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Towards Sustainable Textile Processing: RSM-Based Optimization of Microwave-Assisted Natural Dyeing of Hemp/Cotton Blended Fabrics

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ABSTRACT

*The natural dyes have numerous advantages with fewer problems like color strength and fastness. Different methods have been extensively studied to make them industrial ready. This study optimizes the microwave-assisted natural dyeing of sustainable hemp/cotton blended fabrics using neem bark extract, while henna (*Lawsonia inermis*) extract was used as a natural bio-mordant to improve dye fixation and fastness properties. A Box–Behnken design under response surface methodology (RSM) was employed to evaluate the effects of microwave irradiation time (2–6 min), pH (2–6), powder concentration (4–10 g), and salt concentration (3–7 g/100 mL) on color strength (K/S). A quadratic regression model was developed and statistically validated through analysis of variance (ANOVA). The model was found to be highly significant ($p < 0.0001$) with a high coefficient of determination ($R^2 = 0.9814$), indicating excellent alignment between predicted and experimental values. Among the variables, pH and powder concentration exhibited the most significant influence on K/S, while salt concentration showed a comparatively minor effect. The response surface analysis revealed that lower pH, moderate levels of powder and salt enhanced dye uptake. Numerical optimization predicted the maximum color strength ($K/S = 3.93$) at a microwave irradiation time of 4.66 min, pH 2.0, powder concentration of 7.52 g, and salt concentration of 5.15 g/100 mL, with a desirability of 0.96. The model validation confirmed the reliability of the predicted results with minimal error. This work focuses on sustainable hemp/cotton blends and provides a predictive modeling framework for process optimization, which differs from previous works. The findings demonstrate that RSM is an effective tool for optimizing eco-friendly dyeing processes.*

KEYWORDS

microwave-assisted dyeing, natural dye, response surface methodology, neem, sustainable textile processing

INTRODUCTION

The textile dyeing industry is well recognized for utilizing a lot of water, energy, and synthetic chemicals. It is one of the most resource-intensive and environmentally polluting industries [1]. This perception is only partially accurate, as several other industries possess considerably higher pollution potential, which is difficult to realize in normal eyes [2]. Traditionally dyeing cotton using reactive and direct dyes needs a lot of salts and other chemicals. These chemicals make the wastewater hazardous and pollute the air and water [3]. Researchers also reported that the discharge of untreated dyeing effluents adversely affects aquatic ecosystems and human health. Thus, this has led to stricter regulations and a growing search for more sustainable alternatives [4].

Cotton remains one of the most widely used fibers in the textile industry due to its softness, absorbency, and ease of coloration [5]. Nevertheless, cultivating it takes a lot of water and pesticides. This raises concerns about its long-term sustainability [6]. Mixing cotton with other fibers is now a good approach to protect the environment while retaining the desired textile characteristics [7]. Hemp fiber has attracted significant interest among alternatives as it has a low environmental impact, is biodegradable, and requires fewer agrochemicals [8, 9]. Hemp/cotton blended fabrics offer a balanced combination of durability, moisture management, and environmental friendliness [10]. However, their varied fiber composition makes it hard for dyes to be absorbed and colors to stay the same [11].

Natural dyes have been adopted by researchers rather than synthetic ones in coloring the textiles, which is environmental-friendly [12, 13]. They are developing new fiber technology as well [14, 15]. Plant-based natural colors, such as colorants derived from bark, leaves, and roots are safe, biodegradable and renewable [16]. Natural dyes also have been known to add additional useful properties to fabrics, such as antibacterial, antioxidant, and UV-protective properties [17, 18]. Several studies have reported that plant-derived dyes exhibit antimicrobial activity against pathogenic microorganisms, so they can be used in medical and hygienic textiles [19]. Although natural dyeing offers several environmental and functional advantages, it also presents certain limitations, including poor color strength, limited reproducibility, and inadequate fixation on cellulosic substrates [20]. This suggests that the method has to be improved.

Innovative dyeing methodologies, including microwave-assisted dyeing (MAD), have been implemented to overcome such problems [21]. Microwave irradiation enables rapid and uniform heating during the dyeing process. This leads to better dye uptake and shorter processing times [22]. Microwave-assisted dyeing has been shown to use less energy and get better color results than traditional heating processes, making it a promising green option [23]. Microwave-assisted dyeing has proven beneficial for many natural colors on cotton fabrics [24, 25]. Nevertheless, its applicability to blended systems, such as hemp/cotton, remains largely unexamined.

Dyeing parameter optimization is necessary for obtaining the desired color strength and consistent results [26]. This is particularly the case with natural dyeing systems in which numerous factors are in play simultaneously. The traditional methods of one-factor at a time (OFAT) are insufficient because they do not consider the interaction effects between process factors [27]. On the other hand, statistical methods like response surface methodology (RSM) provide a structured and useful way to model and optimize [28]. The Box–Behnken design (BBD) is a common RSM design since it needs fewer runs and can examine quadratic interactions between variables [29]. RSM application in textile dyeing operations has been effective to enhance various parameters such as pH, temperature, concentration of dye and time which has resulted to enhancement of stronger coloration and efficient operations [30].

Previous studies have predominantly focused on the enhancement of natural dyeing on pure cotton substrates through experimental or statistical methodologies. For example, utilizing RSM to optimize plant-based dyeing processes on cotton garments has shown big gains in how well the dye sticks and how strong the color is [31]. Microwave-assisted natural dyeing has also been studied for cotton materials, revealing better dye penetration and shorter processing times [32]. Nonetheless, there exists a lack of research focusing on the synergistic application of microwave-assisted dyeing and statistical modelling on blended materials, especially hemp/cotton composites. Hemp fibres considerably change dye-fiber interactions and require a specific optimization strategy because they have a higher lignin content and a lower dye affinity than cotton [33].

Moreover, although numerous studies have documented the antibacterial efficacy of natural dyes on textiles [34-36], this research frequently remains confined to qualitative evaluations or single-factorial analyses. A holistic methodology that combines process optimization with the assessment of functional properties is mostly unexamined. Consequently, a systematic and predictive modelling framework is essential to concurrently enhance dyeing performance and assess functional aspects within eco-friendly textile systems.

In this case, the current study tries to make a statistically optimized and predictive model for using plant-based dye extracts in microwave-assisted natural dyeing of long-lasting hemp/cotton blended textiles. The impact of important process variables, including microwave irradiation time, pH, powder concentration, and salt concentration, on color strength (K/S) was investigated using a Box-Behnken design with response surface approach. The antimicrobial properties are not evaluated in this study as it is well established in previous research [37-40]. This research is unlike others that conducted extensive studies on conventional cotton substrates or optimization via experimental studies. It incorporates sustainable fiber blending, microwave-assisted dyeing and advanced statistical modelling. The results of this paper will likely help advance the development of environmentally friendly methods of dyeing fabrics and adopt environmentally friendly materials in the textile sector.

MATERIALS AND METHODS

Materials

A hemp/cotton fabric (20:80) with a fabric weight of 160 GSM was used as the substrate to be dyed in this study. The fabric was sourced from Knit Concern Group, Narayanganj, Bangladesh. The fabric was pre-scoured before experimentation to remove any impurities that were left behind and it was confirmed that the fabric was free of any finishing chemicals so that the uptake of the dye was uniform and that the dyeing process could be duplicated.

The neem (*Azadirachta indica*) bark was collected from a location close to Dhaka, Bangladesh (23.849234° N, 90.424379° E). The material collected was then washed thoroughly with distilled water to remove any impurities attached to it and allowed to dry in the air under ambient temperature, and then ground into fine powder to extract dye later. Henna leaves were collected from the same location. The gathered plant material was washed in distilled water to get rid of any impurities that adhered to it, dried under room temperature and ground into a fine powder to ease the extraction of the bioactive coloring constituents (lawsone). The prepared henna powder was used as a natural bio-mordant in the post-mordanting process without further purification [41]. The chemicals used in this study were analytical grade and were not further purified. pH was adjusted with acetic acid (CH₃COOH; CAS No.: 64-19-7) and sodium chloride (NaCl; CAS No.: 7647-14-5) was used as an electrolyte. All the dye extraction and dyeing were done using distilled water so as to be consistent and reproducible.

Preparation of Natural Dye Extract

The extraction of natural dyes was made using the neem (*Azadirachta indica*) bark powder in an aqueous acidic method [42]. The bark collected was well washed, dried in the air before grinding into a fine powder to facilitate even penetration of the solvent used to extract the color-bearing constituents and to allow efficient release of the color-bearing constituents. Each experimental run involved mixing neem bark powder (4 to 10 g) with 50 mL of distilled water to ensure the presence of an acidic environment where tannin-rich colorants would be extracted out of the bark matrix. The mixture was further heated to boiling and allowed to stand 60 min to permit sufficient diffusion of soluble components of dye into the extraction liquor. The hot liquor was then extracted and left to cool to room temperature and then filtered to eliminate insoluble fibrous residue and particulate matter. The resulting clear filtrate was taken as is the dye liquor in future experiments on microwave-assisted dyeing. This extraction method was chosen, since acidic extraction of the neem bark has been reported to give better color recovery and better dyeing results with regard to non-acidic media [43].

Microwave-Assisted Dyeing Process

Microwave irradiation was used on pre-treatment before the dyeing to increase dye extraction efficiency, improve dye-fiber interaction. Microwave irradiation generates rapid internal heating through dipole rotation and ionic conduction within the aqueous dye solution. This process enhances the rupture of plant cell structures, accelerates the release of colorants, and improves the dissolution and dispersion of dye molecules in the extraction medium [44, 45]. The increased molecular mobility and reduced aggregation of dye constituents subsequently promote more uniform diffusion and adsorption of dye molecules onto the hemp/cotton substrate during the dyeing stage. Therefore, the microwave treatment indirectly enhanced dye uptake by modifying the physicochemical characteristics of the dye liquor prior to fabric immersion. The resulting dye solution of neem bark was placed under microwave irradiation, at selected intervals (2-6 min) depending on the design of the experiment. The internal heating is supported by this treatment using dipole rotation and ionic conduction, which enhance molecular motion and facilitate efficient energy transfer within the dye solution. Thus enhancing the solubility and accessibility of tannin-based colorants [42]. To compare the effect of the microwave treatment on the dyeing performance, an unirradiated dye solution was also utilized. After irradiation, the ratio of the dye bath was prepared to be 1: 25 according to the weight of the fabric used (10 g). The pH of the dye bath was kept between 2 and 6 with the use of acetic acid and sodium chloride 3 to 7 g/100 mL was added to induce exhaustion of the dye.

Dyeing was carried out using an infrared (IR) dyeing machine at 100 °C for 60 min under controlled heating conditions. Fabric samples were dyed using both irradiated and non-irradiated dye baths in order to observe the effect of microwaving on the dyeing process. Besides, a confirmation test was performed using the optimal values as predicted by the established statistical models. Post-mordanting process was done using *Lawsonia inermis* extract (Henna) as bio-mordant at 10% (owf). Mordanting process was done at the material-to-liquor ratio of 1:25 and temperature 80 °C for 60 min in order to allow sufficient interaction of bio-mordant, dye, and fiber surface. Subsequently, the dyed fabrics were washed off using distilled water to get rid of any excess dye and mordant that might not be fixed onto the fabric surface. An illustration of the entire process is shown in Figure 1.

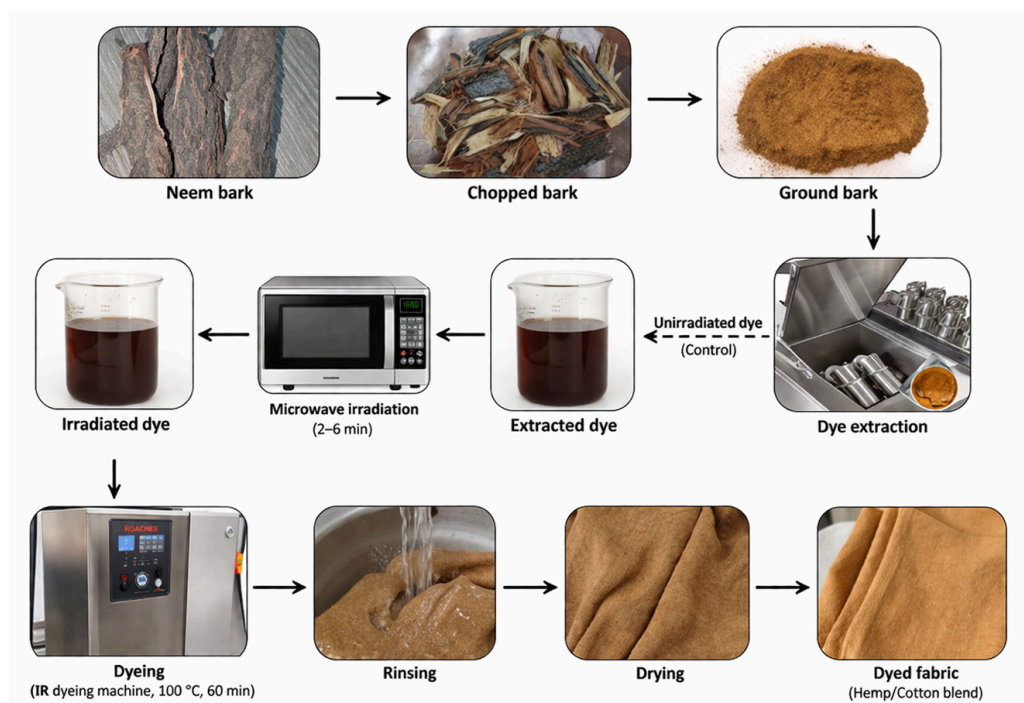


Figure 1. Dye extraction, irradiation and dyeing process of cotton/hemp blended fabric with neem bark.

Experimental Design

A Box–Behnken design (BBD) under response surface methodology (RSM) was employed to investigate the effects of four independent variables on color strength (K/S). The selected variables were microwave irradiation time (A), pH (B), powder concentration (C), and salt concentration (D). Although salt concentration was expected to exert a comparatively smaller influence than pH or microwave irradiation time, it was included in the experimental design because electrolytes play an important supporting role in promoting dye exhaustion

and improving dye–fiber interactions in natural dyeing systems. Since the interaction effects between salt concentration and other process variables were not fully known for the hemp/cotton blended substrate under microwave-assisted conditions, salt concentration was incorporated as an independent factor within the Box–Behnken design to systematically evaluate its individual and interactive contributions to color strength (K/S). Each factor was studied at three levels, coded as –1, 0, and +1, as shown in Table 1.

Table 1. Independent variables and their levels used in Box–Behnken experimental design.

Factor	Variable	Unit	Symbol	–1 (Low)	0 (Center)	+1 (High)
A	Irradiation time	min	A	2	4	6
B	pH of dye bath	–	B	2	4	6
C	Dye powder concentration	g	C	4	7	10
D	Salt concentration	g/100 mL	D	3	5	7

A total of 27 experimental runs were generated by the design, including center points for error estimation. The experimental matrix and corresponding response values were used to develop a predictive model.

Statistical Modeling and Analysis

The experimental data were analyzed using response surface methodology to develop a quadratic regression model. Analysis of variance (ANOVA) was performed to evaluate the significance of the model and its terms. The adequacy of the model was assessed using statistical parameters such as the coefficient of determination (R^2), adjusted R^2 , predicted R^2 , coefficient of variation (C.V.), and adequate precision.

The general form of the quadratic model is expressed as:

$$Y = \beta_0 + \sum \beta_i X_i + \sum \beta_{ii} X_i^2 + \sum \beta_{ij} X_i X_j$$

where Y is the response (K/S), β represents regression coefficients, and X_i and X_j are independent variables.

Optimization of Dyeing Parameters and Model Validation

Numerical optimization was performed to determine the optimal dyeing conditions for maximizing color strength (K/S) using the desirability function approach, where the objective was set to maximize the response within the specified range of process variables. The optimal conditions were identified based on the highest overall desirability value predicted by the model.

To validate the predictive capability of the developed model, confirmation experiments were conducted under these optimized conditions. The experimentally obtained K/S values were compared with the corresponding predicted values, and the percentage error was calculated to evaluate the accuracy and reliability of the model.

RESULTS AND DISCUSSION

The experimental design matrix generated using the Box–Behnken design, along with the corresponding observed color strength (K/S) values, is presented in Table 2. The design consists of 27 experimental runs, including replicated center points, to evaluate the effects of microwave irradiation time (A), pH (B), powder amount (C), and salt concentration (D) on the dyeing performance. The K/S values obtained from the experiments varied from 2.12 to 3.86, indicating a significant influence of process variables on color strength.

Table 2. Experimental design matrix and corresponding color strength (K/S) values.

Run	Irradiation time (min)	pH	Powder (g)	Salt (g/100 mL)	K/S
1	2	4	4	5	2.12
2	4	4	7	5	3.08
3	6	4	7	3	2.74
4	2	4	7	7	2.58
5	6	2	7	5	3.86
6	6	4	7	7	2.66
7	6	6	7	5	2.18
8	4	4	7	5	3.14
9	2	2	7	5	3.62
10	4	4	4	7	2.41
11	4	2	4	5	3.48
12	4	6	7	7	2.34
13	2	6	7	5	2.26
14	2	4	10	5	2.68
15	4	4	7	5	3.11
16	4	4	10	3	2.73
17	6	4	10	5	2.88
18	4	6	10	5	2.42
19	4	6	7	3	2.29
20	4	2	7	7	3.71
21	4	6	4	5	2.21
22	4	4	4	3	2.52

Run	Irradiation time (min)	pH	Powder (g)	Salt (g/100 mL)	K/S
23	6	4	4	5	2.63
24	2	4	7	3	2.51
25	4	4	10	7	2.79
26	4	2	10	5	3.59
27	4	2	7	3	3.55

Effect of Process Variables on Color Strength (K/S)

The comparison plot of the single-factor interpretation of the effects of time, pH, powder quantity, and salt concentration on the color strength (K/S) of the dyed fabric was first made using the time of microwave irradiation. The trends of mean responses clearly showed that all the process variables influenced the dyeing performance, but the magnitudes of their effects were not equal.

The variables that were investigated, pH had the most significant impact on K/S. The color strength was highest at the low pH level and gradually decreased as the pH increased from 2 to 6. Based on this behavior, acidic conditions increase the fixation of dye onto the substrate. When in an acidic medium, the dye molecules are in a more desirable position for the diffusion and the interaction with the hydroxyl-containing cellulosic constituents of the blended fabric and thus increase the depth of color. The main effects of microwave irradiation time, pH, powder amount, and salt concentration on color strength are presented in Figure 2.

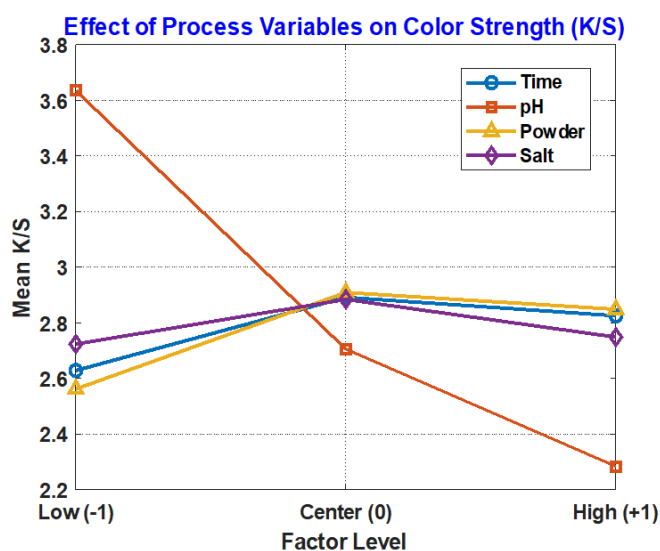


Figure 2. Main effects plot showing the influence of microwave irradiation time (A), pH (B), powder amount (C), and salt concentration (D) on the color strength (K/S) at different factor levels.

Microwave irradiation time also played an important role in determining K/S. An increase in time from the low to the center level led to a notable rise in color strength, indicating improved dye diffusion and more effective microwave-assisted penetration of the dye into the fabric structure. However, a further increase toward the high level did not produce a proportional increase, implying that excessive irradiation time may not be beneficial beyond the optimum range.

The powder amount effect was similar. As the concentration of the dye source was increased (4 to about 7 g), the K/S also enhanced, with the primary improvement being the increased supply of the coloring matter in the dye bath. In the center area, the growth rate was lower and tended even to decrease, which can be explained by the fact that there are a limited number of binding sites and the surface of the fabric is somewhat oversaturated.

Conversely, salt concentration was the least affected of the four variables. Though the addition of moderate salt levels was found to enhance dye exhaustion and marginally the K/S, the overall variation was relatively lower in comparison to that of pH, time and amount of powder. This implies that salt concentration was a supporting factor in the range of experiments chosen and is not a dominant control parameter.

On the whole, the main-effect trend indicates that the pH was the most imperative variable that influenced the color strength, then the powder amount, and the time of microwave irradiation, and lastly, the salt concentration had the least impact.

Statistical Modeling of K/S Response

To quantitatively describe the relationship between the selected dyeing variables and color strength, the experimental data were fitted using response surface methodology based on a Box–Behnken design. A quadratic polynomial model was selected to represent the K/S response. The statistical adequacy of the selected model was summarized in Table 3.

Table 3. Statistical summary of the selected quadratic model for K/S.

Response	Model	Std. Dev.	Mean	C.V. (%)	R ²	Adjusted R ²	Predicted R ²	Adeq Precision	Remark
K/S	Quadratic	0.1048	2.82	3.72	0.9814	0.9598	0.8940	22.17	Suggested

The developed model showed a high coefficient of determination ($R^2 = 0.9814$), indicating that 98.14% of the variation in K/S could be explained by the fitted equation. The adjusted R^2 (0.9598) and predicted R^2 (0.8940)

were in close agreement, with a difference well below 0.20, confirming satisfactory predictive capability of the model. In addition, the model showed a low coefficient of variation (3.72%), suggesting good precision and reproducibility of the experimental results. The adequate precision value of 22.17 further demonstrated that the model had an excellent signal-to-noise ratio and could be reliably used for navigating the design space. These statistical indicators confirm that the selected quadratic model was appropriate for describing the dyeing system and could be used for optimization of the microwave-assisted dyeing process.

Analysis of Variance (ANOVA)

The ANOVA results for the selected quadratic model are presented in Table 4. The model was highly significant, with an F-value of 45.33 and a p-value below 0.0001, confirming that the variation in K/S was strongly associated with the selected process parameters rather than random experimental noise.

Table 4. ANOVA of the selected quadratic model for color strength (K/S).

Model	Sum of Squares	DF	Mean Square	F-value	p-value	Remark
Quadratic (K/S)	6.97	14	0.4976	45.33	<0.0001	Significant
Residual	0.1317	12	0.0110	—	—	—
Lack of Fit	0.1299	10	0.0130	14.43	0.0665	Not significant
Pure Error	0.0018	2	0.0009	—	—	—
Total	7.10	26	—	—	—	—

Importantly, the lack-of-fit was not significant ($p = 0.0665$), which indicates that the developed model adequately described the experimental data and that no serious unexplained variation remained in the system. Therefore, the ANOVA strongly supports the suitability of the quadratic model for prediction and optimization of K/S.

Agreement Between Predicted and Actual Responses

The suitability of the model developed was also checked with the predicted and actual plot. Data points were found to be closely clustered around the diagonal reference line and there was a good correlation between the experimental and model-predicted values of K/S. This tight grouping shows that the quadratic model could achieve high accuracy in the estimation of the response throughout the entire experimental range.

The normal probability plot of residuals also indicated the sufficiency of the model. The pattern of the residual points was mostly on a straight line with few slight exceptions in the far ends. This indicates that the residuals

were normally distributed to a large extent and the regression assumptions were largely met. All of these plots shown in Figure 3, as a combination, verify that the model was statistically sound and paired to undergo optimization analysis.

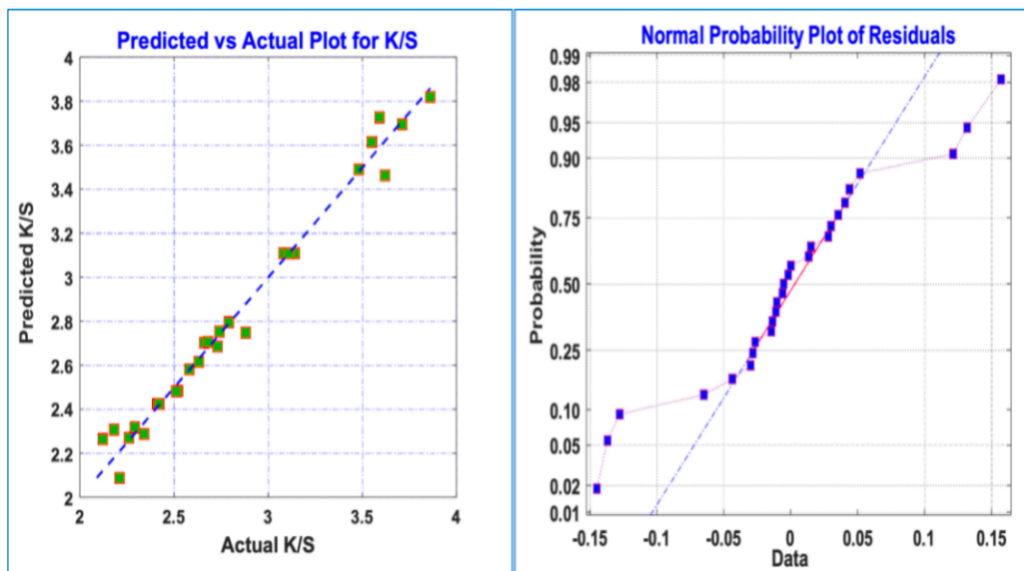


Figure 3. (a) Predicted versus actual plot for color strength (K/S) and (b) Normal probability plot of residuals for K/S

Quadratic Response Surface Equation

The final quadratic model, expressed in terms of actual factors, is given as follows:

$$\begin{aligned}
 K/S = & -0.296748 + 0.759792A - 0.456875B + 0.481620C + 0.603958D - 0.020000AB \\
 & -0.012917AC - 0.009375AD + 0.004167BC - 0.006875BD + 0.007083CD \\
 & -0.061667A^2 + 0.025521B^2 - 0.031019C^2 - 0.058229D^2
 \end{aligned}$$

where A, B, C, and D represent microwave time, pH, powder amount, and salt concentration, respectively.

The positive coefficients of time, powder, and salt in the linear region indicate that moderate increases in these variables initially enhance K/S, whereas the negative coefficient of pH indicates that increasing pH reduces color strength. The negative quadratic coefficients of time, powder, and salt further confirm that excessive levels of these parameters eventually reduce the response, thereby supporting the existence of an optimum within the design range.

Interpretation of 3D Response Surface Plots

The three-dimensional response surface plots provide a visual representation of the combined effects of the process variables on K/S. The interaction between time and pH showed that maximum color strength was obtained at relatively higher microwave irradiation time and lower pH. This trend indicates that microwave energy promotes dye diffusion, while acidic conditions increase the affinity of the natural dye toward the fabric substrate [42, 46]. The combined interaction effects of the selected process variables on K/S are illustrated in Figure 4.

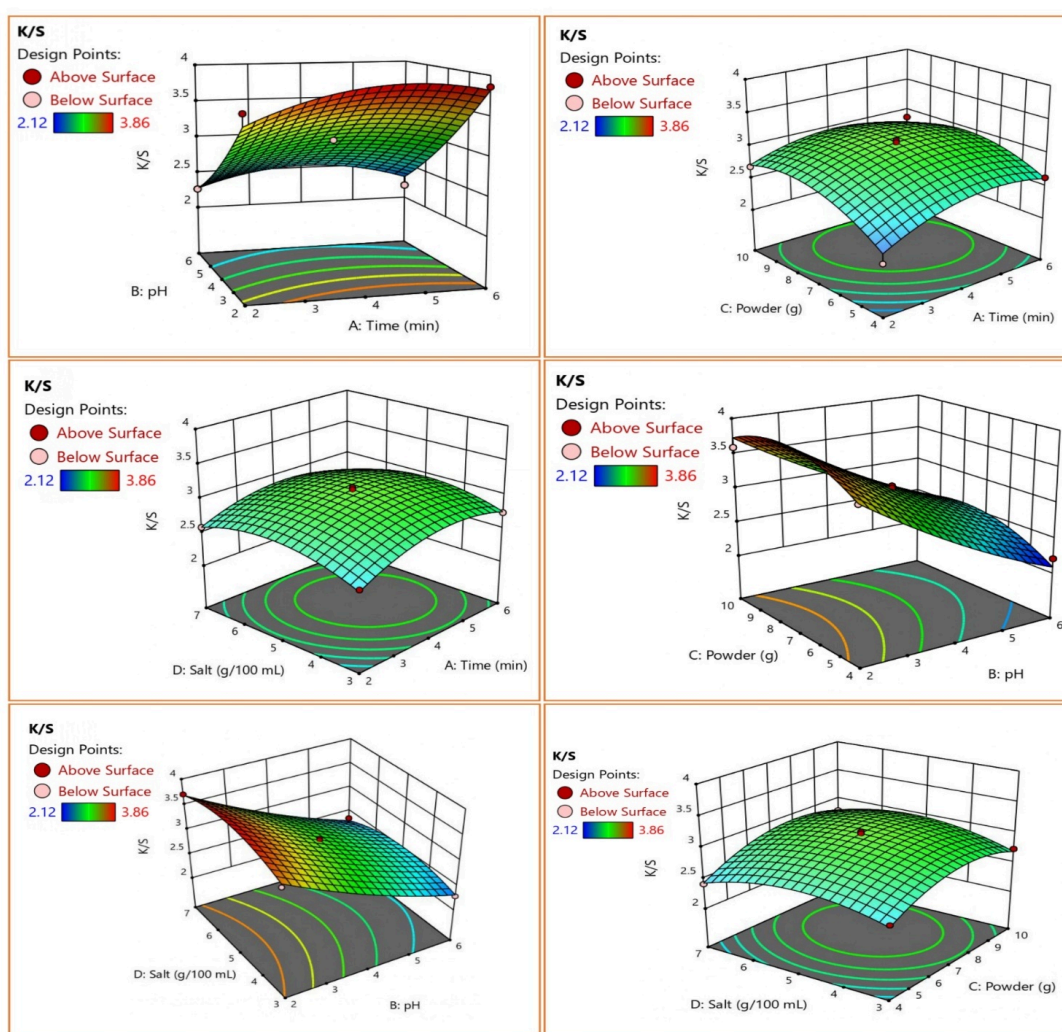


Figure 4. 3D response surface plots showing the interaction effects of microwave irradiation time (A), pH (B), powder amount (C), and salt concentration (D) on the color strength (K/S) of cotton fabric under microwave-assisted dyeing conditions: (a) A–B; (b) A–C; (c) A–D; (d) B–C; (e) B–D; and (f) C–D.

The response surface for time and powder amount revealed that K/S increased with increasing powder amount up to an intermediate-to-high level, after which the gain became limited. This indicates that the dye availability increases with the higher concentration of the dye source, the further the response is limited due to the limited dyeing capacity of the fabric being finite.

In the case of time and salt concentration, the response surface had a moderate range of variation, which proved the relatively weak impact of salt concentration. This phenomenon was also observed in the pH-salt interaction plot, with pH being the main variable with salt only producing slight variations in K/S.

A clear gradient was observed in the pH-powder surface where the highest K/S was found at low pH and moderate-high powder concentration. Similarly, the interaction between the powder and salt showed that K/S attained the favorable region at moderate concentrations of both variables as opposed to the extreme values. On the whole, response surfaces demonstrate that the dyeing system is controlled by the major factor, which is pH, then amount of powder and irradiation period having a relatively small influence and salt impact being of comparative insignificance.

Optimization of Dyeing Conditions

Numerical optimization was performed with the objective of maximizing K/S within the selected factor ranges. The optimum conditions predicted by the model were:

- Microwave irradiation time: 4.6575 min
- pH: 2.0000
- Powder amount: 7.5159 g
- Salt concentration: 5.1502 g/100 mL

Under these conditions, the model predicted a maximum K/S value of 3.9289, with an overall desirability of 0.96. The desirability value indicates that the optimization objective was fully satisfied within the design space. The optimized result agrees well with the trends observed in the main-effect and response surface analyses. In particular, the optimum was located at low pH, moderate irradiation time, moderate-to-high powder amount, and intermediate salt concentration. This confirms that maximum color strength is not achieved at the extreme values of all factors, but rather through a balanced combination of dyeing conditions. Although pH value 2 represents a strongly acidic condition, the dyeing duration in this study was relatively short and conducted under controlled laboratory conditions. Therefore, no visible fiber damage, fabric distortion, or handling deterioration was observed during or after dyeing. Moreover, previous studies on cotton and other

cellulosic substrates under acidic dyeing conditions (pH 2–3) have also reported improved dye fixation and acceptable dyeing performance when exposure time was properly controlled [47, 48]. Nevertheless, prolonged exposure to highly acidic environments may potentially affect the structural integrity of cellulose-based fibers, and therefore future studies should include tensile strength and morphological analyses to comprehensively evaluate substrate durability under optimized dyeing conditions.

Validation of the Optimized Condition

Model validation should be carried out by performing a confirmatory dyeing experiment under the optimized condition and comparing the experimentally obtained K/S with the predicted value of 3.9289. The predicted and experimental K/S values, together with the percentage error, are presented in Table 5. A close agreement between the predicted and experimental results, preferably with a low percentage error, would confirm the robustness and practical applicability of the developed model.

Table 5. Validation of the optimized dyeing condition for color strength (K/S).

Response	Predicted value	Experimental value	Error (%)	Sample image
K/S	3.9289	3.8829	1.17	

Color Fastness Results

The color fastness properties were evaluated only for the optimized dyed sample, developed under the predicted conditions obtained from the RSM model. The fabric was post-mordanted using henna (*Lawsonia inermis*) extract (10% owf), and fastness tests were conducted according to standard ISO methods, namely ISO 105-C06 for washing fastness, ISO 105-X12 for rubbing fastness, ISO 105-E04 for perspiration fastness, and ISO 105-B02 for light fastness using a xenon arc lamp. The results are summarized in Table 6.

Table 6. Color Fastness Ratings of Neem-Dyed Hemp/Cotton Fabric Using Henna Bio-Mordant

Fastness Property	Test Condition	Rating	Assessment
Washing Fastness	Color Change	4–5	Good to very good
	Staining (Cotton)	4–5	Good to very good
	Staining (Wool)	3–4	Fair to good

Fastness Property	Test Condition	Rating	Assessment
Rubbing Fastness	Dry	4–5	Good to very good
	Wet	3–4	Fair to good
Perspiration Fastness	Acidic	4	Good
	Alkaline	4–5	Good to very good
Light Fastness	Xenon arc lamp	4–5 (out of 8)	Good to very good

The optimized dyed cloth showed repeatedly good to very good fastness properties, which suggests that the conditions predicted by RSM are not only efficient to maximize the strength of the color effect but also provide stability to dyes. The good washing fastness values (4-5) indicate robust fixation of the colorants of the neem-derived tannin onto the hemp/cotton substrate. This may be explained by the increased dye fiber interactions at optimized acid conditions, as well as by the development of stable complexes due to the presence of the phenolic component of henna bio-mordant [41].

Rubbing fastness performed well in dry conditions (4-5) and slightly lower levels in wet conditions (3-4), which is characteristic of natural dye systems because surface fixation of freely floating dye molecules is limited [49]. However, the values obtained are acceptable textile values.

Fastness in perspiration during acidic and alkaline environments proved to be consistent in color retention (rating 4-5), which validates the ability of the dyed material to be used in end-use applications with human contact. On the same note, the light fastness rating (4-5) means that it has average resistance to photodegradation. This enhancement can be linked to the UV-absorbing properties of the lawsone contained in henna that helps to improve the photostability of the dyed fabric [50, 51].

Altogether, the results of the fastness indicate the success of the optimized microwave-assisted dyeing technique paired with the use of henna bio-mordanting, as the newly created model has the ability to balance the color efficiency and performance viability. These results confirm the practicability of the suggested eco-friendly dyeing method to apply to the practice of textile processing.

CONCLUSION

This work was able to illustrate a sustainable and effective solution to dyeing of hemp/cotton blended fabrics by incorporating the concept of microwave-assisted dyeing and response surface methodology (RSM). A Box-Behnken design was used to allow systematic analysis of interactive effects of key process parameters, which resulted in the creation of statistically sound and predictive quadratic color strength (K/S) model.

The model created had a high coefficient of determination ($R^2 = 0.9814$), a small coefficient of variation (3.72%), and non-significant lack-of-fit, which proved its good predictability of the process and its optimization. The most affected variable was found to be pH, then the concentration of dye powder, and microwave irradiation time, whilst salt concentration had a relatively small impact. The findings were a clear indication that acidic conditions have a significant positive influence on the dye-fiber interactions, especially in the case of heterogeneous substrate hemp/cotton. The optimum K/S at a microwave irradiation time of about 4.66 min, pH 2.0, dye concentration of 7.52 g and salt concentration of 5.15 g/100 mL with a high degree of desirability of 0.96 was obtained through numerical optimization. The predictive power of the model was successfully validated experimentally with a very small deviation (error 1.17%) between values predicted and observed, and this indicates that the model shows good agreement and can be applied practically.

Besides the enhancement of color strength, the optimized dyed fabric had good to very good fastness during washing, rubbing, perspiration, and light exposure which validated the stability of the dyeing system. Henna application as a natural bio-mordant also added to an increase in fixation and functional performance and retained the eco-friendly character of the process. Previous literature showed the possible antimicrobial potential of the dyed fabrics, which indicates their appropriateness in the development of value-added textile applications.

In general, this article offers an all-round and prospective system of natural dyeing optimization of mixed fabrics through microwave-based technologies. It concerns major shortcomings of traditional natural dyeing, like inadequate reproducibility, low color strength, and minimizes processing time and the use of chemicals to a considerable extent. The results are valuable towards eco-friendly textile processing and provide a plausible way to promote the usage of renewable-based eco-friendly dyeing technology in the industry.

Author contributions

Tanzeena Refat Tumpa: Conceptualization, methodology, validation, resources, software, writing original draft;

Md. Nasir Uddin: Conceptualization, methodology, validation, formal analysis, data curation, writing original draft;

Sanjida Jannat Mim: Laboratory work, formal analysis;

Md. Nazmul Islam: Methodology, validation, formal analysis, curation;

Badhon C. Mazumder: Laboratory work, Methodology, formal analysis;

Md. Lutfor Rahman: Testing, Methodology, formal analysis;

Mohammad Rashel Hawlader: Conceptualization, methodology, overall supervision, Writing, reviewing and editing. All authors read and approved the final manuscript.

Conflicts of Interest

The author declares no conflict of interest.

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Data availability statement

Data will be made available on request.

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Additional information

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