Scientific Research Publishing

American Journal of Plant Sciences, 2026, 17(5), 553-572
https://www.scirp.org/journal/ajps
ISSN Online: 2158-2750
ISSN Print: 2158-2742

Extraction and Characterization of Natural Dyes for Textile Applications from *Hibiscus sabdariffa*

Michael Bong Alang[1]*, Richard Domga[1], Amadou Amadou Oumarou[1], Blaise Kom[2], Epane Victor Mondo[1], Martin Benoit Ngassoum[1]

[1]Department of Applied Chemistry, The University of Ngaoundere, Ngaoundere, Cameroon
[2]Department of Chemistry, Faculty of Sciences, The University of Ngaoundere, Ngaoundere, Cameroon
Email: *balangmichael@yahoo.com

How to cite this paper: Alang, M.B., Domga, R., Oumarou, A.A., Kom, B., Mondo, E.V. and Ngasoum, M.B. (2026) Extraction and Characterization of Natural Dyes for Textile Applications from *Hibiscus sabdariffa*. *American Journal of Plant Sciences*, **17**, 553-572.
https://doi.org/10.4236/ajps.2026.175034

Received: February 9, 2026
Accepted: May 26, 2026
Published: May 29, 2026

Abstract

In this research work, a natural dye was extracted from *H. sabdariffa*, characterized to determine the ideal conditions for textile applications and evaluated the efficiency of the dye on textile fabrics. This natural dye is desirable due to its propensity to reduce environmental pollution when used to substitute synthetic dyes. *H. sabdariffa* contains a pigment known as anthocyanin which is the prime component of this dye responsible for its red color. The extraction of the dye by solvent extraction by maceration and followed by colorimetric analysis and the determination of ideal application conditions was realized using the Box-Behnken Design software with three factors that included concentration of the electrolyte in the dye bath (mg/L), the minimum time of the dyeing process (min.) and the temperature of the dye bath (˚C). The responses analyzed were the % dye exhaustion and the color strength of the dye. The experimental results of the responses allowed us to determine the optimal conditions of application using a validated quadratic polynomial model. The best yield of dye extraction was 9.60 ± 0.79%, the colorimetric characteristics of the dye were respectively 32.57 ± 1.90 for the lightness, 1.08 ± 0.05 and 9.89 ± 0.18 for a^* and b^*, 10.22 ± 0.59 and 263.39 ± 2.92 for the saturation and the hue. The Hydrogen potential was 3.14 ± 0.02. The optimal conditions obtained were: an electrolyte concentration of 80 mg/L, a temperature of 80˚C for 37.71 min to obtain a dye bath exhaustion of 95.02% and a color strength of 6.78 g/Kg. These conditions could therefore be implemented in the application of the bio dye obtained from *H. sabdariffa* to improve the quality of dyeing products with attendant environmental sanitization.

Keywords

Anthocyanin, Bio Dye, Environment, Dyeing Process, Natural Dye, Textile Applications

1. Introduction

The key activities of textile industry include the design, production and distribution of textile products such as yarns, cloths and clothing [1]. The raw materials may be natural or synthetic. The main steps in the production of cloth are producing the fibers, preparing and converting the fibers to yarn, converting the yarn to cloth, and then doing finishing works on the cloth [2]. The cloth is then taken to the producer of various garments. The textile industry is one of the important industries that generates a large amount of industrial effluents [3] [4]. Color is a major ingredient for attraction in many fabrics. The manufacture and use of synthetic dyes for fabric dyeing has therefore become an interesting industrial and commercial activity. Synthetic dyes have provided a wide range of colors for dyeing textile fabrics. However, due to their toxic nature, the persistent use of synthetic dyes has become a cause of dire concern to environmentalists as the use of synthetic dyes produces adverse effects on virtually all forms of life [5] [6]. The presence of sulphur, naphthol, vat dyes, nitrates, acetic acid, soaps, enzymes chromium compounds, and heavy metals like copper, arsenic, lead, cadmium, mercury, nickel, and cobalt and certain auxiliary chemicals all collectively make the textile effluent highly toxic [7] [8]. These organic materials react with many disinfectants, especially chlorine, and form by-products like dichlorobiphenyls (DBPs) or polychlorobiphenyls (PCBs) that are often carcinogenic and therefore undesirable. This effluent, if allowed to flow in the fields, clogs the pores of the soil resulting in loss of soil productivity [9]-[11]. There are two types of dyes namely natural and synthetic dyes. As a result of reported negative impact of synthetic dyes on public health and reported environmental hazards, much research has been conducted to limit the use of synthetic dyes while promoting the use of natural dyes for textile applications [12] [13].

Natural dyes date back to antiquity. People started using natural dyes as a cultural tool to portray their surroundings and embellish themselves by this art [14]. Natural dyes have been used to colour textiles from ancient times. A natural dye is a pigment, or substance derived from plants, animals, microbial and minerals which is renewable, sustainable and biodegradable with attendant environmental friendliness [15]. Natural colouring compounds may be extracted from plant roots, leaves, stems, bark, wood shavings, flowers, fruits, rinds, hulls, husks, and other plant components [16].

Natural dyes have many benefits such as being non-noxious, ecofriendly, pleasing shade to the eye and having attractive aroma [17] [18]. Some limitations of natural dyes may include: poor colour yield, poor colour reproducibility and complex dyeing processing [19]-[22]. It is also difficult to reproduce hues using natural dyes because these plant products can vary from season to season, place to place, species to species, and maturity time [23] [24]. The short comings or few disadvantages of natural dyes may be circumvented if a careful study of the parameters influencing the strength of the dye is done, encompassing temperature, dying time and concentration of the electrolyte.

As a result the challenges encountered in previous research works, the difficulties encountered with synthetic dyes and the potential benefits of natural dyes that this research work was undertaken to extract a bio-colorant (dye) from *H. sabdariffa* flowers rich in anthocyanin chemical compounds having desirable coloring properties [25] and then proceed to determine the optimum conditions which are environmentally friendly targeting textile applications *inter alia*. Hence the prime objective of this work is to extract an environmentally friendly dye (biocolorant) from the flowers of *H. sabdariffa* and to determine the optimum application conditions on a textile fabric such as color strength on the textile fabric.

2. Materials and Methods

2.1. Material

The plant material used in this research work consisted of the flowers *H. sabdariffa*. These were bought in the local market of Ngaoundere in the month of August 2024. The *H. sabbdaiffa* flowers were conveyed to the research laboratory, Department of Applied Chemistry, University of Ngaoundere, Cameroon. The flowers were manually cleaned by winnowing and sorting to remove solid impurities and then preserved in polythene bags.

2.2. Extraction of Color Compounds from the Flowers of *H. sabdariffa*

The dye was extracted from *H. sabdariffa* flowers. To do this, the previously cleaned flowers were ground using a blender (Panasonic MX-895M, Japan) for 15 minutes and the resulting powder was stored in polyethylene bags.

2.3. Determination of Moisture Content

The moisture content was determined following the method of the Association of Official Analytical Chemists [26]. Then, the weight of an empty pan was measured (M_1) and an amount of the sample powder was introduced in the pan and the weight was measured (M_2) then, placed in an oven at 105˚C for a period of 24 hours. The pan was removed from the oven and the weight was measured (M_3). The moisture content was then calculated in accordance with the ISO-18134-3 (2015) analytical procedure using the formula below:

$$\begin{aligned}\text{Moisture content}(\text{Mc}) &= \left(\frac{\text{weight of dry sample}}{\text{weight of wet sample}}\right)\times\frac{100}{1}\\ &= \left(\frac{M_2-M_3}{M_2-M_1}\right)\times\frac{100}{1}\end{aligned} \quad (1)$$

2.4. Extraction of Dyes

The dyes were extracted from the biological material using hot solvent extraction via maceration. In this method, 10 g of powder of *H. sabdariffa* was mixed respectively with 50 mL, 80 mL and 100 mL of aqueous ethanol (70:30 v/v) and heated on a hot plate at 50˚C, 60˚C and 70˚C for 30 min, 45 min and 60 min. The mixtures were filtered using 125mm Whitman filter paper. Each filtrate was put in a rotary evaporator (HEIDOLPH) for efficient and gentle removal of solvent from samples by evaporation. The yield of each sample was determined to know the best conditions of dye extraction [27] [28]. The yield is given by the ratio of the weight of the dried extract and the weight of the powder.

$$\text{Yield}(R) = \left(\frac{M_1}{M_2}\right) \times \frac{100}{1} \tag{2}$$

where, R: yield expressed in %, M_1: weight in gram of the dried extract, M_2: weight in grams of the powdered of hibiscus.

2.5. Characterization of Dye

Colorimetric Analysis of dyes: After the dye extraction, the samples were put in test tubes and affixed on the light source of the colorimeter (GENESYS 10S). Then after transmission of the light through the sample, the computer connected with the colorimeter which gave the different values of the color parameters such as lightness/darkness (L^*), redness/greenness (a^*) and yellowness/blueness (b^*). Positive values of a^* represent redness while negative values of a^* represent greenness. Similarly, positive values of b^* represent yellowness while negative values of b^* represent blueness. Then both the Chroma (C^*) and the Hue (H^*) are determined using the following equations [29]:

$$C^* = \sqrt{a^{*2} + b^{*2}} \tag{3}$$

$$H^* = \arctan\left(\frac{a^*}{b^*}\right) \tag{4}$$

The total colour differences (ΔE) are calculated using the formula

$$\Delta E = \left[(\Delta L)^2 + (\Delta a^*)^2 + (\Delta Lb^*)^2\right]^{\frac{1}{2}} \tag{5}$$

$$L = 116\left(\frac{y}{y_a}\right)^{\frac{1}{3}} - 16 \tag{6}$$

$$a^* = 500\left[\left(\frac{x}{x_a}\right)^{\frac{1}{3}} - \left(\frac{y}{y_a}\right)^{\frac{1}{3}}\right] \tag{7}$$

$$b^* = 200\left[\left(\frac{y}{y_a}\right)^{\frac{1}{3}} - \left(\frac{z}{z_a}\right)^{\frac{1}{3}}\right] \tag{8}$$

$$\Delta L = L_1 - L_2 \tag{9}$$

Determination of pH: The pH of the *Hibiscus sabdariffa* extract was measured following the NF ISO 11265. Measurements were done under agitating using a portable pH-meter (HANNA instruments HI 8033). The electrodes of the pH-meter were rinsed with distilled water and the buffer used was also distilled water at neutral pH. After dipping the electrode into the buffer to stabilize the reading of the pH, the electrodes were put in the *H. sabdariffa* extract to read the pH.

Determination of the Color Strength of the Dye: The ability of a pigment or dye to change the colour of otherwise colourless material is known as colour strength. Its calculations are made on the basis of numerical value related to the amount of light-absorbing material (colourant) contained in a sample [30]-[34]. This colour value is calculated as the sum of K/S values for the sample. It is proportional to the dye concentration on the fibre. According to the Kubelka-Munk equation:

$$\frac{K}{S} = \frac{(1-R)^2}{2R} \tag{11}$$

where K is the light absorption coefficient, S is the light scattering coefficient and R is the spectral reflectance of the substrate.

2.6. Optimizing Dye Adsorption on Textiles

A study on the fixation of the dye from *Hibiscus sabdariffa* has been realized using the soaking method [35]. This work was aimed at evaluating the dyeing process, making it faster and less expensive for use in industries.

The response surface methodology was used to optimize the adsorption process of the dye extracted from *Hibiscus sabdariffa* flowers. Preliminary experiments led us to select appropriate electrolyte concentration ($NaCl/Na_2SO_4$) within the concentration range of 30 to 80 ppm, contact time in the range of 15 to 45 minutes and temperature (30˚C to 80˚C) as variable parameters. The responses were the percentage dye bath exhaustion (**%DBE**) and the color strength on a textile fabric (**CSD**) expressed in grams per kilogram **(g/Kg)**. The Box-Behnken design was used to optimize the adsorption of this dye. Fifteen experiments were conducted using three (3) factors. These factors are designated by (X_1) for electrolyte concentration (ppm), (X_2) for contact time (minutes), and (X_3) for temperature (˚C) as displayed in **Table 1** below. The soaking time should be sufficient to allow maximum diffusion of the dye from the dye bath to the surface of the fiber. However, if the contact time becomes too long, it may become disadvantage for the process. Concerning temperature, if it is low, the fixation may take place after a long time and too high a temperature could destroy some dye molecules. Since in aqueous solutions cellulosic fibers have an anionic surface and the dye molecule also has hydroxyl groups, the electrolytes will react with the surface of the fiber to facilitate the fixation of the dye on the fiber through chemisorption processes [36].

Table 1. The different variables at the three levels used for the Box-Behnken Design.

Parameters	Designation	Low Level	Medium Level	High Level	Unit
		−1	0	+1	
Electrolyte Concentration	X_1	**30**	**55**	**80**	**ppm**
Contact Time	X_2	**15**	**30**	**45**	**minutes**
Temperature	X_3	**30**	**55**	**80**	**°C**

The Design Expert software was used to analyze the results obtained and generated the mathematical model equation, which is a second-degree polynomial equation with interaction between the different parameters (Eq 12).

$$\begin{aligned} Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{11} X_1^2 + \beta_{22} X_2^2 + \beta_{33} X_3^2 \\ + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{23} X_2 X_3 \end{aligned} \tag{12}$$

where Y = Response and in this case the **%DBE** and the **CSD (g/Kg)** on tested fabric.

The coefficients β_0, β_i, β_{ii} and β_{ij} represent respectively the intercept on the vertical axis and the estimated linear, quadratic and interactive effects of the parameters X_1, X_2 and X_3 respectively.

The mathematical model was validated by analysis of variance (ANOVA), determining the linear regression coefficient R^2, adjusted R^2, AADM, bias factor, and accuracy factor, respectively (Table 2).

Table 2. Validation criteria.

Validation Index	Determination Equation	Constant Expressions	Validation Conditions
Coefficient of Determination	$R^2 = \sum_{i=1}^{n}\left(\frac{Y_{theo}}{Y_{exp}}\right)$		$R^2 \geq 0.90$
Adjusted Coefficient	Adjusted R^2		$R^2 \geq 0.80$
Absolute Mean Deviation Analysis	$\text{AMDA} = \frac{\sum_{i=1}^{n}\left(\frac{Y_{iexp} - Y_{itheo}}{Y_{iexp}}\right)}{n}$		$\text{ADMA} = 0$
Bias Factor	$B = \frac{1}{n}\sum_{i=1}^{n}\log\left(\frac{Y_{itheo}}{Y_{iexp}}\right)$	$B_f = 10^B$	$B_f = 1$
Accuracy Factor	$A = \frac{1}{n}\sum_{i=1}^{n}\log\left(\frac{Y_{itheo}}{Y_{iexp}}\right)$	$A_f = 10^A$	$A_f = 1$

The use of 3D curves allowed us to see the surface response and isoreponse curves, the interactions between the different factors and their combined effects on the adsorption of the dye extracted from *Hibiscus sabdariffa* leaves on a textile material.

2.7. The Color Strength on a Textile Fabric

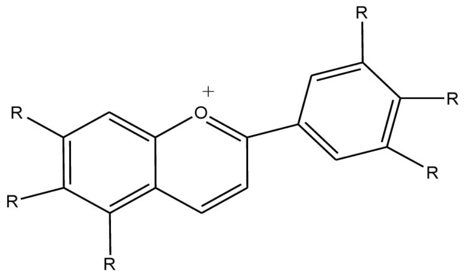
The dyeing of a fabric was conducted using the soaking method [37]. The dyeing process was carried out using the optimized conditions provided by the software application. In order to do this, 10 g of fabric was boiled in a 1% solution of NaOH for 15 min to remove starch, after it was washed with distilled water and air dried. A dye bath was prepared by taking a known mass of dye and dissolving it in a known volume of distilled water and the dried fabric was soaked in the dye bath. Then an electrolyte solution composed of a mixture of sodium chloride and sodium sulfate ($NaCl/Na_2SO_4$) was added to the mix to facilitate the dye adsorption onto cotton by overcoming repulsive forces. The dye bath was heated at a given temperature such as 30, 55 and 80˚C for a specified durations such as 15, 30 and 45 minutes. At the end of the dyeing process, the fabric was washed to remove unfixed dye and air dried again. The weight of the dried fabric was measured and the mass of dyed fixed on the fabric during the dyeing process was determined using the following formula:

$$M_d = M_2 - M_1 \tag{13}$$

where: M_d is the mass of dye fixed, M_1 is the mass of the fabric before the dyeing process M_2 is the mass of the fabric after the dyeing process.

2.8. Determination of the Exhaustion of the Dye Bath

This is a measure of the reduction in colour intensity of the dyeing bath medium and it is an indication of dyeing efficiency on a textile fabric. A calibration curve was used to determine the percentage of the exhaustion of the dye bath. The concentration of the anthocyanin dye bath was measured using a UV-Visible Spectrophotometer (GENESYS 10S) at a wavelength of 630 nm before and after the dye process. The chemical structure of the anthocyanin pigment is made up of a core flavonoid structure called anthocyanidin with a sugar molecule like glucose attached, forming a glycoside. The specific colour of the pigment is a function of the number and position of the hydroxyl and methoxyl groups on the anthocyanidin core and the type of sugar attached [38] [39].



Anthocyanin core molecular structure:

The amount of residual anthocyanin was determined using the calibration curve and from absorption measurements. The percentage of the exhaustion of the dye bath was obtained using the following equation:

$$\text{Dye Exhaustion} = \left(\frac{C_0 - C_1}{C_0}\right) \times \frac{100}{1} \tag{14}$$

where, C_0 is the initial concentration and C_1 is the concentration after dyeing.

From the residual concentration of the dye bath, the adsorbed concentration was deduced from the residual concentration using the formula.

$$C_A = \frac{1}{\varepsilon l}\left(A_i - A_r\right) \tag{15}$$

where C_A is the adsorbed concentration, A_i is the absorbance of the initial dye solution, A_r is the absorbance of the residual solution, l is the path length of cuvette cell and ε is the molar absorption coefficient of the dye.

3. Results and Discussion

3.1. Moisture Content of *H. Sabdariffa* Flowers

The moisture content gave 14.09 ± 1.02%. The lower the moisture content, the better the extraction. The value of moisture content obtained in this work is not far from the value of 10% reported by Chumsri [40] who studied optimum conditions of roselle extraction from *H. sabdariffa* without dyeing testing on a fabric. The relatively low moisture content in this research led to a god extraction yield.

3.2. Yield of Extraction

The best condition for dye extraction was a ratio of 1:10 w/v for the mass of *H. sabdariffa* powder and the solvent. As for time and the temperature, the best results were obtained for 45 min at 60˚C. So, three runs of extraction with aqueous ethanol (70:30) at 60˚C during 45 min gave an optimal yield of **9.60 ± 0.79%**. This value is in the propinquity of that found by Chumsri [40] which was 9.58%. This can be explained by the fact that at 60˚C and 45 min time lapse, the pores of the different cells are dilated enough to release the pigment and sufficient time is available to permit the pigment to flow out into the extraction medium. But if the temperature increases above the optimum value, some cells will undergo denaturation and the pores will be closed and this will lead to lower extraction yields.

3.3. Characteristics of the Dye

The dye extracted from H. sabdariffa was characterized through colorimetric analysis, hydrogen potential and the color strength of the dye**.**

Colorimetric Data:

A colorimetric analysis was realized on the extract of *H. sabdariffa,* the L^*, a^* and b^* values obtained also the saturation and the hue for the extract of *H. sabdariffa* are displayed in Table 3.

Table 3. Colorimetric analysis of the extract of *H. sabdariffa*.

L^*	a^*	b^*	C^*	H^*
32.57 ± 1.49	1.08 ± 0.05	−9.89 ± 0.18	10.22 ± 0.59	263.39 ± 2.92˚

The lightness value (L^*) is 32.57 ± 1.49, this means the extract is nearer to the black (0) than to the white (100). The values of the parameters a^* and b^* which indicate chromaticity coordinates (the direction of colour), are respectively 1.08 ± 0.05 and −9.89 ± 0.18. These values indicate that the extract of *Hibiscus* is greyish. These results are slightly different from those of Samanta [29] which indicated that the colour is reddish. The colour chemical parameters collected also slightly contradict physical observations of the extract which was pink/reddish to the naked eye. These might be due to experimental random errors. The Saturation and the hue have also been calculated for the *Hibiscus* extract. The saturation represents the intensity of a specific hue, it is based on the purity of the colour. The saturation (c^*) of our sample is 10.22 ± 0.59. The hue is the pure nature of a colour, without adjunction of white or black which causes shades. The value of the hue (H^*) is 263.39 ± 2.92˚. Hue refers to the pure name color like red, blue, green or purple. It is a function of the colour wavelength. Hue is measured as the angle clockwise from Red or calculated as arctan of the ratio b^*/a^*. Hue values above 260 degrees represent colors in the purple-blue to blue-purple range (Stacey, 2014). These colors include shades of violet, magenta, and blue, with the specific color depending on the exact degree of the hue. In this work, the hue value was 263.39 ± 2.92˚. Hence the colour of our extract was purple-blue to physical observation.

Hydrogen Potential: The hydrogen potential was 3.14 ± 0.02, at this value, the predominant form of anthocyanin present is the flavylium cation with a redish colour, and this assertion is in adequacy with the results of the colorimetric analysis.

Color Strength of the Dye: After the different analysis, based on the different responses obtained from the Box-Behnken design, the following conditions were obtained as ideal conditions for the dyeing process. These responses are in agreement with the results found by calculation of the mass of dye fixed on the fabric after the dyeing process which was 0.033 ± 0.0009 g on 5 g of fabric (Table 4).

Table 4. The different responses obtained.

Electrolyte (ppm)	Time (minutes)	Temperature (˚C)	Bath Exhaustion (%)	Color Strength (g/Kg)
80	80	37.71	95.03	6.78

This plant extract with intense reddish colour is rich in anthocyanin compounds according to the pH and colour data obtained in this work with confirmatory literature data whose wavelength of maximum absorption in the UV-Visible region ($\lambda_{\max}$) in the range 460 - 560 nm [30] (Table 4).

3.4. Optimization by the Box-Behnken Experimental Plan

Experimental Results

The preliminary study allows us to determine three factors which influence the fixation of the dye on the fibre and they have been introduced into the Box-Behnken experimental plan. The temperature (˚C), the time of soaking (min) and the amount of electrolyte (g/mL) have been studied. The parameters of the 15 experiments realized as the experimental plan and the different results obtained such as the exhaustion of the dye bath (%) and the color strength on a textile fabric (g/Kg) are represented in Table 5.

Exhaustion in dyeing refers to the proportion of dye that is absorbed by a fabric from the dye bath, expressed as a percentage. It indicates the efficiency of the dye transferred from the solution to the fabric. A high exhaustion rate implies more dye has been adsorbed, and a lower rate means more dye is left in the bath [41]. Each experiment is made up with a specific concentration of electrolyte in the dye bath at a specific temperature for a precise time. For each experiment realized, there is a theoretic value of the dye bath exhaustion expected and a real value obtained. The closer the observed % value is to the theoretical exhaustion value, the more effective the process.

Table 5. Real values of Box-Behnken Design, % dye bath exhaustion & dye color strength.

S/N˚	FACTORS			RESPONSE 1		RESPONSE 2	
	Electrolyte Conc. (mg/L)	Time (min)	Temp. (˚C)	Dye Bath Exhaustion (%)		Color Strength on a Textile Fabric (g/Kg)	
				Obtained Exhaustion	Theoretical Exhaustion	Observed Color Strength	Theoretical Color Strength
1	55	45	30	75.33	75.31	3.64	3.98
2	55	45	80	92.43	91.24	4.72	4.62
3	80	15	55	61.28	62.30	3.11	3.50
4	55	15	30	30.60	30.55	1.32	2.01
5	30	30	30	45.88	46.00	2.10	1.98
6	30	30	80	61.28	60.25	3.74	3.62
7	80	30	30	72.99	72.94	3.08	2.97
8	55	30	55	80.72	79.75	4.30	4.25
9	80	30	80	94.41	94.30	6.62	4.80
10	55	30	55	80.72	79.75	4.30	4.25
11	80	45	55	90.75	90.72	4.88	4.81
12	55	15	80	64.31	65.79	3.45	3.50
13	55	30	55	80.72	79.75	4.30	4.25
14	30	15	55	38.63	39.17	1.69	2.00
15	30	45	55	75.87	76.02	3.74	3.72

Each experiment is made up with a specific concentration of electrolyte in the dye bath at a specific temperature for a precise time. For each experiment realized, there is a theoretic value of the colour strength expected and a real value obtained.

The responses are expressed in percentage for the exhaustion of the dye bath which represents the mass of dye taken up by the material divided by the total initial mass of dye in bath and in grams of dye per kilogram of dried material. This is a measure of the ability of the dye to impart colour onto materials. The 15 experiments have a percentage of exhaustion between 30.60% and 94.41% which is similar to some literature reports [42].

3.5. Postulated Mathematical Model and Model Validation

The different responses which are the % exhaustion of the dye bath and the colour strength of the dye can be represented as a second degree polynomial equations where the different factors are directly linked to the different responses (Equation (16) and (Equation (17));

$$\begin{aligned} Y_1 = &+80.72 + 12.22x_1 + 17.44x_2 + 10.95x_3 - 1.94x_1x_2 + 1.51x_1x_3 \\ &- 4.15x_2x_3 - 5.56x_1^2 - 8.53x_2^2 - 6.52x_3^2 \end{aligned} \qquad (16)\ \text{(For the \%DBE)}$$

$$\begin{aligned} Y_2 = &+4.30 + 0.8025x_1 + 0.9262x_2 + 1.05x_3 - 0.07x_1x_2 + 0.475x_1x_3 \\ &- 0.2625x_2x_3 - 0.1712x_1^2 - 0.7737x_2^2 - 0.2437x_3^2 \end{aligned} \qquad (17)\ \text{(For the color strength of dye)}$$

where Y_1 = the percentage dye bath exhaustion (%DBE), Y_2 = colour strength of dye on test fabric, X_1 = electrolyte concentration, X_2 = time duration, X_3 = temperature and the composite variables represent their interactions which produce some effects on the responses.

From the model equations of the dye bath exhaustion, it is observed that the colour strength of the dye varies linearly with the electrolyte concentration, time and temperature while it is negatively affected by the quadratic and interactive effects of these parameters

The different parameters used to validate a model (**Table 6**) are respectively the coefficient of determination R^2 is more than 90% and the adjusted R^2 is between the standard value of 1 and the acceptable value of 80% [43], the Absolute Mean Deviation Analysis (**ADMA**) is between the standard value of 0 and the acceptable values 0.3, the bias factor (B_f) and the exactitude factor (A_f) are between the standard value of 1 and the acceptable values between 0.75 - 1.25. Hence the model used for the study of both the exhaustion of the dye bath and the color strength of the dye is valid.

Table 6. Model validation criteria.

MVI	R^2	Adjusted R^2	ADMA	B_f	A_f
%DBE	98.22%	95.00%	0.19	0.99	1.00
CSD	97.20%	92.15%	0.24	0.97	1.12
AV	$R^2 \geq 0.90$	$R^2 \geq 0.90$	$\text{ADMA} = 0 - 0.3$	$B_f = 0.75 - 1.25$	$A_f = 0.75 - 1.25$

Where MVI = Model validation indicator, %DBE = % dye bath Exhaustion and AV = acceptable values.

The Analysis of Variance allows us to evaluate the pertinence of the variables involved in the experimental design and to give a graphic representation and the importance of each parameter on the responses obtained which include: the % dye bath exhaustion and the color strength on the test fabric. The principle of ANOVA consists of the comparison of the variances of the different experiments realized to determine their deviation from the mean. The comparison between the variance of each experiment with the mean variance gave the "F-ratio" which is the ratio between the compared variances. Table 7 shows the effect of each factor and various interactions amongst one another. The value of the "F-ratio" depends on the number of Degrees of Freedom (DOF) involved in the model. So with a 95% degree of reliance, the effects with a P-value lower than 0.05 are considered as significant. The lower the uncertainty, the more significant effect becomes. The uncertainty is represented by the different P-values. For values of P less than 0.05 (5%), the effect is significant and its influence is considerable on the dye bath exhaustion or color strength of the dye. Significant factors for the dye bath exhaustion include the following: the linear effect of the electrolyte, the linear effect of the time, the linear effect of the temperature, the quadratic effect of the temperature and the quadratic effect of the time.

Table 7. Analysis of variance for the dye bath exhaustion and the color strength.

		Dye Bath Exhaustion (a)				Color Strength (b)			
SOURCE	DOF	Sum of squares	Mean square	*F*-value	*p*-value	Sum of squares	Mean square	*F*-value	*p*-value
Model	9	5133.99	572.67	30.58	0.0008	24.3597	2.70664	19.36	0.002
X_1: Electrolyte	1	1194.87	1194.87	63.80	0.0005	5.1440	5.14403	36.80	0.002
X_2: Cont. time	1	2434.62	2434.62	129.99	0.0001	6.8635	6.86351	49.10	0.001
X_3: Temperature	1	959.88	959.88	51.25	0.0008	8.7885	8.78853	62.87	0.001
X_1^2	1	114.04	114.04	6.09	0.0567	0.1091	0.10907	0.78	0.417
X_2^2	1	268.66	268.66	14.34	0.0128	2.2070	2.20697	15.79	0.011
X_3^2	1	157.08	157.08	8.39	0.0339	0.2205	0.22050	1.58	0.265
X_1X_2	1	15.09	15.09	0.81	0.4105	0.0196	0.01960	0.14	0.723
X_1X_3	1	9.06	9.06	0.48	0.5177	0.8978	0.89776	6.42	0.052
X_2X_3	1	68.97	68.97	3.68	0.1131	0.2756	0.27562	1.97	0.219
Residual	5	93.65	18.73			0.6989	0.13978		
Lack of Fit	3	93.65	31.22			0.6989	0.23296		
Pure error	2	0.00	0.00			0.0000	0.00000		
Total	**14**	**5247.63**				25.0586			

DOF: Degrees of freedom.

From this statistical study, we have these Pareto diagrams (**Figure 1**). The significant effects can be seen in the linearly effect of the electrolyte (X_1) on both the %DBE and CSD, the linear effect of the time (X_2), the linear effect of the temperature (X_3) on both responses, the quadratic effect of the electrolyte (X_1^2) on the percentage dye bath exhaustion alone but not on the CSD, the quadratic effect of the time (X_2^2) on both responses and the interactive effect of the electrolyte and temperature (X_1X_3) have positive significant effects on the color strength of the dye but not on the %DBE. However, the quadratic effects of the electrolyte (X_1^2), the quadratic effect of time (X_3^2), the interactive effect of the electrolyte and time (X_1X_2) as well as the interactive effect of time and temperature (X_2X_3) have a negative effect on the color strength of the dye (**Figure 2**).

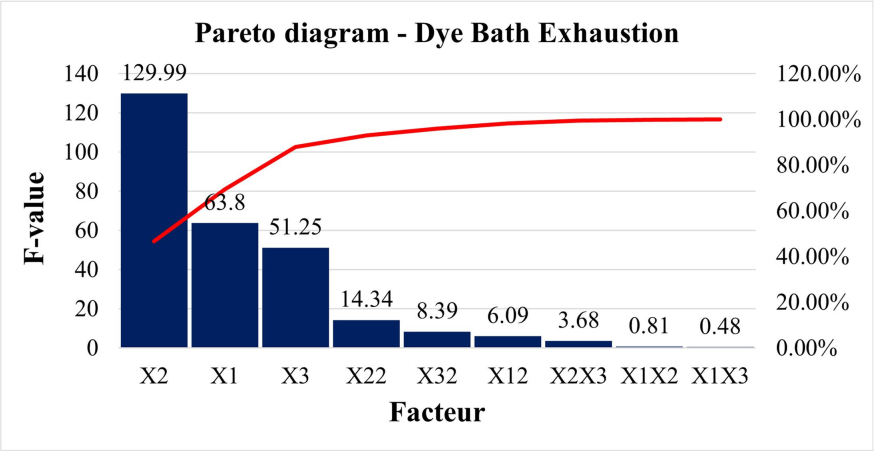


Figure 1. Pareto diagram of the %DBE.

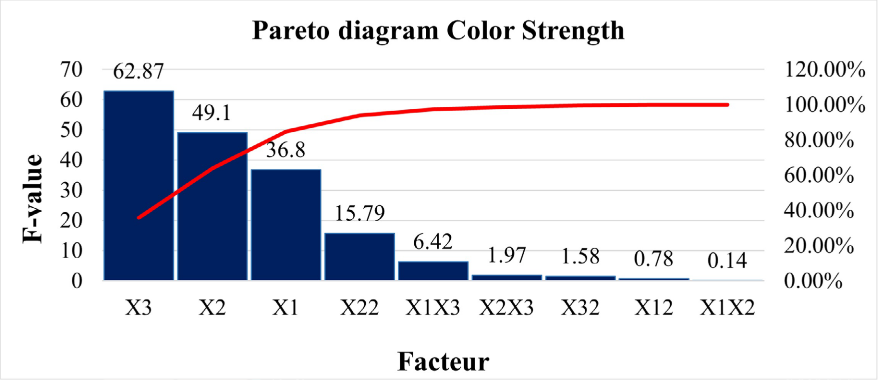


Figure 2. Pareto diagram of the CSD on the test fabric.

3.6. Surface Response Diagrams and Isoresponses

The mathematical equations Y_1 and Y_2 are used to plot the surface response curves. Two of the three variables are considered and varied while the third remains constant at it's central value. The **Figures 3(a)-(c)** below provide information on the influence of the independent variables and their interactions on the responses which in this research, encompass the %DBE and CSD. The response curves thus show that the depletion of the dye bath and the intensity of the dye color depend on the fixed variables.

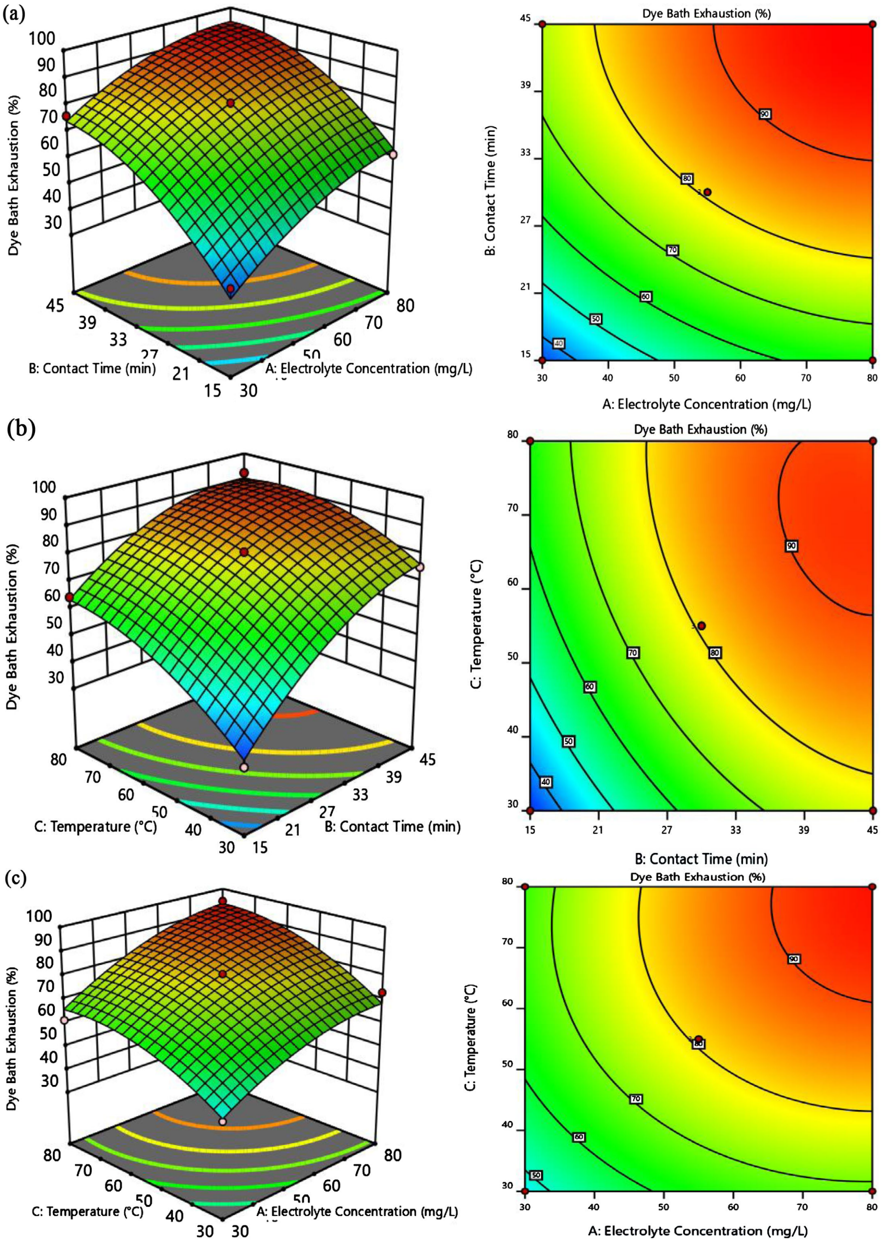


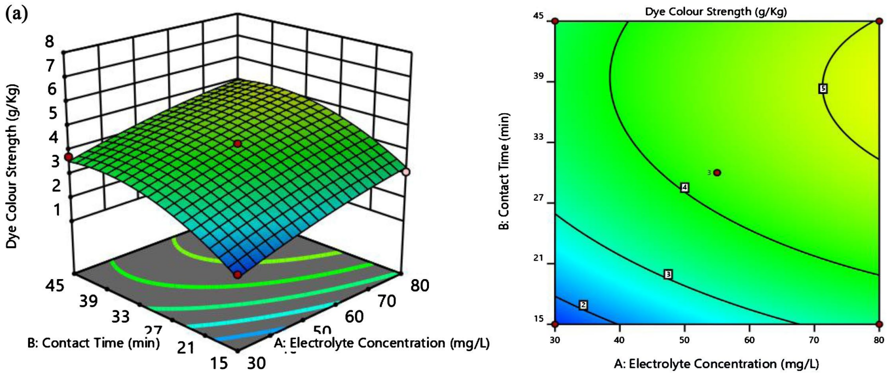
Figure 3. (a)-(c). Response surface curves of the behavior of the DBE.

Figure 3(a) shows the 3D surface diagram for the simultaneous effect of the electrlyte concentration and Temperature on %DBE at a constant contact time of 30 minutes. It can be seen from **Figure 2(a)** that the %DBE increased rapidly with the simultaneous increase in electrolyte concentration and temperature. The increasing electrolyte concentration facilitated the creation of active centres on the surface of the textile fabric that enhanced the adsorption of the dye molecules from the dye bath medium onto the fabric by processes which are probably more of chemisorption while the increasing temperature facilitated the kinetics of migration of dye molecules from the dye bath to be adsorbed onto the textile fabric thereby depleting the concentration of the dye molecules in the dye bath medium.

Then the highest percentage dye bath exhaustion of 95.02% was obtained at an electrolyte concentration of 80 ppm and at a temperature of 80˚C.

Figure 3(b) shows the 3D diagram for influence of electrolyte concentration and contact time on the percentage dye bath exhaustion at a constant temperature (55˚C). The simultaneous increase in the concentration of the electrolyte and contact time led to increasing depletion of coloring substances from the dye bath. As explained above, the increasing electrolyte concentration led to the creation of active adsorption sites on the textile test fabric while the increasing time progressively permitted the coloring molecules longer periods to migrate at the temperature of the medium to the active sites of the active sites on the fabric where adsorption took place by chemisorption processes due to the electronic active sites on the fabric created by the electrolyte. This trend continued until a maximum dye bath exhaustion of 95.02% was obtained at 80ppm electrolyte concentration and a contact time of 37.71 minutes. Longer contact times led to poorer %DBE probably due to excess time that developed an osmotic pressure in the dye bath medium that tends to favour the desorption of the dye from the fabric to maintain some developing adsorption versus desorption equilibrium. On the other hand, excessive electrolyte concentration is also detrimental to %DBE because lots of charges are created in the medium which might produce more repellent effects and tend to reduce the quantities of coloring substances adsorbed and hence poorer dye bath exhaustion [44].

Figure 3(c) which depicts the effect of the simultaneous variation of contact time and temperature on the %DBE at constant electrolyte can be similarly explained. The increasing temperature improves on the kinetics of the dye molecules moving from the dyeing medium to the textile fabric while increasing time allows for longer periods for the coloring molecules to migrate and get adsorbed onto the fabric, thus depleting the dyeing medium or improving the %DBE. This trend continued until a temperature of 80˚C and a contact time of 37.71 minutes. Higher temperatures led to lower responses, probably due to the destruction of some thermolabile coloring substances in the medium [45] [46] and possible desorption of some adsorbed molecules as the kinetics of the medium increases with temperature.



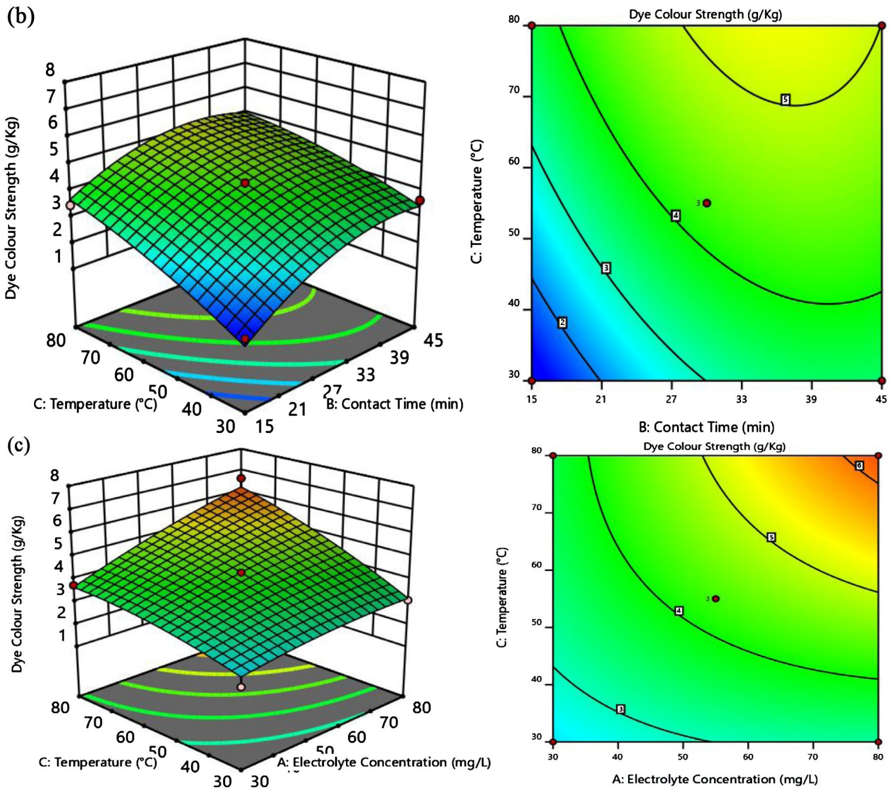


Figure 4. (a)-(c) Response surface curves behavior of the color strength.

Figure 4(a) shows the 3D diagram and isoreponse curves for the effect of contact time and electrolyte concentration on the color strength of the dye (CSD) at constant temperature. The color strength of the dye on the test textile fabric improves with the simultaneous increase in the electrolyte concentration and contact time. This trend continued until maximum color strength on the textile fabric was 6.78 g/Kg at a contact time of 37.71 minutes and an electrolyte concentration of 80 ppm. Longer contact times led to lower responses (lower CSD) as this could give room to desorption processes when equilibrium is attained. Higher concentrations of the electrolyte above 80 ppm also led to lower CSD on the textile test fabric probably due to repellent effects of excess charged species in the medium which resulted to reduced adsorption processes leading to lower CSD. The other 3D diagrams and isoreponse curves have similar interpretations of their effects on the CSD on the textile test fabric.

4. Conclusion

In this work, local bio resource materials were valorized to produce bio colorant dyes in order to limit importation of synthetic dyes which are more nefarious to pubic public health and the environment. These biodegradable and eco-friendly dyes are being produced to be consumed by the textile and related industries in Cameroon and beyond. Hence, in this work, a bio colorant was extracted from *H. sabdariffa* and suitable application conditions for application were determined such that when applied to the textile industry, optimum results shall be obtained. The best yield of extraction was 9.60%, the colorimetric characteristics of the dye were respectively 32.57 for the lightness (L), 1.08 and −9.89 for a^* (redness/greenness) and b^* (yellowness/blueness), 10.22 and 263.39˚ for the saturation and the hue. The Hydrogen potential was 3.14. The optimum conditions included an electrolyte concentration of 80ppm, a temperature of 80˚C for a time lapse of 37.71 min to obtain a dye bath exhaustion of 95.02% and a colour strength of 6.78 g/Kg. The model used could explain the behavior of the different factors.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

References

[1] Hole, G. and Hole, A.S. (2019) Recycling as the Way to Greener Production: A Mini Review. *Journal of Cleaner Production*, **212**, 910-915. https://doi.org/10.1016/j.jclepro.2018.12.080

[2] Sandin, G. and Peters, G.M. (2018) Environmental Impact of Textile Reuse and Recycling—A Review. *Journal of Cleaner Production*, **184**, 353-365. https://doi.org/10.1016/j.jclepro.2018.02.266

[3] Hassaan, M.A. (2016) Advanced Oxidation Processes of Some Organic Pollutants in Fresh and Seawater. PhD Thesis, Faculty of Science, Port Said University.

[4] Paosangthong, W., Torah, R. and Beeby, S. (2019) Recent Progress on Textile-Based Triboelectric Nanogenerators. *Nano Energy*, **55**, 401-423. https://doi.org/10.1016/j.nanoen.2018.10.036

[5] Mahajan, S.P. (2004) Pollution Control in Process Industries. Summer Report, IIT, 5.

[6] Kolya, H. and Kang, C. (2024) Toxicity of Metal Oxides, Dyes, and Dissolved Organic Matter in Water: Implications for the Environment and Human Health. *Toxics*, **12**, Article No. 111. https://doi.org/10.3390/toxics12020111

[7] Kant, R. (2012) Textile Dyeing Industry an Environmental Hazard. *Natural Science*, **4**, 22-26. https://doi.org/10.4236/ns.2012.41004

[8] Ardila-Leal, L.D., Poutou-Piñales, R.A., Pedroza-Rodríguez, A.M. and Quevedo-Hidalgo, B.E. (2021) A Brief History of Colour, the Environmental Impact of Synthetic Dyes and Removal by Using Laccases. *Molecules*, **26**, Article No. 3813. https://doi.org/10.3390/molecules26133813

[9] Ananthashankar, R. (2012) Treatment of Textile Effluent Containing Reactive Red 120 Dye Using Advanced Oxidation. MSc Thesis, Dalhousie University.

[10] Yaseen, D.A. and Scholz, M. (2018) Treatment of Synthetic Textile Wastewater Containing Dye Mixtures with Microcosms. *Environmental Science and Pollution Research*, **25**, 1980-1997. https://doi.org/10.1007/s11356-017-0633-7

[11] Makene, V.W., Tijani, J.O., Massima, E., Petrik, L.F. and Pool, E.J. (2019) Toxicity Study of a Textile Effluent Treated with Electrohydraulic Discharge and Coagulant/Flocculants. *Water, Air, & Soil Pollution*, **230**, Article No. 167. https://doi.org/10.1007/s11270-019-4212-z

[12] Adeel, S., Ali, S. and Zsila, F. (2009) Dyeing of Cotton Fabric Using Pomegranate (*Punica granatum*) Aqueous Extract. *Asian Journal of Chemistry*, **21**, 3493-3496.

[13] Ragab, M.M., Hassabo, A.G. and Othman, H.A. (2022) An Overview of Natural Dyes Extraction Techniques for Valuable Utilization on Textile Fabrics. *Journal of Textiles, Coloration and Polymer Science*, **19**, 137-153.

[14] Kiron, M.I. (2021) Kenaf Fiber: Properties, Cultivation, Production, Uses and Advantages. https://textilelearner.net/kenaf-fiber-properties

[15] Mansour, R. (2018) Natural Dyes and Pigments: Extraction and Applications. In: Yusuf, M., Ed., *Handbook of Renewable Materials for Coloration and Finishing*, John Wiley & Sons, Inc., 75-102.

[16] Muthu, S.S. (2017) Textile Sc. and Clothing Technology. Springer Science.

[17] Chavan, R.B. (1999) Chemical Processing of Handloom Yarns and Fabric. Department of Textile Technology, IIT, 6.

[18] Shirsath, N.B., More, M., Ahmed, H.S. and Savale, T.S. (2024) Extraction of Natural Dyes, Characterizations, and their Ecofriendly Applications. *International Journal for Multidisciplinary Research*, **6**, 1-10.

[19] Ghosh, P., Samanta, A.K. and Das, D. (1994) Effect of Selective Pretreatments and Different Resin Post-Treatments on Jute-Viscose Upholstery Fabric. *Indian Journal of Fibre and Textile Research*, **19**, 298.

[20] Narayanaswamy, V., Ninge Gowda, K.N. and Sudhakar, R. (2013) Dyeing and Color Fastness of Natural Dye from *Psidium guajuva* on Silk. *Journal of Natural Fibers*, **10**, 257-270. https://doi.org/10.1080/15440478.2013.797948

[21] Ahmed, M.A., Eltahir, Y.A. and Saeed, H.A.M (2018) Eco-Friendly Utilization of Banana Plant Extract for Dyeing of Textile Materials and Related Purposes. *International Journal of Recent Scientific Research*, **9**, 26465-26473.

[22] Chia, P.W., Lim, B.S., Tan, K.C., Yong, F.S.J. and Kan, S. (2019) Water Extract of Onion Peel for the Synthesis of Bisindolylmethanes. *Journal of King Saud University—Science*, **31**, 642-647. https://doi.org/10.1016/j.jksus.2018.05.029

[23] Affat, S.S. (2021) Classifications, Advantages, Disadvantages, Toxicity Effects of Natural and Synthetic Dyes: A Review. *University of Thi-Qar Journal of Science*, **8**, 130-135.

[24] Habib, N., Akram, W., Adeel, S., Amin, N., Hosseinnezhad, M. and Haq, E.u. (2022) Environmental-Friendly Extraction of Peepal (*Ficus religiosa*) Bark-Based Reddish Brown Tannin Natural Dye for Silk Coloration. *Environmental Science and Pollution Research*, **29**, 35048-35060. https://doi.org/10.1007/s11356-022-18507-5

[25] Welch, C.R., Wu, Q. and Simon, J.E. (2008) Recent Advances in Anthocyanin Analysis and Characterization. *Current Analytical Chemistry*, **4**, 75-101. https://doi.org/10.2174/157341108784587795

[26] AOAC (1990) The Association of Official Analytical Chemists. 14th Edition.

[27] Geetha, B. and Sumathathy, V.J.H. (2013) Extraction of Natural Dyes from Plants. *International Journal of Chemistry and Pharmaceutical Sciences*, **1**, 502-509.

[28] Lakshmana Naik, R., Sai Shireesha, V., Mayuri, A., Prakash, P. and Swetha, S. (2019) Extraction of Natural Dyes from the Floral Parts of Plants and Its Applications in Fabrics. *Journal of Emerging Technologies and Innovative Research*, **6**, 377-380.

[29] Samanta, A.K. (2020) Bio-Dyes, Bio Mordants and Bio-Finishes: Scientific Analysis for Its Application on Textiles. In: Samanta, A.K., Awwad, N. and Algarni, H.M., Eds., *Chemistry and Technology of Natural and Synthetic Dyes and Pigments*, InTechOpen, 3-37.

[30] Giusti, M.M. and Wrolstad, R.E. (2001) Characterization and Measurement of An-

thocyanins by UV-Visible Spectroscopy. *Current Protocols in Food Analytical Chemistry*, F1.2.1-F1.2.13. https://doi.org/10.1002/0471142913.faf0102s00

[31] Teli, M.D., Valia, S.P. and Kolambakar, D. (2014) Colouration Properties of Indigo Dyed Wool Top Dyed with Marigold. *Journal of the Textile Association*, **75**, 175-177.

[32] Zarkogianni, M., Mikropoulou, E., Varella, E. and Tsatsaroni, E. (2010) Colour and Fastness of Natural Dyes: Revival of Traditional Dyeing Techniques. *Coloration Technology*, **127**, 18-27. https://doi.org/10.1111/j.1478-4408.2010.00273.x

[33] Smith, S.D. (2014) Quantifying Color Variation: Improved Formulas for Calculating Hue with Segment Classification. *Applications in Plant Sciences*, **2**, Article ID: 1300088. https://doi.org/10.3732/apps.1300088

[34] Kumar Samanta, A. (2022) Colorimetric Evaluations and Characterization of Natural and Synthetic Dyes/Pigments and Dyed Textiles and Related Products. In: *Colorimetry*, IntechOpen. https://doi.org/10.5772/intechopen.104774

[35] Siddiqua, U., Ali, S., Hussain, T., Bhatti, H. and Asghar, M. (2017) The Dyeing Process and the Environment: Enhanced Dye Fixation on Cellulosic Fabric Using Newly Synthesized Reactive Dye. *Polish Journal of Environmental Studies*, **26**, 2215-2222. https://doi.org/10.15244/pjoes/68430

[36] Kitkulnumchai, Y., Ajavakom, A. and Sukwattanasinitt, M. (2008) Treatment of Oxidized Cellulose Fabric with Chitosan and Its Surface Activity towards Anionic Reactive Dyes. *Cellulose*, **15**, 599-608. https://doi.org/10.1007/s10570-008-9214-8

[37] Yasukawa, R., Higashitani, H., Yasunaga, H. and Urakawa, H. (2008) Dye Fixation Process in Ink-Jet Printing of Cotton Fabric by Reactive Dye. *Sen'i Gakkaishi*, **64**, 113-117. https://doi.org/10.2115/fiber.64.113

[38] Castañeda-Ovando, A., Pacheco-Hernández, M.d.L., Páez-Hernández, M.E., Rodríguez, J.A. and Galán-Vidal, C.A. (2009) Chemical Studies of Anthocyanins: A Review. *Food Chemistry*, **113**, 859-871. https://doi.org/10.1016/j.foodchem.2008.09.001

[39] Fogorasi, M., Dochia, M. and Stanescu, M.D. (2025) Improvement of Cotton Dyeing by Simultaneous Treatment with Cellulases. *Cellulose Chemistry and Technology*, **59**, 939-948. https://doi.org/10.35812/cellulosechemtechnol.2025.59.79

[40] Chumsri, P., Anchalee, S. and Itharat, A. (2008) Studies on the Optimum Conditions for the Extraction and Concentration of Roselle (*Hibiscus sabdariffa* Linn.) Extract. *Songklanakarin Journal of Science and Technology*, **30**, 133-139.

[41] Bhuiyan, M.A.R., Shaid, A., Bashar, M.M. and Sarkar, P. (2016) Investigation on Dyeing Performance of Basic and Reactive Dyes Concerning Jute Fiber Dyeing. *Journal of Natural Fibers*, **13**, 492-501. https://doi.org/10.1080/15440478.2015.1069233

[42] Daglı, A. and Oktav Bulut, M. (2025) Sustainable Color Stripping for Cotton Fabric Dyed with C.I Reactive Red 195 in Green Chemicals and UV Irradiation. *Cellulose*, **32**, 5083-5099. https://doi.org/10.1007/s10570-025-06483-1

[43] Elamir, E.A.H. (2015) Analysis of Mean Absolute Deviation for Randomized Block Design under Laplace Distribution. *American Journal of Theoretical and Applied Statistics*, **4**, 138-149. https://doi.org/10.11648/j.ajtas.20150403.19

[44] Bhuiyan, M.R., Shaid, A., Bashar, M.M., Haque, P. and Hannan, M.A. (2013) A Novel Approach of Dyeing Jute Fiber with Reactive Dye after Treating with Chitosan. *Open Journal of Organic Polymer Materials*, **3**, 87-91. https://doi.org/10.4236/ojopm.2013.34014

[45] Gligor, O., Mocan, A., Moldovan, C., Locatelli, M., Crişan, G. and Ferreira, I.C.F.R. (2019) Enzyme-Assisted Extractions of Polyphenols—A Comprehensive Review.

Trends in Food Science & Technology, **88**, 302-315. https://doi.org/10.1016/j.tifs.2019.03.029

[46] Chemat, F., Abert Vian, M., Ravi, H.K., Khadhraoui, B., Hilali, S., Perino, S., *et al.* (2019) Review of Alternative Solvents for Green Extraction of Food and Natural Products: Panorama, Principles, Applications and Prospects. *Molecules*, **24**, Article No. 3007. https://doi.org/10.3390/molecules24163007