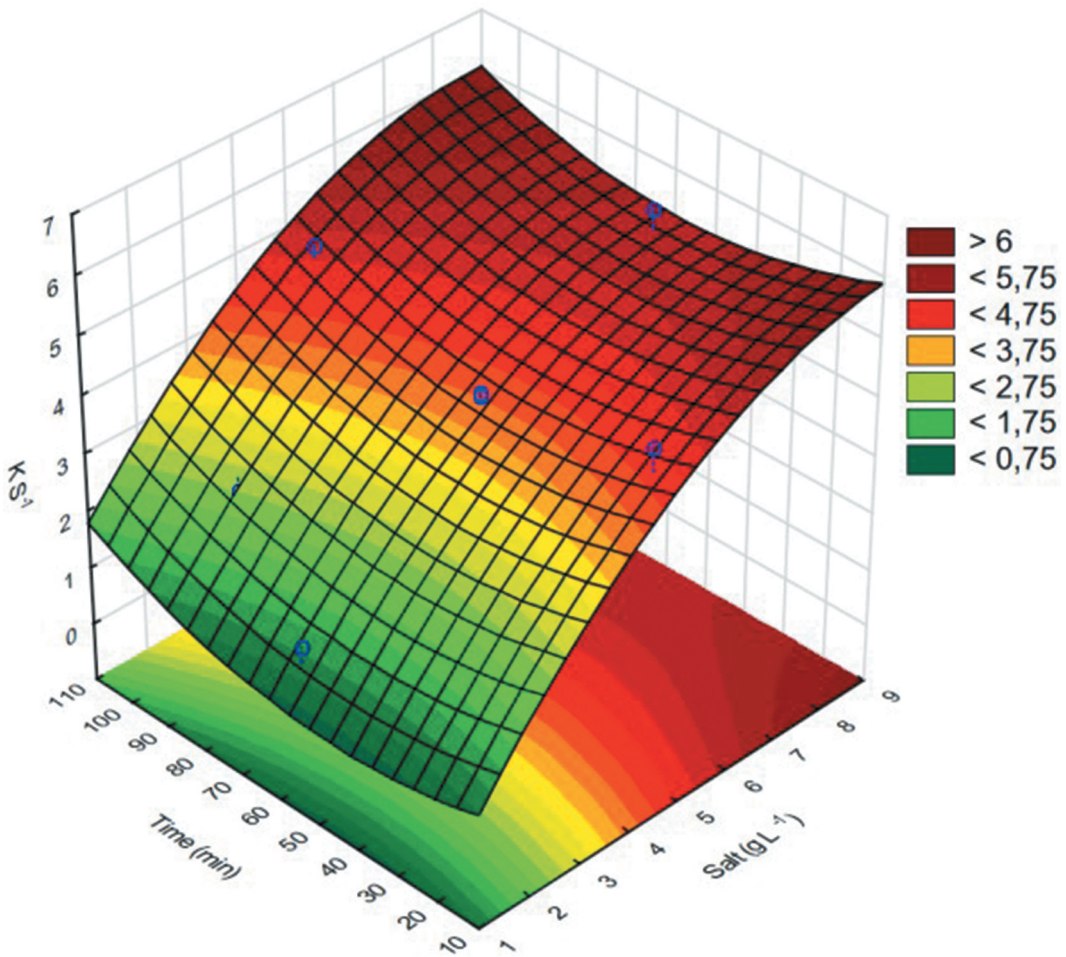
**Table 3.** ANOVA

RESULTS	Statistic	ANOVA				
		gl	SQ	MQ	F <sub>Calc</sub>	F <sub>Tab</sub>
Regression	-	2	16.5253	8.2626	23.4752	2.80
Residual	-	8	2.8157	0.3519	-	
Multiple R	0.9243	-				
Multiple R <sup>2</sup>	0.8544					
Adjusted R <sup>2</sup>	0.8180					

The RSM analysis of the present study is described in the [Figure 5, 6](#).

As demonstrated by ANOVA and by the model, the RSM corroborates the significance of the amount of salt in higher concentrations. The ideal time predicted by the RSM is in the values near to the central point in order to provide higher  $K S^{-1}$  values.

The critical values of salt and time obtained by the modeling (ANOVA, RSM, and model equation) were  $92 \text{ g L}^{-1}$  and  $57.8 \text{ min}$ , respectively. The experiment done with the ideal predicted conditions presented a  $K S^{-1}$  value of  $5.35$  against  $5.94$  obtained by the exhaustion process recommended by dyestuff supplier.



**Figure 5.** Interaction between independent variables



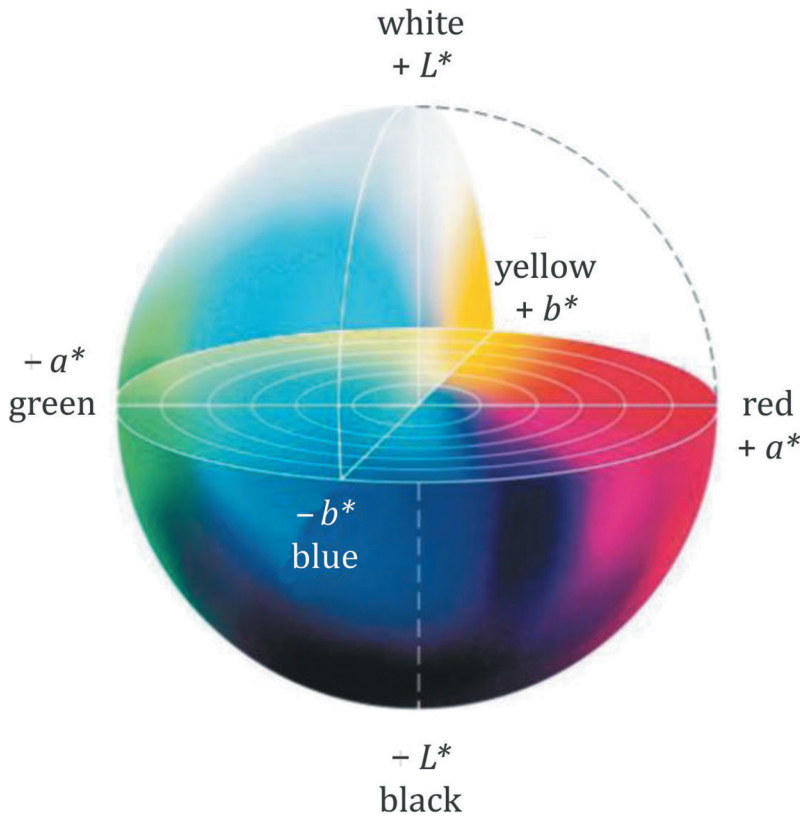


Figure 6. Color space in CIE Lab system (Konica Minolta, 2018)

### Experimental validation of the model

In order to validate the model experimentally, 30 dyeings were executed by pad-batch process applying the amount of dyestuff and salt obtained by the model. All results were assessed adopting the CIE Lab system. CIE Lab was determined by an organization called “*Commission Internationale de l’Eclairage*” (CIE). The system can be plotted in a color space with three axis (Figure 4), where  $L^*$  axis represents the difference between white [ $+ L^*$ ] and black [ $- L^*$ ];  $a^*$  axis represents the difference between green [ $- a^*$ ] and red [ $+ a^*$ ] and  $b^*$  axis represents the difference between yellow [ $+ b^*$ ] and blue [ $- b^*$ ] (Konica Garcia, Rosa, and Borrelly 2020; Minolta 2018).

The system is widely used techniques for measuring and demonstrating color of textile (Shams-Nateri and Hasanlou 2018; Silva et al. 2020b) and also in other areas, as example by Almeida et al. (2020), that studied the color variations in softwoods and hardwoods under the influence of artificial and natural weathering, or even by Von Gersdorff et al. (2021), where the authors observed the dehydration of beef slices.

The colors measurements were determined by spectrophotometry, under illuminant  $D_{65}$ ,  $10^\circ$  (Konica Minolta CM-3600d). The values of Euclidean distances ( $\Delta E^*$ ), also known by color difference or even total deviation, in the colors space between the first dyeing and all the other ones was calculated using the equation 8 given below (Rosa et al. 2020).

$$\Delta E^* = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \quad (8)$$

The results are described in the Table 4.

As demonstrated, the difference between  $\Delta E^*$  values did not exceed 2.0, and these data are acceptable when compared with the standards used in the Brazilian industry of clothing (Rosa et al. 2015). Besides, the predicted variance of  $\Delta E^*$  was just 0.1130, with 95% of confidence interval.

### Ecological costs

The theoretical calculated values of consumption of water, electrical, and thermal energy spent in the both exhaustion and pad-batch processes are presented in Table 5.

The dyeing executed by pad-batch process presented lower values of ecological costs than the dyeing executed by exhaustion process. The consumption of electrical and thermal energy of the pad-batch process were  $1.417 \times 10^6$  J  $\text{kg}^{-1}$  and  $1.10 \times 10^3$  J  $\text{kg}^{-1}$  lower than the exhaustion process, respectively. Moreover, the consumption of water in the pad-batch process was 1.0 L  $\text{kg}^{-1}$  compared with 20.0 L  $\text{kg}^{-1}$  consumed by exhaustion process, representing an economy of about 95%.

### Color fastness

The color fastness properties were tested and assessed according to the standards laid down by the Brazilian Association of Technical Standards (ABNT). All color changes and staining were evaluated by spectrophotometry under illuminant D<sub>65</sub>, 10°, and are described in the Table 6. Before the tests, the samples were conditioned for 24 h in a standard atmosphere at temperature of  $20 \pm 2^\circ\text{C}$  and relative humidity of  $65 \pm 2\%$  (Gun and Tiber 2011).

As can be observed, the difference presented in all assessments did not exceed a half point, in a scale from 1 (poor) up to 5 (excellent). Those data demonstrate that both processes can be applied with no detriment in the color fastness proprieties assessed.

**Table 4.** Values of Euclidean distances of all experiments

n	K S <sup>-1</sup>	L*	a*	b*	DE*
1	5.317	83.19	-2.77	44.31	<b>0</b>
2	5.283	83.16	-2.89	45.27	<b>0.968</b>
3	5.322	83.40	-2.57	44.87	<b>0.631</b>
4	5.273	82.97	-2.70	45.28	<b>0.997</b>
5	5.274	82.98	-2.61	45.36	<b>1.083</b>
6	5.244	82.62	-2.28	45.53	<b>1.433</b>
7	5.243	82.81	-2.33	45.88	<b>1.674</b>
8	5.323	83.44	-3.19	44.68	<b>0.613</b>
9	5.248	82.84	-2.77	45.70	<b>1.433</b>
10	5.279	83.01	-2.95	45.21	<b>0.935</b>
11	5.398	83.97	-3.25	43.80	<b>1.048</b>
12	5.347	83.82	-3.73	44.91	<b>1.296</b>
13	5.356	83.69	-3.37	44.51	<b>0.806</b>
14	5.455	83.21	-2.57	43.27	<b>1.059</b>
15	5.442	84.15	-3.63	43.64	<b>1.453</b>
16	5.321	84.02	-2.78	45.03	<b>1.099</b>
17	5.444	84.08	-3.60	43.54	<b>1.440</b>
18	5.500	83.49	-3.61	43.20	<b>1.424</b>
19	5.352	83.83	-3.38	44.85	<b>1.036</b>
20	5.376	83.38	-2.85	42.76	<b>1.564</b>
21	5.314	83.21	-3.35	44.15	<b>0.602</b>
22	5.263	82.94	-3.16	45.11	<b>0.924</b>
23	5.175	82.95	-2.03	45.23	<b>1.205</b>
24	5.115	83.03	-2.94	45.09	<b>0.814</b>
25	5.103	82.91	-2.64	45.88	<b>1.600</b>
26	5.221	82.90	-2.73	45.30	<b>1.032</b>
27	5.292	82.94	-2.67	44.64	<b>0.426</b>
28	5.278	82.77	-2.66	44.63	<b>0.539</b>
29	5.206	82.45	-2.41	44.83	<b>0.973</b>
30	5.111	82.56	-2.62	45.32	<b>1.200</b>

**Table 5.** Comparison of ecological costs

Process	Water (L kg <sup>-1</sup> )	Energy (J kg <sup>-1</sup> )	
		Electrical	Thermal
Exhaustion	20	$1.421 \times 10^6$	$1.100 \times 10^3$
Pad-batch	1	$4.061 \times 10^3$	0
$\Delta$	19	$1.417 \times 10^6$	$1.100 \times 10^3$

**Table 6.** Results of color fastness

Process		Water	Rubbing	
			Dry	Wet
Exhaustion	Color change	5	-	-
	Staining	4/5	5	4/5
Pad-batch	Color change	4/5	-	-
	Staining	4/5	5	4/5

## Conclusions

RSM has been applied efficiently as an appropriate computational tool to optimize different dyeing processes. Ideal conditions for the application of C. Longa in the dyeing of cotton fabrics in order to find higher  $K S^{-1}$  values obtained by pad-batch process were obtained satisfactorily.

The application of the model in the studied process *versus* the exhaustion process demonstrated that it was possible to promote an economy of  $1.418 \times 10^6$  J kg<sup>-1</sup> of energy, in addition to an economy of 95% in water consumption, without significant detriment in the color fastness indexes tested.

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## ORCID

Jorge M. Rosa  <http://orcid.org/0000-0003-1351-5516>  
 Sueli I. Borrelly  <http://orcid.org/0000-0002-9692-5539>

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