UMYU Scientifica, Vol. 5 NO. 1, March 2026, Pp 370 – 381
https://doi.org/10.56919/usci.2651.031
ISSN: 2955 – 1145 (print); 2955 – 1153 (online)
A periodical of the Faculty of Natural and Applied Sciences, UMYU, Katsina

ORIGINAL RESEARCH ARTICLE

Optimization of extraction and dyeing conditions of *Ixora coccinea* on cotton

Khadija Sufyan Yahaya[1], Sani Muhammad Gumel[1], Dini Sabo[1], Shehu Habibu[1]*, and Ahmad Ado Usman[1]

[1]Department of Pure and Industrial Chemistry, Bayero University Kano, PMB 3011, Kano, Nigeria

ARTICLE HISTORY

Received November 12, 2025
Accepted March 23, 2026
Published March 27, 2026

KEYWORDS

Ixora coccinea; natural dye, cotton fabric, extraction optimization, dyeing optimization, response surface methodology (RSM), Box–Behnken design, mordanting, color fastness, sustainable textiles.

ABSTRACT

This study focuses on optimizing the extraction and dyeing conditions to standardize the procedure for the natural colorant derived from the flower of *Ixora coccinea*. Utilizing Response Surface Methodology, specifically the Box-Behnken Design (BBD), we systematically investigated the process parameters for both extraction and dyeing. The colorant obtained under optimized extraction conditions was characterized using FTIR and UV-Visible spectroscopy. It was then applied to cotton fabric to examine the effects of time, pH, and mordant concentration. Analysis of variance (ANOVA) revealed that the quadratic model significantly contributed to the extracted yield and percentage exhaustion, as illustrated by the 3D response surfaces. Optimal extraction conditions were determined to be 90 minutes of extraction time, a solvent-to-material ratio of 1:20 g/mL, and a pH of 7, resulting in a dye yield of 49 %. The optimized dyeing conditions included a mordant concentration of 4.5 % ow, a dyeing time of 30 minutes, and a pH of 7, achieving a maximum percentage exhaustion of 26.99 %. Under these optimal dyeing conditions, cotton fibers dyed with the *Ixora coccinea* extract exhibited excellent color fastness properties: a light fastness rating of grade 6, a washing fastness rating of grade 4, and outstanding ironing fastness (grades 5 for dry, wet, and damp conditions) with negligible staining of adjacent white samples.

INTRODUCTION

Natural dyes have attracted renewed scientific and industrial interest as sustainable alternatives to synthetic colorants due to environmental and health concerns associated with conventional dyes, including toxicity, non-biodegradability, and effluent pollution. Derived from plant, animal, and mineral sources, natural colorants offer advantages such as biodegradability, low toxicity, and aesthetic appeal, making them attractive for eco-textile applications. However, despite these benefits, the application of natural dyes on cellulosic fibers such as cotton remains limited by low color yield, poor reproducibility, inconsistent shade development, and inadequate fastness properties. These limitations highlight the need for systematic and statistically guided optimization of both extraction and dyeing processes to achieve commercially viable and reproducible results (Aggarwal and Technology, 2025; Islam *et al.*, 2024).

Ixora coccinea L. (Rubiaceae), a flowering shrub widely distributed in tropical regions, contains anthocyanins and other pigment compounds that can be extracted and applied as natural dyes. Previous studies demonstrate that floral extracts from *Ixora coccinea* can effectively impart colour to cotton fabrics, with dye uptake and shade variation influenced by solvent type, mordants, and dyeing conditions (Dey *et al.*, 2025; Hamdy *et al.*, 2021; Islam *et al.*, 2025). The use of ecological mordants and optimized process conditions has shown improved colour fastness properties, highlighting the potential of *I. coccinea* as a viable natural dye source for sustainable textile coloration (Che and Yang, 2022). However, these studies are largely limited to isolated optimization of either extraction or dyeing parameters, and often lack comprehensive statistical validation, reproducibility assessment, and integrated performance evaluation. Although Response Surface Methodology (RSM) has been widely applied in optimizing natural dye extraction and application processes, existing studies predominantly focus on single-stage optimization or generalized dye systems. Furthermore, many reported models lack complete validation metrics such as predicted R^2, adequate precision, and lack-of-fit analysis, which are essential for confirming model reliability and predictive capability (Aftab *et al.*, 2024; Pervez *et al.*, 2023; Rosa *et al.*, 2021) .

Accordingly, this study aims to optimize the extraction and dyeing conditions of *Ixora coccinea* floral dye on cotton fabric using RSM (Box–Behnken Design), characterize the extracted colorant using spectroscopic techniques, and systematically evaluate the effects of time, pH, and mordant concentration on dyeing performance and color fastness (Mahfud; Nor, 2019).

Correspondence: Shehu Habibu. Department of Pure and Industrial Chemistry, Bayero University Kano, PMB 3011, Kano, Nigeria. ✉ shabibu.chm@buk.edu.ng.

How to cite: Sufyan, K. Y., Muhammad, S. G., Sabo, D., Habibu, S., & Ado, A. U. (2026). Optimization of extraction and dyeing conditions of *Ixora coccinea* on cotton. *UMYU Scientifica*, 5(1), 370 – 381. https://doi.org/10.56919/usci.2651.031

MATERIALS AND METHOD

Materials

Fresh flowers of *Ixora coccinea* were randomly collected from the vicinity of Bayero University, Kano, Nigeria, and taxonomically authenticated at the Department of Plant Science, Bayero University Kano. The collected flowers were thoroughly washed with distilled water to remove adhering impurities, air-dried at room temperature, and subsequently ground into a fine powder. A 100% cotton fabric (12-ply) was used as the substrate for dyeing experiments. Analytical characterization of the extracted dye was carried out using a UV–Visible spectrophotometer (CE-7315) and a Fourier Transform Infrared (FTIR) spectrometer (Cary-630, Agilent Technologies).

Methods

Extraction of natural dye from Ixora coccinea flowers

Fresh *Ixora coccinea* flowers were washed, air-dried at room temperature (25 ± 2 °C) for 72 h, and further oven-dried at 50 °C until constant weight was achieved to ensure moisture consistency. The dried samples were ground into fine powder and stored in airtight containers prior to use.

Aqueous extraction was performed in closed conical flasks placed on a thermostatically controlled hot plate with magnetic stirring at 300 rpm. The extraction temperature was maintained at 80 ± 2 °C. The investigated parameters included extraction time (30–90 min), pH (3–7), and solvent-to-material ratio (1:10–1:30 mL g^{-1}). All experiments were conducted in triplicate (n = 3) to ensure reproducibility, and mean values ± standard deviation were reported (Guddi *et al.*, 2024a).

Optimization of dye extraction from *Ixora coccinea* flowers

For each experimental run, 1 g of powdered sample was mixed with the required volume of distilled water. The pH was adjusted using 1 M HCl or 1 M NaOH and verified using a calibrated digital pH meter. After extraction, the mixture was filtered using Whatman No. 1 filter paper.

The filtrate was dried in a hot-air oven at 60 °C until constant weight was achieved. Experimental errors were minimized by maintaining identical drying time and environmental conditions across all runs (Guddi *et al.*, 2024a; Samant and Gaikwad, 2020).

the percentage dye yield was calculated using Equation (1).

$$\%\ Yield = \frac{W_{f-Wi}}{W_p} \quad \text{..... (1)}$$

where W_p is the initial weight of the raw plant material, W_i is the weight of the empty evaporating dish, and W_f is the final weight of the evaporating dish containing the dried dye extract.

Experimental design for optimization of the extraction process

Response Surface Methodology (RSM) was employed to determine the optimum extraction conditions that maximize dye yield while minimizing the number of experimental trials. Using Design-Expert software (version 10.0.3), a Box–Behnken Design (BBD) was selected as the experimental design. The percentage dye yield was considered as the response variable, while extraction time, solvent-to-material ratio, and pH were chosen as the independent process variables (Le et al., 2026).

The BBD generated 17 experimental runs consisting of different combinations of the selected variables. The experimental data obtained were fitted to a quadratic regression model, and analysis of variance (ANOVA) was performed to evaluate the significance and adequacy of the model. The suitability of the developed model was assessed based on statistical parameters, particularly the p-values, which were used to determine the significance of individual factors and their interactions (Boukhatem et al., 2026; Burbano-Pulles et al., 2026).

Box–Behnken experimental design for optimization of dye extraction

Table 1 presents the Box–Behnken Design (BBD) matrix used to optimize the extraction of natural dye from *Ixora coccinea* flowers. The experimental design consists of 17 runs with different combinations of three independent variables: extraction time (Factor A, min), solvent-to-material ratio (Factor B), and pH (Factor C). The percentage dye yield was selected as the response parameter to evaluate the effectiveness of each experimental condition.

Fourier transform infrared (FTIR) spectroscopic analysis

Fourier Transform Infrared (FTIR) spectroscopy was employed to identify the functional groups present in the *Ixora coccinea* dye extract. The analysis was carried out using an IR spectrometer (Cary-630, Agilent Technologies) with a spectral resolution of 8 cm^{-1} over the wavenumber range of 4000–650 cm^{-1}. A small quantity of the dried dye extract was placed directly in the infrared beam for analysis. As the infrared radiation passed through the sample, the transmitted energy was recorded and used to generate the FTIR spectrum, enabling the identification of characteristic functional groups within the extract(Manikandan and Nagarasampatti Palani, 2025).

Ultraviolet–Visible (UV–Vis) spectroscopic analysis

Ultraviolet–Visible (UV–Vis) spectroscopy was used to characterize the optical properties of the *Ixora coccinea* dye extract. For the analysis, 1 mL of the extract was diluted to 100 mL with distilled water. An aliquot of the diluted solution was transferred into a quartz cuvette and analyzed using a UV–Vis spectrophotometer (CE-7315). The absorption spectrum was recorded over the wavelength range of 200–700 nm to determine the characteristic

absorption bands and the maximum absorption wavelength (λ_{max}) of the extract .Calibration curves were prepared using standard dilutions to ensure linearity ($R^2 \geq 0.99$). (Lakshmi S *et al.*, 2025).

Table 1: Box–Behnken design matrix for dye extraction

Run	Factor A: Time (min)	Factor B: Solvent to material ratio	Factor C: pH	Response Yield %
1	60	20	5	
2	60	30	7	
3	90	20	7	
4	30	20	3	
5	60	20	5	
6	60	20	5	
7	60	10	3	
8	30	10	5	
9	60	30	3	
10	90	10	5	
11	30	20	7	
12	60	10	7	
13	60	20	5	
14	60	20	5	
15	90	20	3	
16	30	30	5	
17	30	30	5	

This design enabled efficient evaluation of the individual and interactive effects of the selected extraction parameters on dye yield.

Table 2: Experimental design matrix and dye exhaustion Results

Std	Run	Factor 1 A. Mordant con. %ow	Factor 2 B. Time min	Factor 3 C. pH	Response 1 exhaustion E %
7	1	1	37.5	7	
11	2	4.5	30	7	
16	3	4.5	37.5	6	
1	5	1	30	6	
5	6	1	37.5	5	
12	7	4.5	45	7	
13	8	4.5	37.5	6	
4	9	8	45	6	
10	10	4.5	45	5	
6	11	8	37.5	5	
9	12	4.5	30	5	
8	13	8	37.5	7	
15	14	4.5	37.5	6	
3	15	1	45	6	
17	16	4.5	37.5	6	
2	17	8	30	6	

This design enabled systematic evaluation of the effects and interactions of dyeing parameters on exhaustion efficiency and supported the identification of optimal dyeing conditions.

Pre-treatment of cotton fiber

Scouring and bleaching of cotton fiber

Scouring and bleaching of the cotton fiber were carried out prior to dyeing to remove natural impurities such as fats, waxes, and residual coloring matter, thereby improving the whiteness, wettability, and dye absorbency of the fabric. Both processes were performed in a single combined stage following established procedures (Mozumder and Majumder, 2016).

The treatment bath contained detergent (1 gL^{-1}), sodium hydroxide (3 gL^{-1}), sodium peroxide (4 gL^{-1}), peroxide stabilizer (1 gL^{-1}), and sequestering agent (citric acid, 0.7 gL^{-1}). A cotton fabric sample weighing 30 g was treated at a material-to-liquor ratio of 1:10, adjusted to pH 10.7, and processed at a temperature of 100 °C for 60 minutes. After treatment, the fabric was thoroughly rinsed and prepared for subsequent dyeing experiments(Hannan *et al.*, 2019).

Mercerization of cotton fiber

Mercerization of the cotton fabric was carried out to enhance dye affinity, absorbency, and dyeing performance. The process involved treating the cotton fabric with a 25% (w/v) sodium hydroxide (caustic soda) solution for 2 minutes at room temperature. Following treatment, the fabric was thoroughly washed and rinsed with water to completely remove residual alkali, and then air-dried. This treatment is known to improve the accessibility of hydroxyl groups in cotton, thereby

significantly increasing dye uptake and uniformity (Lin *et al.*, 2022).

Dyeing and mordanting of cotton fabric

Pre-mordanting was employed prior to dyeing to improve the fixation and color strength of the *Ixora coccinea* natural dye on cotton fabric. The pre-treated cotton samples were mordanted using varying concentrations of alum (potassium aluminum sulfate) in accordance with the experimental design (Moniruzzaman *et al.*, 2018).

Dyeing optimization was carried out using 5 % (owf) of *Ixora coccinea* natural dye extract at a material-to-liquor ratio of 1:30. The dyeing process was performed at a temperature of 70 °C for 30 minutes, following established dyeing procedures for natural dyes(Darmawan *et al.*, 2024).

Optimization of dyeing conditions using response surface methodology

Design-Expert software (version 10.0.3) was employed for experimental design and statistical analysis of the dyeing process. Response Surface Methodology (RSM) was applied to optimize the key operating variables influencing dye uptake. The selected independent variables were mordant concentration (1–8%), dyeing time (30–45 min), and pH (5–7). The effect of these variables on the percentage exhaustion of the dye was investigated. Each independent variable was coded at two levels, low (–1) and high (+1), according to the experimental design.

The adequacy and significance of the developed model were evaluated based on p-values obtained from statistical analysis. Analysis of variance (ANOVA) was performed using Design-Expert software to assess the significance of individual model terms and their interactions. The quality of the fitted model was determined using the coefficient of determination (R^2) and the adequate precision ratio, which indicates the signal-to-noise ratio. Three-dimensional response surface plots were generated to visualize the effects of the process variables and their interactions on dye exhaustion (Rosa *et al.*, 2021).

Experimental design for optimization of dyeing conditions

The experimental design for the dyeing process comprised 17 experimental runs, which were carried out according to the design matrix presented in Table 2. The experiments were structured using Response Surface Methodology to evaluate the combined and individual effects of the selected process variables on dyeing performance. The percentage dye exhaustion (E%) was chosen as the response parameter and was analyzed as a function of the independent variables.

The three input factors considered were mordant concentration (% owf), dyeing time (min), and pH of the dye bath. Based on the experimental data obtained, a second-order (quadratic) polynomial equation was developed to describe the relationship between the response and the process variables. This model was subsequently used to predict and optimize the dyeing conditions (Rosa *et al.*, 2021).

Measurement of dye exhaustion using UV–Vis spectroscopy

The percentage of dye exhaustion indicates the amount of dye absorbed by the fiber from the dye bath during the dyeing process. This was determined using a UV–Visible spectrophotometer (CE-7315) at the maximum absorption wavelength (λmax), with a quartz cuvette. The absorbance of the dye solution was measured before and after dyeing to evaluate the extent of dye uptake by the fabric. The percentage of dye exhaustion (E%) was calculated using the equation (Jain *et al.*, 2026).

$$E_{\%} = \frac{C_1 - C_2}{C_1} \times 100 \quad \text{.....(2).(Jain } et\ al.\text{, 2026)}$$

Where C_1 = initial concentration of the dye in the bath (before dyeing), C_2 = concentration of the dye in the bath after dyeing

The concentrations were measured using a UV–Vis spectrophotometer over a wavelength range of 200–700 nm (Alves *et al.*, 2022) . This method allows quantitative determination of dye transfer efficiency from the bath to the fiber.

Evaluation of color fastness properties

The color fastness properties of the dyed cotton fabrics were assessed in accordance with established standard testing methods. The evaluated fastness parameters included resistance to light, washing, and pressing.

Light fastness

Light fastness was determined following the standard method BS 1006: B01 (1978). The test was carried out using an artificial light fastness tester (MK 1) equipped with a 500 W mercury–tungsten (MBTF) lamp. Dyed fabric samples measuring 5 × 4 cm were exposed to light for 96 hours alongside eight blue wool reference standards. The degree of fading of the samples was visually compared with the blue wool standards to assign the light fastness rating (Abdullahi *et al.*, 2021).

Wash fastness

Wash fastness was evaluated using a standard method. Dyed fabric samples of 5 × 4 cm were placed between two pieces of undyed cotton fabric of equal dimensions to form a composite specimen, which was then stitched together. The specimen was immersed in 100 cm^3 of washing liquor containing 4 g dm^{-3} detergent solution and agitated for 30 minutes at 50 °C. After washing, the samples were thoroughly rinsed, air-dried, and the degree of staining on the adjacent undyed fabric was assessed using the grey scale (Shariful Islam *et al.*, 2020).

Pressing fastness

Fastness to pressing under dry, damp, and wet conditions was assessed in accordance with ISO 105-P01:1993. Dyed samples measuring 5 × 4 cm were sandwiched between

two pieces of dry white cotton fabric to form a composite specimen. A heated pressing iron was applied for 15 seconds, after which the degree of color transfer to the adjacent fabric was evaluated using the grey scale (Uzumcu *et al.*, 2021).

These tests collectively provide a comprehensive evaluation of the durability and performance of the dyed fabrics under typical use and care conditions.

RESULTS AND DISCUSSION

Optimization of the extraction process

Response Surface Methodology (RSM) was employed as a mathematical optimization tool to determine the optimum extraction conditions within the defined ranges of the process variables. The optimization was performed by evaluating the combined effects of the control factors on the response, while ensuring that all calculated responses remained within the experimental limits of the selected parameters.In this study, the optimum extraction conditions were identified as pH 7, solvent-to-material ratio of 1:20 mL g^{-1}, and extraction time of 90 min, under which a maximum experimental dye yield of 49% was achieved.

The experimental runs were designed according to the Box–Behnken Design (BBD), and the corresponding extraction yields are presented in Table 3. A total of 17 experiments were conducted with different combinations of the three independent variables: extraction time (A), solvent-to-material ratio (B), and pH (C).

Table 3: Box–Behnken experimental design and extraction yield

Run	Factor A: Time (min)	Factor B: Solvent to material ratio	Factor C: pH	Response Yield %
1	60	20	5	33
2	60	30	7	48
3	90	20	7	49
4	30	20	3	14
5	60	20	5	32
6	60	20	5	32
7	60	10	3	16
8	30	10	5	30
9	60	30	3	17
10	90	10	5	35
11	30	20	7	46
12	60	10	7	48
13	60	20	5	33
14	60	20	5	34
15	90	20	3	18
16	30	30	5	28
17	30	30	5	34

Table 4: ANOVA for response quadratic model

Variables	Sum of Squares	Df	Mean Square	F Value	P Value Prob > F	
Model	2029.67	9	225.52	232.15	< 0.0001	significant
A	40.50	1	40.50	41.69	0.0003	
B	0.50	1	0.50	0.51	0.4963	
C	1984.50	1	1984.50	2042.87	> 0.0001	
AB	0.25	1	0.25	0.26	0.6275	
AC	0.25	1	0.25	0.26	0.6275	
BC	0.25	1	0.25	0.26	0.6275	
A^2	2.53	1	2.53	2.60	0.1507	
B^2	0.32	1	0.32	0.33	0.5849	
C^2	0.32	1	0.32	0.33	0.5849	
Residual	6.8	7	0.97			
Lack of fit	4.00	3	1.33	1.90	0.2702	not significant
Pure error	2.80	4	0.70			
Cor total	2036.47	16				

The experimental data were analyzed using multiple regression analysis to develop a second-order quadratic model, which describes the relationship between the independent variables and the extraction yield. This model effectively explains the influence and interaction of the process parameters on the response, in agreement with previous studies (Nekkaa *et al.*, 2021) .

$$\%Y=32.80 + 2.25\mathbf{A} + (-0.25\mathbf{B}) + 15.75C + 0.25\mathbf{AB} + (-0.25\mathbf{AC}) + (-0.25\mathbf{BC}) + (-0.78\mathbf{A^2}) + (0.28\mathbf{B^2}) + (-0.28\mathbf{C^2}) \quad (3)$$

Y = extraction yield, A = extraction time (minutes), B = solvent to material ratio ml/g C = pH

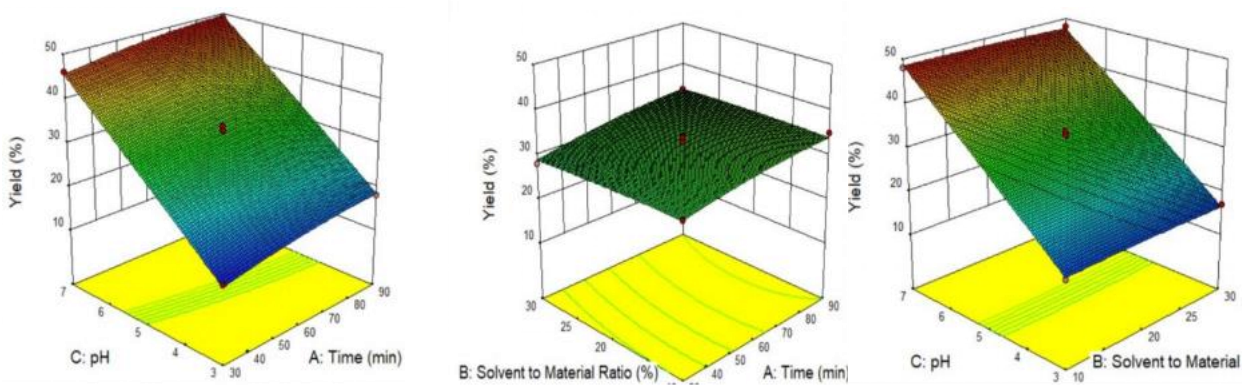
The regression equation expressed in terms of coded variables can be used to predict the response at specified

levels of each factor. In this coding system, the high and low levels of the variables are represented by +1 and −1, respectively. The coded form of the equation is particularly useful for evaluating the relative influence of each factor, as the magnitude of the coefficients allows direct comparison of their effects on the response.

The statistical parameters of the model indicate a high level of reliability and accuracy, with a standard deviation of 0.99 and a mean response value of 32.18. The coefficient of determination (R^2) was 0.9967, while the adjusted R^2 and predicted R^2 were 0.9924 and 0.9964, respectively, demonstrating excellent agreement between the experimental and predicted values. The adequate precision value of 47.624 indicates a strong signal-to-noise ratio, and the low coefficient of variation (CV) of 3.08% confirms the good reproducibility of the experimental data.

The adequacy of the developed model was evaluated using analysis of variance (ANOVA). As shown in Table 4, the high F-value of 232.15 confirms that the model is statistically significant, with only a 0.01% probability that such a value could arise from random noise. The *Prob* > *F* value (< 0.0500) further indicates the significance of the model terms, with factors A and C identified as significant contributors to the response. Model terms with *Prob* > *F* values greater than 0.1000 were considered insignificant.

The lack-of-fit F-value of 1.90 was not significant relative to the pure error, which is desirable and indicates that the model adequately fits the experimental data. The coefficient of variation (CV) of 3.06% demonstrates good precision and reproducibility, as values below 10% indicate a reliable model. Model fitness was further confirmed by the high coefficient of determination (R^2 = 0.9967), showing excellent agreement between experimental and predicted results. In addition, the predicted R^2 (0.9964) was in reasonable agreement with the adjusted R^2 (0.9924), indicating a strong correlation between predicted and actual values. The adequate precision value of 47.624, well above the recommended threshold of 4, signifies a satisfactory signal-to-noise ratio, confirming that the model can be effectively used to navigate the design space. These findings are consistent with previous reports (Stephanie Martins de Freitas *et al.*, 2021).



(A) (B) (C)

Figure 1: Three-dimensional response surface plot illustrating the effect of extraction parameters on the extraction yield.

Table 5: Experimental design for dyeing and corresponding percentage exhaustion

Run	Factor 1A. Mordant con. %ow	Factor 2B. Time min	Factor 3C. pH	Response 1 exhaustion E %
1	1	37.5	7	3.7
2	4.5	30	7	26.98
3	4.5	37.5	6	26.43
5	1	30	6	3.44
6	1	37.5	5	2.68
7	4.5	45	7	26.45
8	4.5	37.5	6	26.98
9	8	45	6	12.09
10	4.5	45	5	25
11	8	37.5	5	14.57
12	4.5	30	5	26.98
13	8	37.5	7	12.9
14	4.5	37.5	6	26.43
15	1	45	6	1.05
16	4.5	37.5	6	26.43
17	8	30	6	11.799

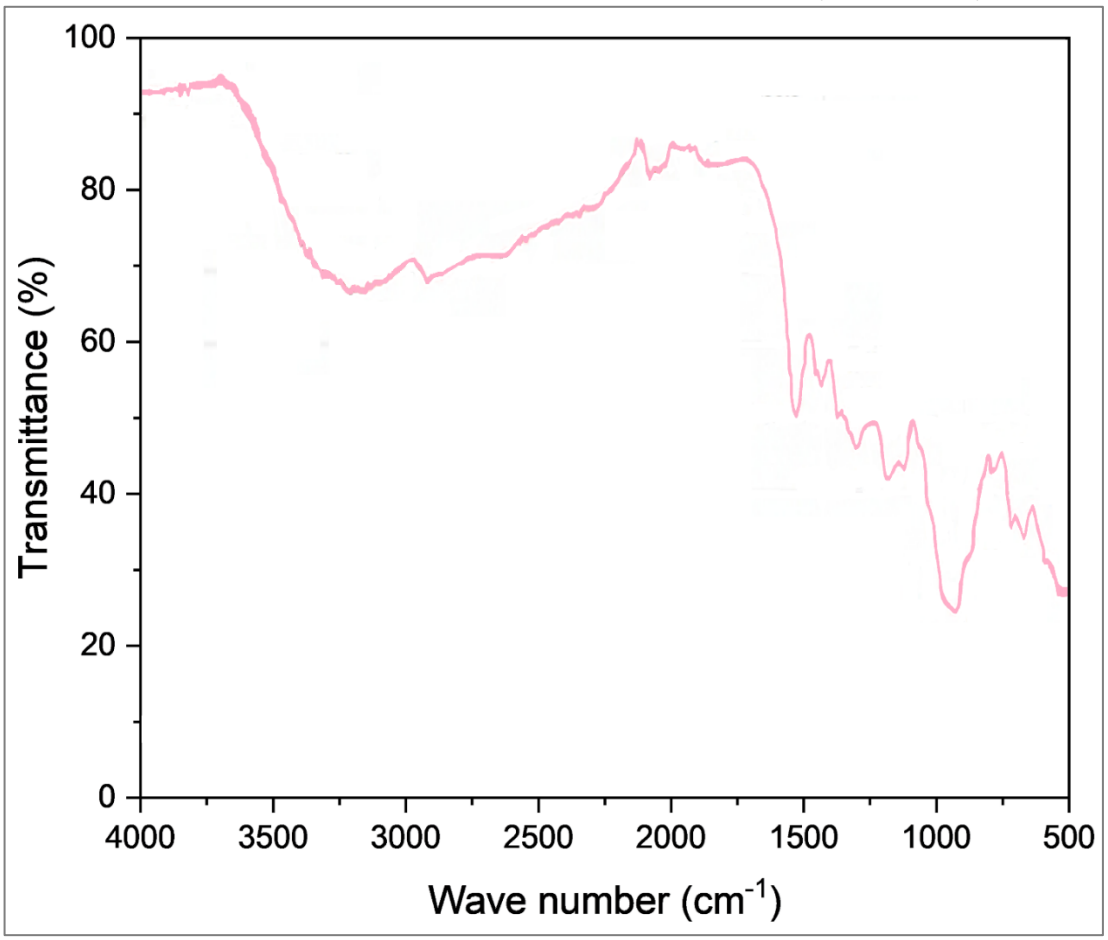


Figure 2: FTIR spectrum of the extracted dye.

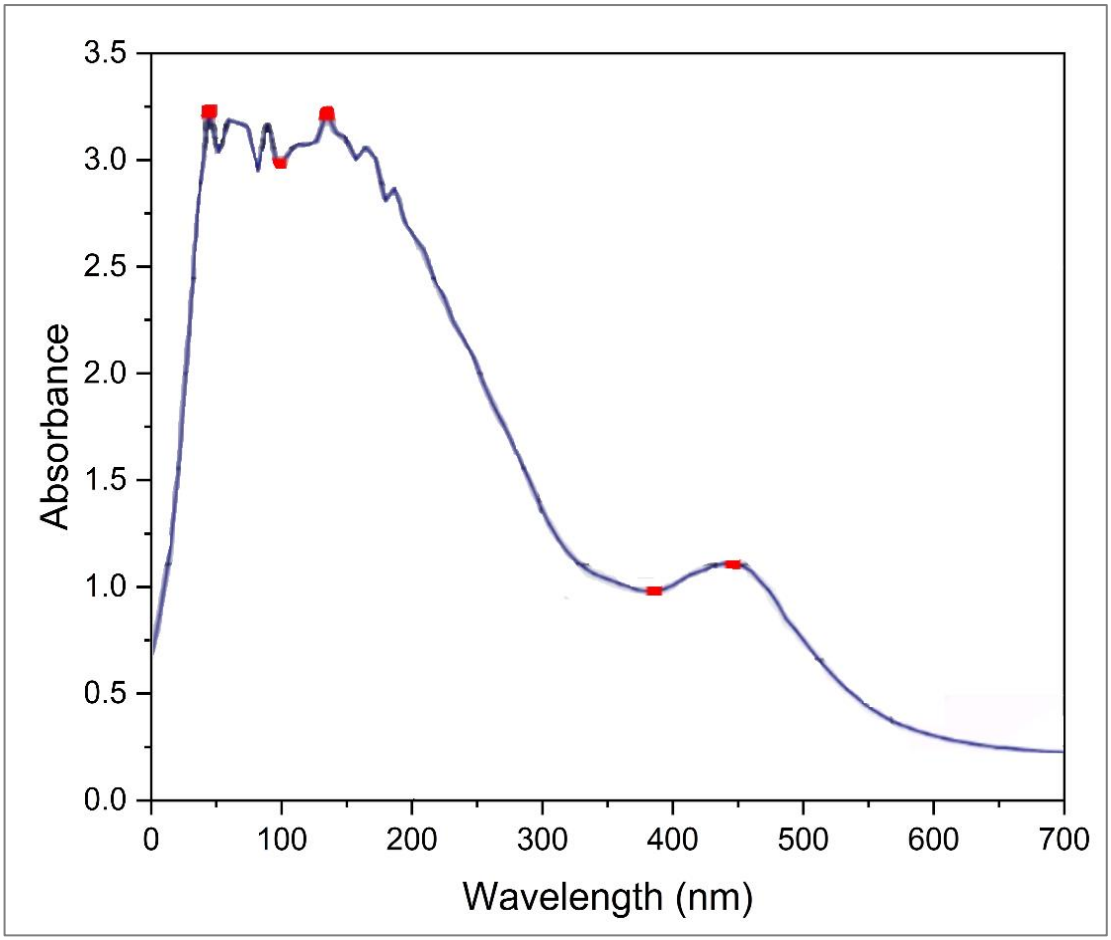


Figure 3: UV–Visible absorption spectrum of the *Ixora coccinea* dye extract.

Table 6: ANOVA results for the quadratic model of dye exhaustion (%)

Variables	Sum of Squares	Df	Mean Square	F Value	P Value Prob > F
Model	1690.51	9	187.83	1075.27	<0.0001significant
A- mordant	204.92	1	204.92	1173.08	< 0.0001
B- time	2.66	1	2.66	15.20	0.0059
C- pH	0.080	1	0.080	0.46	0.5203
AB	1.80	1	1.80	10.29	0.0149
AC	1.81	1	1.81	10.36	0.0147
BC	0.53	1	0.53	3.01	0.1264
A^2	1464.93	1	1464.93	8386.08	< 0.0001
B^2	2.45	1	2.45	14.02	0.0072
C^2	1.54	1	1.54	8.83	0.0208
Residual	1.22	7	0.17		
Lack of fit	0.93	3	0.31	4.23	0.0986 not significant
Pure error	0.29	4	0.073		
Cor Total	1691.73	16			

Table 7: Color fastness evaluation of cotton fabric dyed with *Ixora coccinea* extract

S/N	Light fastness	Washing fastness	Ironing fastness		
			Dry	Wet	Damp
1	6	4	5	5	5
2	5	3	5	5	5
3	6	4	5	5	5
4	6	4	5	5	5
5	6	4	5	5	5
6	5	4	5	5	5
7	6	4	5	5	5
8	6	3	5	5	5
9	5	3	5	5	5
10	6	3	5	5	5
11	5	3	5	5	5
12	5	4	5	5	5
13	5	4	5	5	5
14	6	4	5	5	5
15	5	4	5	5	5
16	6	4	5	5	5
17	6	4	5	5	5

Figure 1 illustrates the three-dimensional response surface plots showing the combined effects of (A) extraction time and pH, (B) extraction time and solvent-to-material ratio, and (C) solvent-to-material ratio and pH on the percentage dye yield. The 3D response surface plots provide a clear visualization of the interactions between the process variables and the response. It is evident from the plots that the extraction yield of *Ixora coccinea* increases with increasing extraction time and pH. This observation is in agreement with the ANOVA results, which indicate that factors A (time) and C (pH) are statistically significant ($p < 0.05$), whereas factor B (solvent-to-material ratio) shows no significant influence on the extraction yield. Figure 1 further demonstrates the progressive increase in dye yield as time and pH increase throughout the extraction process. These findings are consistent with those reported by (Bras *et al.*, 2020) .

Fourier transform infrared (FTIR) spectroscopy of the extracted dye

FTIR spectroscopy was performed to characterize the chemical composition and identify the functional groups present in the extracted *Ixora coccinea* dye. This analysis provides insights into the molecular structure of the natural dye, including the presence of hydroxyl, carbonyl, aromatic, and other characteristic groups, which are important for understanding its dyeing behavior and interactions with textile fibers.

The FTIR spectrum of the *Ixora coccinea* dye extract (Figure 2) reveals the presence of several characteristic functional groups. The broad absorption band at approximately 3480 cm^{-1} corresponds to O–H stretching vibrations, while the peak at 2918 cm^{-1} is attributed to C–H stretching, in agreement with the observations of (Munene *et al.*, 2020) . The absorption band at 1205 cm^{-1} falls within the range of 1000–1300 cm^{-1} and is associated with C–O stretching, indicating the presence of phenolic compounds. The prominent peak at around 1719 cm^{-1} is assigned to C=O stretching vibrations, suggesting the presence of carbonyl functional groups. Additionally, the band observed at 1598 cm^{-1} corresponds to C=C stretching vibrations, while the peak at approximately 702 cm^{-1} is characteristic of aromatic C–H bending. These functional groups

confirm the complex chemical nature of the extracted natural dye, consistent with earlier reports (Mamand *et al.*, 2025).

Ultraviolet–Visible (UV–Vis) analysis of *Ixora coccinea* extract

The UV–Visible spectroscopy analysis was conducted to investigate the optical properties and characteristic absorption behavior of the *Ixora coccinea* dye extract. This technique provides information on the electronic transitions within the dye molecules, which can be correlated with the presence of chromophoric groups and overall color characteristics. The absorption spectrum helps in identifying the maximum wavelength (λ_{max}) of the extract, which is essential for understanding its potential application as a natural dye in textile dyeing.

The UV–Visible spectrum of the *Ixora coccinea* dye extract (Figure 3) exhibits absorption bands at 230, 265, and 328 nm, along with two prominent bands in the visible region at 465 and 510 nm. The bands at 230 and 265 nm are attributed to phenolic compounds, while the band at 328 nm corresponds to the presence of flavonoids. The strong absorption in the visible region at 465 and 510 nm is indicative of anthocyanins, which are responsible for the characteristic coloration of this biologically derived dye. UV–Vis spectroscopy thus serves as a valuable tool for identifying phenolic and flavonoid constituents in natural dyes, consistent with previous findings reported by (Nistor and Butnariu, 2023).

Optimization of dyeing process

Experimental design for dye exhaustion

The optimization of the dyeing process was performed by evaluating different combinations of factor levels to achieve the maximum response while satisfying the constraints for each variable. The Box–Behnken Design (BBD) was used to define the experimental factors and their levels mordant concentration, dyeing time, and pH across 17 experimental runs, as presented in Table 5.

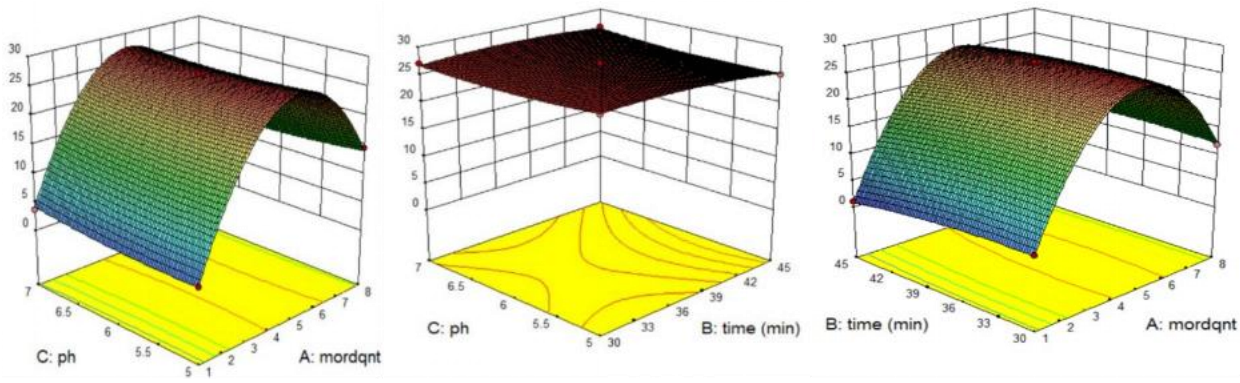
A full quadratic response surface model was developed using the experimental data for the response variable, percentage dye exhaustion (%E). The maximum dye exhaustion of 26.98% was obtained under the optimized conditions: 4.5 % owf mordant concentration, 30 minutes dyeing time, and pH 7. The significance of the model coefficients confirmed the influence of these factors on dye uptake, validating the predictive capability of the response surface methodology for process optimization.

Response Surface Methodology (RSM) was employed to evaluate and visualize the effects of the three independent factors on color absorption (percentage exhaustion, %E), and to develop statistical models describing the dyeing process. The experimental conditions and corresponding %E values are summarized in Table 6. The resulting regression equation defines the quantitative relationship between the response (%E) and the independent process variables.

$$E\% = 26.51 + 5.06\mathbf{A} + (-0.58\mathbf{B}) + 0.100C + 0.67\mathbf{AB} + (-0.67\mathbf{AC}) + 0.36 + (-18.65\mathbf{A^2}) + (0.76\mathbf{B^2}) + 0.61\mathbf{C^2} \quad \text{..... (4)}$$

The statistical analysis of the dyeing process demonstrates the robustness and reliability of the developed quadratic model for predicting percentage dye exhaustion (%E). The model exhibited a standard deviation of 0.42, a mean value of 17.46, and a high degree of fit with $R^2 = 0.9993$, adjusted $R^2 = 0.9983$, and predicted $R^2 = 0.9909$, indicating excellent agreement between the experimental and predicted values. The coefficient of variation (CV) was low at 2.3%, reflecting good precision and reproducibility, while the adequate precision of 82.369 confirms a strong signal-to-noise ratio, well above the recommended threshold of 4.

Analysis of variance (ANOVA) showed that model terms with p-values < 0.05 are statistically significant, including A (mordant concentration), B (time), the interactions AB and AC, and the quadratic terms A^2, B^2, and C^2. Terms with p-values > 0.1 were considered insignificant (Table 6). The high F-value of 1075 further supports the significance of the model. The close agreement between the predicted and adjusted R^2 values (difference < 0.2) indicates the model reliably predicts dye exhaustion across the design space. these results confirm that the quadratic model is appropriate for navigating and optimizing the dyeing process (Aysha *et al.*, 2022; Lim *et al.*, 2022)



(a) (b) (c)

Figure 4: Three-Dimensional response surface plot showing the effect of process variables on dyeing (% Exhaustion)

Figure 4 presents the three-dimensional response surface plots illustrating the combined effects of the process variables on dye exhaustion (%E): (a) mordant concentration and pH, (b) dyeing time and pH, and (c) mordant concentration and time. These 3D plots provide a clear visualization of the interactions between the independent variables and the response, facilitating a better understanding of their influence on dye uptake. It is evident that mordant concentration and dyeing time have the most significant impact on %E, followed by pH, which is consistent with the ANOVA results ($p < 0.05$). These findings also align with previously reported observations on the effect of process parameters on dyeing performance and fastness properties (Criado *et al.*, 2020) .

Analysis of color fastness

The color fastness of dyed cotton fabric was evaluated to assess the durability and stability of the *Ixora coccinea* dye under various conditions. Fastness testing provides crucial information on the resistance of the dyed fabric to fading or staining when exposed to light, washing, and pressing, which are key indicators of the practical performance of natural dyes in textile applications. Standardized methods were employed to ensure reliable and reproducible evaluation of the dye's fastness properties.

The color fastness of cotton fabric dyed with *Ixora coccinea* extract was evaluated for light, washing, and ironing resistance, and the results are summarized in Table 7.

Light fastness grades of 6 were observed for runs 1, 3, 4, 5, 7, 8, 10, 14, 16, and 17, indicating very good resistance to light. Runs 2, 6, 9, 11, 12, 13, and 15 showed a grade of 5, representing good light resistance. Wash fastness tests showed grades of 4 for runs 1, 3, 4, 5, 6, and 7, demonstrating very good resistance to washing. Runs 2, 8, 9, 10, and 11 received a grade of 3, indicating good wash fastness, with no staining observed on adjacent white fabric. The dyed fabrics exhibited excellent resistance to ironing under dry, wet, and damp conditions, with a consistent grade of 5 and negligible staining for all runs. Under the optimal dyeing conditions, the cotton fiber demonstrated very good light fastness (grade 6), good wash fastness (grade 4) with no staining, and excellent ironing fastness (grade 5), confirming the effectiveness and durability of the *Ixora coccinea* natural dye (Guddi *et al.*, 2024b) (Hamdy *et al.*, 2021; Narmatha, 2020).

CONCLUSION

The present study successfully optimized the extraction and dyeing conditions of *Ixora coccinea* using Response Surface Methodology (RSM) based on a Box–Behnken Design (BBD, n = 17). The developed quadratic models for both dye yield and dye exhaustion were statistically significant ($p < 0.05$), with high coefficients of determination (R^2), adjusted R^2, and predicted R^2 values, confirming strong model fit and predictive reliability. Model adequacy was further supported by low coefficient of variation (CV), non-significant lack-of-fit, and Adeq Precision values greater than 4, indicating a satisfactory signal-to-noise ratio. Optimization results revealed that maximum dye yield of 49% was achieved at 90 min extraction time, solvent-to-material ratio of 1:20 g/mL, and pH 7. Similarly, optimal dyeing conditions (4.5% owf mordant concentration, 30 min dyeing time, and pH 7) resulted in a maximum dye exhaustion of 26.99%, demonstrating a significant improvement compared to non-optimized conditions. These results confirm the effectiveness of the applied statistical approach in enhancing both extraction efficiency and dye uptake. Spectroscopic characterization (FTIR and UV–Vis) confirmed the presence of functional groups associated with phenolic and anthocyanin compounds, which contribute to dye–fiber interaction. Under optimized conditions, the dyed cotton fabric exhibited good to excellent fastness properties, including light fastness (grade 6), washing fastness (grade 4), and excellent ironing fastness (grade 5), indicating satisfactory durability for textile applications. Generally, the integration of statistically validated optimization, chemical characterization, and performance evaluation demonstrates that *Ixora coccinea* is a viable and sustainable natural dye. The study provides a robust and reproducible framework for natural dye development, with clear advantages in process efficiency, model reliability, and eco-friendly textile application.

REFERENCES

Abdullahi, S. S. a., Musa, H., Habibu, S., Birniwa, A. H., & Mohammad, R. E. A. J. B. o. t. C. S. o. E. (2021). Facile synthesis and dyeing performance of some disperse monomeric and polymeric dyes on nylon and polyester fabrics. 35(3), 485–497. [Crossref]

Aftab, M., Ahmad, T., Adeel, S., & Javed, M. J. S. C. (2024). Ecofriendly process optimization of natural dye extraction from clove (Syzygium aromaticum L.) using response surface methodology and artificial neural network modeling. 37, 101401. [Crossref]

Aggarwal, S. J. P. (2025). Advancement in extraction and characterization techniques of natural dyes from dye yielding plants sources: a review. 54(2), 293–301. [Crossref]

Alves, B., Silva, L., Soares, G., & Santos, J. (2022). Substrate color prediction through active monitoring of the exhaustion dyeing process. Paper presented at the *Materials Science Forum*. [Crossref]

Aysha, T. S., Ahmed, N. S., El-Sedik, M. S., Youssef, Y. A., & El-Shishtawy, R. M. J. S. R. (2022). Eco-friendly salt/alkali-free exhaustion dyeing of cotton fabric with reactive dyes. 12(1), 22339. [Crossref]

Boukhatem, G., Bencheikh, M., Bekkouche, S. R., Kırgız, M. S., Manikandan, D., & Krishnaraj, R. J. S. R. (2026). Data-driven optimization and pressuremeter modulus prediction using response surface methodology for smarter geotechnical design. *Scientific Reports*. [Crossref]

Bras, T., Paulino, A. F., Neves, L. A., Crespo, J. G., & Duarte, M. F. J. I. c. (2020). Ultrasound assisted

extraction of cynaropicrin from Cynara cardunculus leaves: Optimization using the response surface methodology and the effect of pulse mode. 150, 112395. [Crossref]

Burbano-Pulles, M. R., Maldonado-Alvarado, P. G., Rojas-Porras, S. A., Sciarini, L. S., Samman, N. C., & Lobo, M. O. J. A. S. (2026). Multivariate integration of functional and compositional transitions in gluten-free composite flours based on *Amaranthus caudatus* and *Lupinus mutabilis*. *Applied Sciences, 16*(8), Article 4027. [Crossref]

Che, J., & Yang, X. J. H. (2022). A recent (2009-2021) perspective on sustainable color and textile coloration using natural plant resources. 8(10). [Crossref]

Criado, S. P., Gonçalves, M. J., Tavares, L. B. B., & Bertoli, S. L. J. J. o. C. P. (2020). Optimization of electrocoagulation process for disperse and reactive dyes using the response surface method with reuse application. 275, 122690. [Crossref]

Darmawan, A., Riyadi, A., Muhtar, H., & Adhy, S. J. I. J. o. B. M. (2024). Enhancing cotton fabric dyeing: Optimizing Mordanting with natural dyes and citric acid. 276, 134017. [Crossref]

Dey, P., Dey, P., Hoque, M. B., Baria, B., Rahman, M. M., Shovon, S., & Das, D. J. T. R. J. (2025). Sustainable and eco-friendly natural dyes: a holistic review on sources, extraction, and application prospects. 00405175251321139.

Guddi, K., & Sarkar, A. J. S. C. (2024a). Optimization of green extraction technologies for recovering bioactive compounds from Ixora coccinea waste flower biomass: A comparative response surface methodology and artificial neural network modeling. 42, 101830. [Crossref]

Guddi, K., Sur, S., & Sarkar, A. J. B. C. (2024b). Analysis of floral biomass utilization feasibility of Ixora coccinea for its cost-effective application as a natural colorant. 14(14), 15661–15675. [Crossref]

Hamdy, D., Hassabo, A. G., & Othman, H. A. J. J. o. T. (2021). Various natural dyes using plant palette in coloration of natural fabrics. 18(2), 121–141. [Crossref]

Hannan, M. A., Haque, P., Kabir, S. F., & Rahman, M. M. J. C. (2019). Chemical-free scouring and bleaching of cotton knit fabric for optimum dyeing performance. 37(4), 265–280. [Crossref]

Islam, S., Jalil, M. A., Motaleb, K. A., Saeed, M. A., Belowar, S., Rahamatolla, M., Hossain, S., Mukit, M. A., Khan, A. N. J. S., & NOW, C. (2025). Toward a greener fabric: innovations in natural dyes and biomordants for sustainable textile applications. 2(continuous publication). [Crossref]

Islam, T., Islam, K. M. R., Hossain, S., Jalil, M. A., & Bashar, M. M. (2024). Understanding the fastness issues of natural dyes. In *Dye Chemistry-Exploring Colour From Nature to Lab*: IntechOpen. [Crossref]

Jain, D., Jain, H., Kushwah, S. S., Solanki, V. S., Malviya, L. N., & Singh, D. J. E. S. G. U. R. C. (2026). Enhancing Sustainability Through Plastic Waste Utilization in Construction: A Review. 179. [Crossref]

Lakshmi S, S., Jahan Elamkulavan, H., Keloth, C., Peediyekkal, J., M, S., & P, S. J. Z. f. P. C. (2025). Third-order nonlinear optical characteristics of natural dye anthocyanin extracted from Ixora coccinea. *(0)*. [Crossref]

Le, N. T., Tran, T.-H., Nguyen, V. K. T., Mai, T. C. C., & Nguyen, H. T. J. S. (2026). Aqueous hydrotrope-based green extraction of alkaloids: insights into mechanism and experimentation with *Coptis chinensis*. *Separation and Purification Technology*, Article 137195. [Crossref]

Lim, J., Jeong, S., Lim, S., Cho, H., Shim, J. Y., Hong, S. I., Kwon, S. C., Lee, H., Moon, I., & Kim, J. (2022). Development of dye exhaustion behavior prediction model using deep neural network. In *Computer aided chemical engineering* (Vol. 49, pp. 1825–1830): Elsevier. [Crossref]

Lin, L., Jiang, T., Liang, Y., Zhu, W., Inamdar, U. Y., Pervez, M. N., Navik, R., Yang, X., Cai, Y., & Naddeo, V. J. M. (2022). Combination of pre-and post-mercerization processes for cotton fabric. 15(6), 2092. [Crossref]

Mahfud, M. J. A. J. o. C. E. (n.d.). The Effect of Temperature on Extraction of Swietenia Mahagoni by Ultrasound-assisted Extraction (UAE) Method.

Mamand, D. M., Ahmed, B. Y., Aziz, D. M., Hama, P. O., Mohammed, P. A., Abdalkarim, K. A., Muhammad, D. S., Hussein, A. M., Hussen, S. A., & Aziz, S. B. J. S. A. P. A. M. (2025). Advanced spectroscopic approach for exploring the structural, optical, and electronic properties in dye-functionalized chitosan biopolymers. 329, 125485. [Crossref]

Manikandan, G., & Nagarasampatti Palani, K. J. I. J. o. M. R. (2025). Adsorption of methylene blue using Ixora coccinea leaf: unveiling the extraordinary potential for sustainable wastewater treatment. *(0)*. [Crossref]

Moniruzzaman, M., Mondal, M. S., & Hossain, M. N. J. J. E. S. (2018). The influence of mordant and mordanting techniques on ecofriendly dyeing of cotton fabric by extracted used tea. 9(1), 111–117.

Mozumder, S., & Majumder, S. J. D. I. U. J. S. T. (2016). Comparison between cotton and silk fabric dyed with turmeric natural colorant. 11, 73.

Munene, J., Onyatta, J., & Yusuf, A. J. A. J. A. C. R. (2020). Characterization of water hyacinth powder using FTIR spectroscopy and the adsorption behaviour of Pb2+, Cd2+, Zn2+, Ni2+ and Cr2+ in aqueous solution. 6, 47–55. [Crossref]

Narmatha, P. J. A. J. o. I. R. R. (2020). Extraction of natural dye from Ixora coccinea Linn. flowers and evaluation of cosmetics as face powder dyeing using different chemical mordants. 5(1), 7–14.

Nekkaa, A., Benaissa, A., Lalaouna, A. E., Mutelet, F., & Canabady-Rochelle, L. J. J. o. A. R. o. M. (2021).

Optimization of the extraction process of bioactive compounds from Rhamnus alaternus leaves using Box-Behnken experimental design. 25, 100345. [Crossref]

Nistor, A., & Butnariu, M. J. I. J. B. P. (2023). Physical properties and identification of flavonoids by ultraviolet-visible spectroscopy. 8(2), 239. [Crossref]

Nor, N. A. M. (2019). Natural Dye Extract from Ixora Siamensis as Light Harvester for Dye-Sensitized Solar Cells with Nanostructured Tio2 [Master's thesis, University of Malaya (Malaysia)].

Pervez, M. N., Yeo, W. S., Lin, L., Xiong, X., Naddeo, V., & Cai, Y. J. S. r. (2023). Optimization and prediction of the cotton fabric dyeing process using Taguchi design-integrated machine learning approach. 13(1), 12363. [Crossref]

Rosa, J. M., Guerhardt, F., Ribeiro Júnior, S. E. R., Belan, P. A., Lima, G. A., Santana, J. C. C., Berssaneti, F. T., Tambourgi, E. B., Vanale, R. M., & Araújo, S. A. d. J. C. T. (2021). Modeling and optimization of reactive cotton dyeing using response surface methodology combined with artificial neural network and particle swarm techniques. 23(8), 2357–2367. [Crossref]

Samant, I. A., & Gaikwad, D. (2020). Optimization of natural dye extraction from coconut husk.

Shariful Islam, S., Alam, M., & Akter, S. J. F. (2020). Investigation of the color fastness properties of natural dyes on cotton fabrics. 27(1), 1–6.

Stephanie Martins de Freitas, T., Menezes de Rodrigues, G., Matta Fakhouri, F., da Silva, C., Andrea Lima Cardoso, C., Ignacio Velasco, J., Tostes Filgueiras, C., & Augusto dos Santos Garcia, V. J. J. o. F. P. (2021). Application of the Box-Behnken experimental design for the extraction of phenolic compounds from araçá-roxo (Psidium myrtoides). 45(3), e15260. [Crossref]

Uzumcu, M. B., Celik, P., Gulumser, T., & Kadoglu, H. J. J. o. N. F. (2021). A comparison of color fastness properties of mulberry silk and tussah silk fabrics in blends with cellulosic fibers. 18(11), 1834–1843. [Crossref]