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Optimization of Process of Dyeing Alpaca Yarn Using Indigo Carmine (C.I. Natural Blue 2)

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Highlights

What are the main findings?

- A dyeing process for alpaca fiber using indigo carmine (C.I. Natural Blue 2) was optimized through a central composite design (CCD) based on response surface methodology (RSM), considering mordant concentration, temperature, and time as variables.
- The process exhibited a strong fit to a quadratic model, with a high coefficient of determination (R^2 and adjusted R^2 values exceeding 95%), despite some limitations in specific response regions.
- The fastness properties of the dyed fiber were acceptable according to the ISO gray scale, indicating a stable interaction between indigo carmine (C.I. Natural Blue 2) and alpaca fiber, which may be attributed to lasting chemical bonding facilitated by the process conditions.

What are the implications of the main finding?

- The optimized dyeing process offers a more sustainable alternative for protein-based fibers such as alpaca by utilizing food-grade dyes like indigo carmine (C.I. Natural Blue 2) under controlled conditions.
- The resulting dyed fibers demonstrate consistent color intensity and acceptable durability, supporting the viability of this approach for Peruvian textile coloration.
- These findings may promote further investigation into compatible food-grade dyes and encourage their industrial application in dyeing technologies.

Abstract: As part of an implementation in the Peruvian textile industry, the use of different sources to obtain blue hues in alpaca fiber has taken on a prominent role. The present study investigated the optimization of the dyeing process of alpaca fibers using indigo carmine as dye. The methodology was based on a central composite design (CCD) and response surface methodology (RSM) with color strength (K/S) as response variable. The results demonstrate that the independent variables significantly affected the color strength (K/S). In this context, an increase in both mordant concentration (3.9887 g/L) and dyeing temperature (95 °C), coupled with lower exhaust time (30.0019 min), enhanced levels of superficial dye adsorption. Additionally, color fastness properties provided tolerable values according to the gray scale. In conclusion, the optimization of the dyeing process of alpaca fibers using indigo carmine enabled the achievement of a blue shade with satisfactory fastness properties in the fiber yarns.

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Keywords: alpaca yarn; indigo carmine; fastness properties; RSM; CCD

1. Introduction

Alpacas are domestic camelids that belong to the South American camelid family. They are native to the Andean highlands (e.g., Chile and Bolivia), originating mainly from the Altiplano region of Peru [1,2]. According to [3], there are two types of alpacas in Peru: Huacaya and Suri. Huacaya alpacas account for more than 90% of the total alpaca population [1]. Alpaca fiber is widely valued in the Peruvian textile industry because of its unique properties (e.g., thermal qualities, mechanical resistance, and impermeability), which enhance its potential for innovative fashion [4]. Chemically, alpaca fiber is based on the same protein (i.e., keratin) as wool, which gives it the same dyeing properties [2]. However, alpaca fiber dyeability uses different conditions that are adjusted to suit its various characteristics, such as medullation and larger diameter, among others.

Generally, the alpaca fiber dyeing process uses synthetic dyes, such as acid dyes, reactive dyes, basic dyes, and vat dyes, which enable consistent colors and achieve significant characteristics and results for high-quality products. However, these dyes have disadvantages related to their environmental impact and the associated toxicity of their complex molecular structures, which cause several diseases in humans [5]. According to [6], the application of azo dyes has deleterious effects on all living organisms. The detrimental nature of these substances has raised growing concerns among environmentalists, thereby stimulating efforts to identify and develop sustainable and environmentally friendly alternatives.

Based on the aforementioned, dyes obtained from natural sources, such as plant extracts (e.g., leaves from *Cinnamomum camphora* (L.) J.Presl, flowers from *Indigofera suffruticosa* Mill, roots from *Rubia tinctorum L.*, and others), animals (e.g., cochineal lac from *Dactylopius coccus* and Tyrian purple from sea mollusk), and microbial sources (e.g., prodigiosin from *Vibrio* ssp., anthraquinones from *F. oxysporum*, and others) [7,8] are chosen as the friendliest alternatives. However, there are some general limitations. Regarding their extraction, the labor-intensive isolation of the coloring compounds [9] is complex; plant sources carry low amounts of dye or pigment and contain many compounds apart from the principal molecules, making the process time-consuming and difficult [7]. Another limitation concerns fading and instability, where plants and microbial sources present potential challenges. According to [10], many microbial pigments present have a limited life cycle under ambient conditions, making them unusable; even their production could be affected by contamination of raw materials. The challenges of dyeing procedures (e.g., difficulty in achieving precise control, low color yield, extended processing times, and other related issues) limit their feasibility for mass production [6,11,12].

As these challenges restrict the large-scale use of many dyes, certain artificial alternatives have shown potential [6]. One dye that has found a place in the food and textile industry is indigo carmine (IC), also known as Indigotine, C.I. Natural Blue 2, and indigo-5,5′-disulfonic acid disodium salt, among others [13]. Despite being an artificial dye, IC could offer advantages that would make it attractive from both a technical and environmental perspective for dyeing protein fibers such as alpaca fiber [14]. As a dye approved by regulatory bodies such as the European Food Safety Authority (EFSA), its toxicity levels are lower than other synthetic dyes, making it easier to handle [15,16]. IC is characterized by moderate heat stability and good resistance to reducing agents, although it has poor pH, light, and oxidation stability [17,18]. In addition, its solubility in aqueous media (10 g/L) and stability would allow a more efficient dyeing process [16]. However, recent

studies have raised concerns about its potential toxicity at higher concentrations and its environmental impact when released into aquatic environments without proper treatment. For instance, the untreated disposal of IC may contribute to water contamination and ecotoxicological risks. Therefore, while its regulated and limited use remains acceptable, these findings emphasize the importance of responsible management and the adoption of environmentally conscious dyeing practices. As discussed earlier, IC could be used for dyeing protein fibers, such as alpaca fibers.

The present study aims to determine the optimal conditions for dyeing alpaca fiber with indigo carmine. For this purpose, response surface methodology (RSM) was employed in order to optimize the process, due to the freedom to evaluate the effect and interaction of multiple process factors simultaneously [19]. Furthermore, the design of experiments (DOE) employed central composite design (CCD) in order to enable the establishment of a comprehensive correlation between the independent variables [19,20]. Experimental variables (mordant concentration, dyeing temperature, and exhaust time) were evaluated using color strength value (K/S, dependent variable), which allowed us to comprehensively understand the relationships and effects among these variables. This approach enabled us to comprehend the dyeing process using indigo carmine on alpaca fiber.

2. Materials and Methods

2.1. Materials and Reagents

For all dyeing experiments, alpaca yarn was purchased from INCA TOPS S.A. (Arequipa, Peru), and its specifications are listed in Table 1. Indigo carmine ($C_{16}H_8N_2Na_2O_8S_2$) was acquired from Aromas del Peru S.A. Tartaric acid ($C_4H_6O_6$) was purchased from Delta Quimica S.R.L. (Arequipa, Peru). Both tartaric acid and indigo carmine were food-grade quality. A non-ionic detergent (Hepalwed PDA) was kindly supplied by CITEtextil Camélidos Arequipa, and was used to wash and moisturize the fibers before dyeing.

Table 1. Specifications of the alpaca yarn used in this study.

Description	Data
Fabric	100% Baby Alpaca
Diameter	~20.1–23 µm
Count N/M [2]	2/28
Color	SFN10 [1]

[1] INCA TOPS S.A.'s internal color code for the natural undyed white alpaca fiber. [2] Yarn count in the Number/Metric system (Nm).

2.2. Alpaca Fiber Sample Preparation

Before the dyeing process, alpaca fibers were initially processed using an electronic wrap reel for yarns (161M_XYW, MESDAN, Arequipa, Peru) in order to produce skeins of uniform weight. This step ensured consistency in sample preparation, which was essential for subsequent stages of the process. Then, alpaca yarn samples were immersed in a non-ionic detergent solution (1 g detergent/L distilled water) as pre-treatment to moisturize their surface and remove excess paraffin and fat [21]. The solution was heated to 60 °C for 10 min, after which the samples were rinsed with distilled water to remove superfluous detergent.

2.3. Alpaca Yarn Mordanting Process

The yarn samples were mordanted by a simultaneous mordanting method as shown in [21]. In this context, the samples were immersed in the dyeing bath containing the dye liqueur and diluted with acidic mordant [20]. From the point of view of sustainability, ref. [22] reported eco-friendly alternatives for conventional mordants. Herein, we used tar-

taric acid ($C_4H_6O_6$) as an acidic mordant [23,24]. It is worth noting that other experimental conditions were explored using no mordant and alternative agents. These supplementary tests, including fastness assessments and coordination behavior analysis, are presented in the Supplementary Materials (Table S1).

2.4. Dyeing Process with Indigo Carmine

The samples were dyed using an exhaustion method in a dyeing machine (AHIBA IR PRO, DATA COLOR, Lawrenceville, GA, USA). The dyeing experiments were performed using a bath ratio of 1:30 (a liquor ratio of 1 g fiber per 30 mL of dye solution) in separate baths, with a dye concentration of 3% (o.w.f.). The dyeing temperature, exhaust time, and mordant concentration were determined according to the proposed experimental design outlined in Table 2. Upon completion, the dyed samples were rinsed thoroughly with distilled water and dried at room conditions in the shade [20,25,26].

Table 2. Experimental design variables and factor levels in the response surface design.

Design	Independent Variables		Factor Levels			Response
	Factor	Symbol	Low (−1)	Center (0)	High (+1)	
Central Composite Design (CCD)	Mordant concentration (g/L)	X_1	2	3	4	Color strength (K/S)
	Dyeing temperature (°C)	X_2	85	90	95	
	Exhaust time (min)	X_3	30	45	60	

2.5. Color Analysis and Color Strength of the Alpaca Yarn Samples

The spectral reflectance of the dyed alpaca yarn samples was measured using a Datacolor® 550 series spectrophotometer in the visible region (400–700 nm). In order to evaluated the chromatic characteristics (L^*, a^*, b^*, and C), measurements were taken under a D65 illuminant with a 10° observation angle. The color strength (K/S) values were calculated based on the Kubelka–Munk equation (1) as follows:

$$\frac{K}{S} = \frac{(1-R)^2}{2R} \quad (1)$$

where K is the absorption coefficient, S is the scattering coefficient, and R is the reflectance value of the dyed samples at maximum absorbance (λ_{max}) [27].

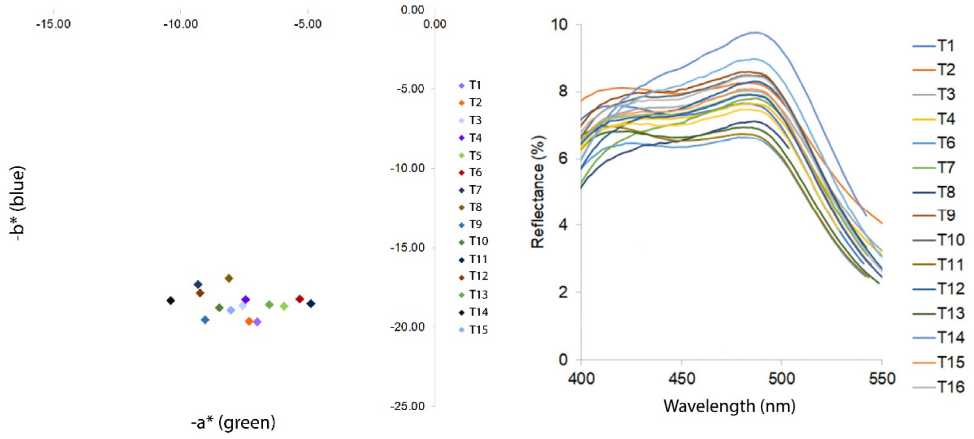
2.6. Fastness Testing

The color fastness to washing was determined according to the ISO 105-C06 (Reaffirmed 2022) Peruvian standard based on ISO 105-C06:1989 [3]. Wet and dry rub fastness tests were performed according to AATCC TM 8-2007 [28]. The change in the color of the specimens and staining of the adjacent fabric were evaluated using the ISO gray scale.

3. Results and Discussions

3.1. Color Analysis of Alpaca Yarns Dyed Using Indigo Carmine

According to the experimental results, the a*−b* plot (Figure 1a) clearly shows a predominant hue in all dyed samples. The color coordinates were positioned in the third quadrant of the CIE L*a*b* color space diagram, indicating that dyeing alpaca yarn samples with indigo carmine caused a particular blue shade. This is consistent with the spectral reflectance of the dyed samples, as shown in Figure 1b. In this context, the dyed samples have more reflectance in the wavelengths associated with the violet (~380–435 nm), indigo/blue (~436–480 nm), and greenish-blue (~491–500 nm) range, and begin to decrease noticeably in the green range (~500–560 nm). This suggests that the dyed samples absorbed green light and reflected blue light [27].



(**a**) (**b**)

Figure 1. Colorimetric properties: (**a**) CIELAB color coordinates; (**b**) spectral reflectance of alpaca yarn samples dyed using indigo carmine. Details of the assigned dyeing conditions are available in Supplementary Materials Table S2.

3.2. Model Fitting and Regression Analysis

In order to determine the effects of dyeing conditions (mordant concentration, dyeing temperature, and exhaust time) on the color strength (K/S) of dyed alpaca yarns, central composite design tests were performed. Therefore, in order to verify the model's adequacy, the experimental data were fitted to four potential models (linear, 2Fl, quadratic, and cubic), which are presented in Table 3.

Table 3. ANOVA results for the experimental results fitted to several models.

Source Model	Sequential *p*-Value	Lack of Fit (*p*-Value)	Adjusted R^2	Predicted R^2	
Linear	0.0401	0.0019	0.2828	−0.1122	
2Fl	<0.0001	0.1155	0.8854	0.8629	
Quadratic	0.0050	0.6293	0.9564	0.9072	Suggested
Cubic	0.8740	0.1933	0.9391	−0.3367	

According to the ANOVA fitting results analyzed using Design Expert® (v.13.0) software, the process of dyeing alpaca yarns using indigo carmine was most suitably described by a quadratic model, as indicated by the sequential *p*-value, the adjusted R^2, and the *p*-value of lack of fit. In regression analysis, the appropriate selection of a model that describes the relationship between variables is essential [29]. In this context, the quadratic model outperformed the two-factor interaction (2Fl) model based on statistical metrics. Firstly, the sequential *p*-value for the quadratic model was 0.0050, which is much lower than 0.05, indicating that adding the quadratic terms improved the model compared to the 2Fl model (<0.0001). The adjusted R^2 and predicted R^2 suggest that the quadratic model provided a better explanation of variability and had predictive capabilities [29,30]. Moreover, the probability of the lack-of-fit test was also relatively high (>0.05). This non-significant value indicates that the quadratic model did not have significant unexplained variation [31]. In contrast, the lack-of-fit *p*-value for the 2Fl model was 0.1155, which is closer to the significance threshold, suggesting a less reliable fit.

To verify the adequacy of the model selected, analysis of variance (ANOVA) was carried out as shown in Table 4, where the F-value and probability value (p-value < 0.05) were used as hypothesis values to indicate whether the factors were statistically significant or non-significant to the regression model [31,32].

Table 4. ANOVA of the alpaca dyeing process using indigo carmine.

Source	Sum of Squares	df	Mean Square	F-Value	*p*-Value	
Model	11.14	9	1.24	47.32	<0.0001	Significant
A: mordant concentration	0.2779	1	0.2779	10.62	0.0086	
B: dyeing temperature	1.48	1	1.48	56.55	<0.0001	
C: exhaust time	2.76	1	2.76	105.48	<0.0001	
AB	0.9555	1	0.9555	36.52	0.0001	
AC	0.0208	1	0.0208	0.7937	0.3939	
BC	5.02	1	5.02	191.75	<0.0001	
A^2	0.0306	1	0.0306	1.17	0.3051	
B^2	0.5680	1	0.5680	21.71	0.0009	
C^2	0.0093	1	0.0093	0.3572	0.5633	
Residual	0.2616	10	0.0262			
Lack of fit	0.1106	5	0.0221	0.7328	0.6293	Not significant
Pure error	0.1510	5	0.0302			
Cor total	11.40	19				

From Table 4, it can be observed that the mordant concentration (A), dyeing temperature (B), and exhaustion time (C) effects were statistically significant, since their *p*-values were less than 0.05. In the case of two-factor interaction effects, AB and BC were significant model terms. In contrast, the AC value interaction was much higher than the probability value (p-value < 0.05). The quadratic term B^2 reached the conventional level of statistical significance compared to A^2 and C^2 [31]. Likewise, the high F-values for the independent factors indicate that the predicted ones had a strong effect on the color strength (K/S) of alpaca yarns dyed with indigo carmine.

In order to enhance the model's predictive performance, a systematic model reduction approach was employed to eliminate non-significant terms [20]. As a result, the final quadratic model is expressed in Equation (2):

$$\frac{K}{S} = 52.73467 - 6.07815X_1 - 1.24086X_2 + 0.920311X_3 + 0.069120X_1X_2 - 0.010559X_2X_3 + 0.008016X_2^2 \quad (R^2 = 97.14\%; Adjusted\ R^2 = 95.82\%) \tag{2}$$

where X_1, X_2, and X_3 represent the mordant concentration, dyeing temperature, and exhaust time, respectively. Moreover, the positive and negative signs indicate the direction of the effect of each coefficient term on the response variable [19]. The coefficient of determination (R^2) reflects the model correlation degree where the larger the R^2, the better the correlation [32]. In this context, the R^2 of the model was 97.14%, which could explain the relationships between color strength (K/S) and mordant concentration (X_1), dyeing temperature (X_2) and exhaust time (X_3). The adjusted coefficient of determination (Adjusted R^2) was 95.82%, which indicates that nearly 96% of the variance in color strength (K/S) was explained by the model. This high value demonstrates that the model fits the observed data very well and could be used for prediction and interpretation. Finally, the lack of fit (F-value = 0.72) was not significant relative to the pure error. There is a 67.40% chance that a lack-of-fit F-value this large could occur due to noise. However, it is important to note that the predictive reliability of this model is confined to the experimental design space. Outside these bounds, the model may not account for additional nonlinear phenomena, such as dye thermal degradation at elevated temperatures, fixative saturation on the fiber surface, or shifts in dye–fiber interactions at high concentrations. These effects could introduce significant deviations that the current second-order terms are not structured to predict. Therefore, while the model is valid and robust within the defined experimental conditions, caution should be exercised when extrapolating its use to conditions beyond

this range. Future work may explore more complex modeling approaches or expanded experimental designs to capture these extended behaviors.

The normal probability distribution of the residual (errors between predicted and actual values) is plotted in Figure 2a. In this context, the data points were aligned closely to the red line, indicating that the quadratic model follows a normal distribution, which explained its behavior between the predicted and actual values [33]. A comparison of predicted and actual values is shown in Figure 2b. In this case, most data points were close to the line, which suggested that the model has good predictive accuracy; although there are some deviations (indicating slight errors), they do not appear large enough to compromise the validity of the model.

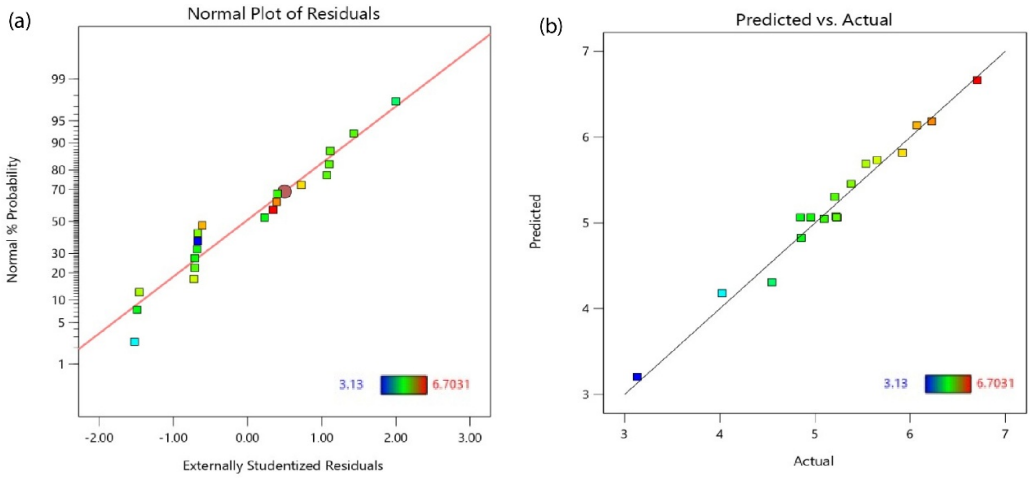
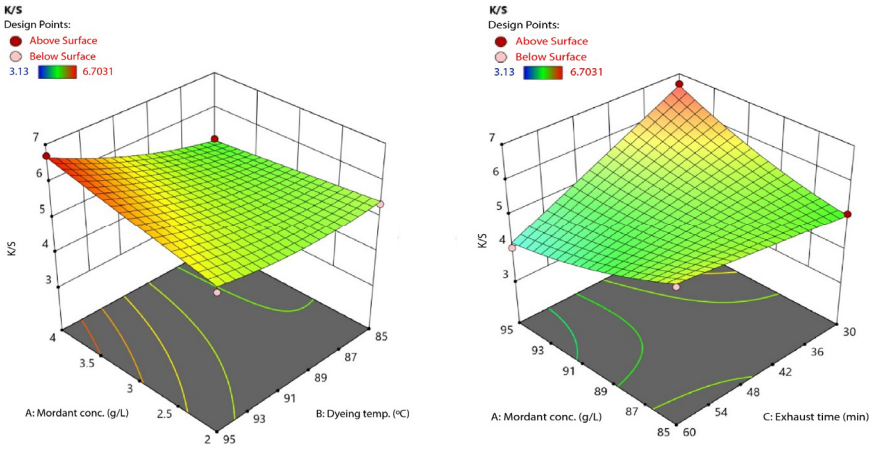


Figure 2. Validation of alpaca yarns dyed using indigo carmine. (**a**) Normal probability distribution plot; (**b**) predicted K/S values vs. actual values. The color gradient represents Studentized residuals, where cooler tones (blue–green) indicate lower residuals (better model fit), while warmer tones (yellow–red) indicate higher residuals or greater deviations from the model.

3.3. Effect of Dyeing Conditions on Color Strength (K/S)

To assess the influence of dyeing conditions on the color strength (K/S) of indigo carmine-dyed Alpaca yarns, a response surface methodology was employed in order to generate several diagnostic plots, where a green color represents lower K/S values and a red color represents a higher value [29]. Figure 3a,b presents a three-dimensional surface plot. Figure 3a shows the variation curves of the color strength (K/S) values with mordant concentration and dyeing temperature when the exhaust time was set to 30 min. In this context, it is evident that increments of both mordant concentration and dyeing temperature enhanced K/S values, suggesting a synergistic effect of these parameters on dye uptake. The reasons for this might be twofold. Firstly, the use of mordant enhances dye–fiber bonding by forming coordination complexes [7,22]. In this case, tartaric acid acts as not only an acidic mordant [22,23] but also a pH modifier, which could influence not only the interaction between the indigo carmine and alpaca fiber, but also the dyeing potential and color strength [34]. A higher concentration could enhance washing fastness properties, but there is a risk of excessive product accumulation on the surface when a high concentration is applied. This suggests that sufficient tartaric acid concentration ensures adequate protonation of biding sites on alpaca fiber, thereby promoting dye–fiber interaction [7]. Secondly, higher temperature exerts a more pronounced effect with a noticeable increase. This high temperature enhances dye diffusion and reaction kinetics [35], yet overly elevated temperatures risk partial dye degradation or altered fiber properties.



(**a**) (**b**)

Figure 3. Three-dimensional plots showing the effect of dyeing conditions on color strength (K/S). (**a**) Variation curves of color strength (K/S) with mordant concentration and dyeing temperature; (**b**) variation curves of color strength (K/S) with dyeing temperature and exhaust time.

Figure 3b shows the variation curves of the color strength (K/S) values with the dyeing temperature and exhaust time when the mordant concentration was set to 4 g/L. In this context, a progressive increase in K/S is observed that could be achieved not only at a prolonged exhaust time, but also at a shorter time if the temperature were sufficiently elevated. According to the topmost region of the surface plot, the higher temperature suggests that temperature exerts a strong influence on dye diffusion and fixation, reflecting the combined influence of thermal energy and duration on dye–fiber interactions. However, the pronounced peak at shorter times indicates that higher temperatures can accelerate dye uptake and promote the rapid formation of dye–fiber bonds [35,36].

3.4. Process Optimization Results and Analysis

To obtain a higher color strength (K/S), an optimization analysis was performed using Design Expert® (v.13.0) software to determine the best process for dyeing Alpaca fiber with indigo carmine. The process parameters were selected using the "the-large-the-better" method [19]. This approach was adopted because the K/S value is directly correlated with the dye molecule concentration and the dyed fiber's scattering value. A higher K/S value indicates higher absorption, which in turn indicates greater surface uptake [27]. Under this criterion, the software proposed the following optimum design point, shown in Table 5. In this, it is observed that desirability has a value very close to 1, i.e., 0.995, which indicates how well the combination of parameters impacts the response variable.

Table 5. Response optimization results: K/S value.

Mordant Concentration (g/L)	Dyeing Temperature (°C)	Exhaust Time (min)	Expected Value for K/S	Desirability
3.98873	95.000	30.0019	6.65839	0.995

To validate the optimization of the dyeing process, confirmatory experiments were repeated three times to ensure repeatability. Table 6 shows a comparison between the predicted and experimental values for the response variable (color strength, K/S).

In the table, a difference between the predicted mean and observed (data mean) value is observed. In this case, the observed value was lower than the predicted one with a

percentage difference of 0.3026%. This suggests strong agreement between experimental data and the prediction.

Table 6. Summary of results for optimization conditions.

Predicted Mean	Predicted Median	Std Dev	n	SE Pred [1]	95% PI Low	Data Mean	95% PI High
6.65839	6.65839	0.158383	3	0.146482	6.34193	6.63824	6.97484

[1] Standard error of prediction.

3.5. Color Fastness Properties of Alpaca Yarn Dyed Using Indigo Carmine

Table 7 presented the color fastness after rubbing and washing of alpaca yarn dyed using indigo carmine. The dyeing process was carried out according to the optimized recipe (Table 4) in order to find the color fastness properties.

Table 7. Summary of results for color fastness properties.

Fabric	Rubbing Fastness		Washing Fastness						Color Change
	Dry	Wet	CA	CO	PA	PES	PAN	WO	
Alpaca fiber	3	3/4	5	4/5	5	5	4/5	5	4/5

Standard textile fiber codes: CA = Acetate, CO = Cotton, PA = Polyamide (Nylon), PES = Polyester, PAN = Acrylic, and WO = Wool.

The rubbing and washing fastness results were evaluated according to the gray scale. In this context, it can be seen that wet and dry rubbing fastness achieved 3 and 3/4 ratings, respectively, denoting tolerable color fastness. It can be inferred that the dyeing process using indigo carmine withstands rubbing conditions, since it did not present significant color transfer to the cotton witness, indicating good fixation of the dye molecules on the fiber [25]. For washing fastness results using cotton (CO) and polyacrylic (PAN), the alpaca fiber achieved a 4/5 rating. In the case of other adjacent fibers, it achieved a rating of 5. This suggests a favorable interaction between the dye molecules and the alpaca fiber surface. In addition, these findings are relevant for selecting materials to produce blended yarn fibers [25].

4. Conclusions

Indigo carmine is an outstanding dye for alpaca fiber. Response surface methodology (RSM) was used to enhance the dyeing conditions for color strength (K/S) in dyed alpaca yarns. This value was raised by mordant concentration, dyeing temperature, and exhaust time, where higher color strength values were the results of higher mordant concentration and dyeing temperature with a lower exhaust time. Fastness properties for both washing and rubbing tests were tolerable, showing good color permanence and textile quality. In summary, this approach not only enhances the efficiency of the dyeing process but also presents significant findings for industrial-scale application for potential resource savings in textile production.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/fib13060082/s1, Table S1: Comparative dyeing performance and fastness properties of Alpaca yarns dyed with Indigo Carmine under different mordanting conditions; Table S2: Assignments dyeing condition of the Alpaca dyeing process using Indigo carmine.

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investigation, E.G.-H., C.M.L.-J., E.M.d.J. and J.C.B.; data curation, C.M.L.-J. and J.C.B.; writing—original draft preparation, C.M.L.-J. and E.M.d.J.; writing—review and editing, C.M.L.-J., E.M.d.J. and E.G.-H.; visualization, C.M.L.-J. and E.M.d.J.; supervision, E.G.-H. and C.M.L.-J.; project administration, C.M.L.-J. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest: The authors of this article declare that they have no conflict of interest that could inappropriately influence their work. All materials, supplies, and all sources of information are properly referenced. The authors have no financial, personal or contractual relationship with other individuals or organizations that may present a conflict of interest. The funders had no role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript; or in the decision to publish the results.

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