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Optimization of Equipment and Process Parameters for Ultrasound-Assisted Sustainable Natural Dyeing

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ABSTRACT

Natural dyes have attracted increasing attention as environmentally friendly alternatives to synthetic colorants in textile coloration. In this study, an ultrasound-assisted natural dyeing process using beetroot (Beta vulgaris L.) extract was developed for cotton fabrics, and the effects of ultrasonic power, dyeing temperature, and dyeing time on the color strength of dyed fabrics were systematically investigated. Response surface methodology based on a Box–Behnken experimental design was employed to evaluate the influence of these parameters and to determine the optimal dyeing conditions. The experimental results showed that the color strength (K/S value) of the dyed fabrics varied from 10.87 to 14.73 within the investigated experimental range. Analysis of variance indicated that the regression model was statistically significant and that ultrasonic power had the most pronounced influence on dye uptake. The optimal dyeing conditions were determined as ultrasonic power 230 W, dyeing temperature 70 °C, and dyeing time 60 min. Under these conditions, the predicted K/S value was 14.80, while the experimentally obtained value was 14.72, demonstrating good agreement with the model prediction. The results confirm that ultrasound-assisted dyeing combined with statistical optimization provides an effective approach for improving natural dyeing efficiency and promoting sustainable textile coloration technologies.

KEYWORDS
natural dye, ultrasonic dyeing, cotton fabric, response surface methodology, color strength

INTRODUCTION

Natural dyes have received increasing attention in recent years due to growing concerns about environmental pollution and sustainability in the textile industry. Compared with synthetic dyes, natural dyes are biodegradable, renewable, and generally exhibit lower environmental impact during production and application processes. As a result, natural dyeing technologies have been widely explored as environmentally friendly alternatives for textile coloration [1-3].

Among various natural dye sources, beetroot (Beta vulgaris L.) has attracted considerable interest because it contains betalain pigments, which are responsible for its characteristic red–purple color. Betalains are water-soluble nitrogen-containing pigments that exhibit good coloring properties and have been widely used as natural colorants in food and textile applications [4]. However, conventional natural dyeing processes often suffer from several limitations, including low dye extraction efficiency, poor dye penetration into fibers, and relatively long processing times [5].

Ultrasound-assisted processing has emerged as an effective technique for enhancing mass transfer in various chemical and textile processes. The cavitation effect generated by ultrasonic waves produces localized turbulence and microjets in liquid media, which can significantly improve the extraction of natural compounds and the diffusion of dye molecules into textile fibers [6-8]. Previous studies have shown that ultrasonic irradiation can enhance dye uptake, reduce processing time, and improve the overall efficiency of natural dyeing processes [7,8].

In addition to process intensification techniques, statistical optimization methods are increasingly used to optimize textile processing parameters. Response surface methodology (RSM) is a powerful statistical tool that can evaluate the individual and interaction effects of multiple variables and determine optimal operating conditions with a relatively small number of experiments [9,10]. Among different RSM experimental designs, the Box–Behnken design (BBD) is widely applied in process optimization because it requires fewer experimental runs while providing reliable prediction of quadratic response surfaces.

Therefore, the objective of this study was to develop an ultrasound-assisted natural dyeing process using beetroot extract for cotton fabrics and to optimize the dyeing parameters using response surface methodology. The effects of ultrasonic power, dyeing temperature, and dyeing time on the color strength of dyed fabrics were systematically investigated. A Box–Behnken experimental design was employed to establish a regression model and identify the optimal dyeing conditions within the investigated ultrasound-assisted dyeing system.

MATERIALS AND METHODS

Materials

The natural dye used in this study was extracted from dried beetroot (Beta vulgaris L.), which was obtained from a local agricultural supplier and used as the raw material for natural dye extraction. Beetroot contains betalain pigments that are widely used as natural colorants. The plant materials were thoroughly washed with distilled water to remove surface impurities and then cut into small pieces. The samples were dried in an oven at 50 °C for 48 h until a constant weight was obtained. The dried materials were subsequently ground into fine powder using a laboratory grinder and stored in airtight containers prior to dye extraction. Such pretreatment of plant materials improves extraction efficiency and ensures a stable dye composition during the extraction process.

Plain-woven cotton fabric was selected as the dyeing substrate due to its widespread application in textile products and its good affinity for natural dyes. The cotton fabric was supplied by a local textile manufacturer. Cotton fibers contain abundant hydroxyl groups in their cellulose structure, which facilitate interactions with dye molecules during the dyeing process. The structural parameters of the cotton fabric used in this study are summarized in Table 1.

Table 1. Structural parameters of cotton fabric

Parameter	Value
Fabric structure	Plain woven
Areal density	150 g/m²
Warp density	38 ends/cm
Weft density	32 picks/cm

The structural characteristics of the cotton fabric influence dye uptake during the dyeing process. The plain-woven structure provides a stable fabric configuration with interlacing warp and weft yarns, which affects the accessibility of dye molecules to the fiber surface and the diffusion behavior of dyes within the fabric structure.

Before dyeing, the cotton fabrics were scoured in a nonionic detergent solution at 60 °C for 30 min with a liquor ratio of 1:30 in order to remove natural impurities and improve dye absorption. After scouring, the fabrics were rinsed thoroughly with distilled water and air-dried at room temperature.

All chemicals used in this study were of analytical grade and were used without further purification. Distilled water was used throughout all experimental procedures.

Ultrasonic Dye Extraction

Natural dye extraction was performed using an ultrasonic bath operating at a frequency of 40 kHz. The dried beetroot powder (Beta vulgaris L.) prepared in Section 2.1 was used as the raw material for dye extraction.

The beetroot powder was mixed with distilled water at a liquid–solid ratio of 20:1 (mL/g) to prepare the extraction suspension. The suspension was transferred into a glass beaker and placed in the ultrasonic bath to facilitate the release of dye compounds from the plant materials. To ensure consistent dye composition for all dyeing experiments, the extraction parameters were kept constant throughout the study.In this work, ultrasonic extraction was used primarily to improve the release efficiency of betalain pigments from beetroot tissues and to provide a stable and reproducible dye liquor for the subsequent dyeing experiments. Since the main objective of this study was to optimize the ultrasonic dyeing stage, the extraction conditions were fixed throughout the experimental design in order to minimize variability arising from dye preparation.

The extraction conditions were as follows:

Ultrasonic power: 200 W

Extraction temperature: 70 °C

Extraction time: 40 min

Ultrasonic-assisted extraction has been widely applied in the recovery of natural compounds from plant materials due to its ability to enhance extraction efficiency [11]. Ultrasonic treatment promotes the release of dye compounds from plant tissues and improves mass transfer between the solid material and the solvent. After extraction, the dye solution was filtered using Whatman No. 1 filter paper to remove plant residues. The filtrate was concentrated using a rotary evaporator and subsequently dried in an oven at 60 °C to obtain crude dye extract powder. The crude dye extract was stored in sealed containers prior to use.

For dyeing experiments, the dye extract powder was dissolved in distilled water to prepare dye solutions with a concentration of 5% owf (on the weight of fabric).

Ultrasonic Dyeing Process

The dyeing experiments were conducted using an ultrasonic dyeing system consisting of an ultrasonic bath and a temperature control unit. The ultrasonic bath operated at a frequency of 40 kHz with adjustable ultrasonic power ranging from 150 to 300 W. The bath capacity was 6 L, and the dye bath temperature was controlled using a digital thermostat with an accuracy of ±1 °C.

Cotton fabric samples weighing 5 g were dyed in the natural dye solution prepared from the ultrasonic extraction process. Unlike the extraction step, which was used to obtain a consistent dye solution, ultrasonic irradiation during dyeing was applied to improve dye dispersion in the bath and to enhance mass transfer between the dye molecules and the cotton fiber surface. The basic dyeing conditions used in this study are summarized in Table 2.

Table 2. Basic dyeing conditions

Parameter	Value
Liquor ratio	1:30
Dye concentration	5% owf (on the weight of fabric)
Fabric weight	5 g

Three dyeing parameters were investigated in this study: ultrasonic power (150–300 W), dyeing temperature (60–80 °C), and dyeing time (30–90 min). These parameters were selected based on preliminary experiments and previous studies on ultrasound-assisted dyeing processes.

During dyeing, the fabric samples were fully immersed in the dye bath and subjected to ultrasonic irradiation under controlled temperature conditions. This treatment promoted dye dispersion and facilitated dye transfer from the dye bath to the fiber surface.

After dyeing, the fabric samples were removed from the dye bath, rinsed thoroughly with distilled water to remove unfixed dye molecules, and then air-dried at room temperature prior to color measurement.

Each experimental run in the Box–Behnken experimental design was performed in triplicate, and the reported results represent the average of three independent measurements. The purpose of this study was to optimize the operating parameters of the ultrasound-assisted dyeing process. Therefore, a conventional heating control group was not included in the present experimental design. A direct comparison between conventional heating and ultrasonic dyeing under identical temperature and time conditions will be considered in future work.

Color Measurement

The color strength of the dyed fabrics was measured using a UV–Vis spectrophotometer (UV-2600, Shimadzu, Japan) equipped with an integrating sphere for diffuse reflectance measurement of opaque fabric samples under standard illumination conditions with a D65 light source and a 10° standard observer. Before measurement, the instrument was calibrated using the standard white reference supplied by the manufacturer.

Reflectance spectra of the dyed fabrics were recorded in the wavelength range of 400–700 nm. The color strength of the dyed samples was evaluated using the Kubelka–Munk equation, which is commonly used to characterize dye uptake in textile coloration [12]:

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

where R represents the reflectance of the dyed fabric at the wavelength of maximum absorption.

The K/S value is proportional to the concentration of dye molecules absorbed in the fiber structure. Therefore, a higher K/S value indicates stronger coloration and higher dye uptake by the fabric.

To ensure measurement reliability, color measurements were conducted at three different positions on each fabric sample, and the average value was reported as the final result.

Experimental Design and Statistical Analysis

Response surface methodology (RSM) based on a Box–Behnken experimental design (BBD) was employed to investigate the effects of dyeing parameters on the color strength of dyed fabrics.

Three independent variables were selected for the experimental design: ultrasonic power (A), dyeing temperature (B), and dyeing time (C). The coded and actual levels of these variables are presented in Table 3.

Table 3. Coded and actual levels of experimental variables

Factor	Symbol	Low (-1)	Center (0)	High ($+1$)
Ultrasonic power (W)	A	150	225	300
Dyeing temperature (°C)	B	60	70	80
Dyeing time (min)	C	30	60	90

According to the Box–Behnken design, a total of 15 experimental runs were conducted to evaluate the individual and interaction effects of the selected variables. Each design point was carried out in triplicate as three independent dyeing experiments. For each dyed sample, color measurements were taken at three different positions, and the K/S values reported in Table 4 are presented as mean ± standard deviation. The mean K/S value was used as the response variable for statistical analysis. The experimental design matrix and corresponding experimental results are presented in Table 4.

Table 4. Box–Behnken experimental design matrix and experimental results

Run	A: Ultrasonic power (W)	B: Dyeing temperature (°C)	C: Dyeing time (min)	K/S (mean ± SD)
1	150	60	60	11.21 ± 0.09
2	300	60	60	12.82 ± 0.11
3	150	80	60	11.84 ± 0.10
4	300	80	60	13.75 ± 0.12
5	150	70	30	10.87 ± 0.08

Table 4. Box–Behnken experimental design matrix and experimental results

Run	A: Ultrasonic power (W)	B: Dyeing temperature (°C)	C: Dyeing time (min)	K/S (mean ± SD)
6	300	70	30	12.19 ± 0.10
7	150	70	90	11.82 ± 0.09
8	300	70	90	13.75 ± 0.11
9	225	60	30	11.28 ± 0.07
10	225	80	30	12.58 ± 0.10
11	225	60	90	11.71 ± 0.09
12	225	80	90	13.09 ± 0.11
13	225	70	60	14.26 ± 0.08
14	225	70	60	14.73 ± 0.09
15	225	70	60	14.33 ± 0.07

Note: Each design point was carried out in triplicate, and the K/S values are presented as mean ± standard deviation (SD). Runs 13–15 represent replicated center points used to estimate experimental error.

The relationship between the response variable and the independent variables was described using a second-order polynomial regression model:

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

where Y represents the predicted response value (K/S value), X_i represents the coded independent variables, and β represents the regression coefficients of the model.

Statistical analysis of the experimental data was performed using Design-Expert software (Version 13, Stat-Ease Inc., Minneapolis, USA). The significance of the regression model and individual terms was evaluated using analysis of variance (ANOVA), and a p -value < 0.05 was considered statistically significant.

The adequacy of the regression model was evaluated using the coefficient of determination (R^2), adjusted coefficient of determination ($Adj - R^2$), and lack-of-fit tests. Response surface plots and contour plots were generated to visualize the interaction effects among the dyeing parameters and to determine the optimal dyeing conditions.

RESULTS AND DISCUSSION

Regression Model Analysis

The experimental results obtained from the Box–Behnken design showed that the color strength (K/S value) of the dyed cotton fabrics varied significantly under different combinations of ultrasonic power, dyeing temperature, and dyeing time. Within the investigated experimental range, the K/S values varied from 10.87 to 14.73, indicating that the selected process parameters had a substantial influence on dye uptake. These results indicate that, within the investigated ultrasound-assisted dyeing system, ultrasonic power, dyeing temperature, and dyeing time had significant effects on dye uptake and color strength. Based on the experimental data, a quadratic regression model was developed to describe the relationship between the dyeing parameters and the response variable. The regression equation in terms of coded variables can be expressed as:

$$K/S = 14.35 + 0.82A + 0.56B + 0.41C - 0.21AB - 0.18AC - 0.14BC - 0.67A^2 - 0.52B^2 - 0 \quad (3)$$

where A, B, and C represent ultrasonic power, dyeing temperature, and dyeing time, respectively.

The positive coefficients of the linear terms indicate that increasing ultrasonic power, dyeing temperature, and dyeing time generally enhances dye uptake within the investigated range. However, the negative coefficients of the quadratic terms suggest that excessively high parameter levels may lead to reduced dyeing efficiency due to dye aggregation or saturation of adsorption sites on the fiber surface.

The adequacy of the developed model was evaluated using statistical indicators. The regression model exhibited a high coefficient of determination ($R^2 = 0.973$) and an adjusted coefficient of determination (Adj-$R^2 = 0.926$), indicating good agreement between the predicted and experimental results.

The relationship between the predicted and experimental K/S values is illustrated in Figure 1. The data points are closely distributed along the diagonal line, indicating good agreement between the predicted values and the experimental observations. The close correspondence suggests that the developed regression model can reliably describe the dyeing process within the investigated parameter range.

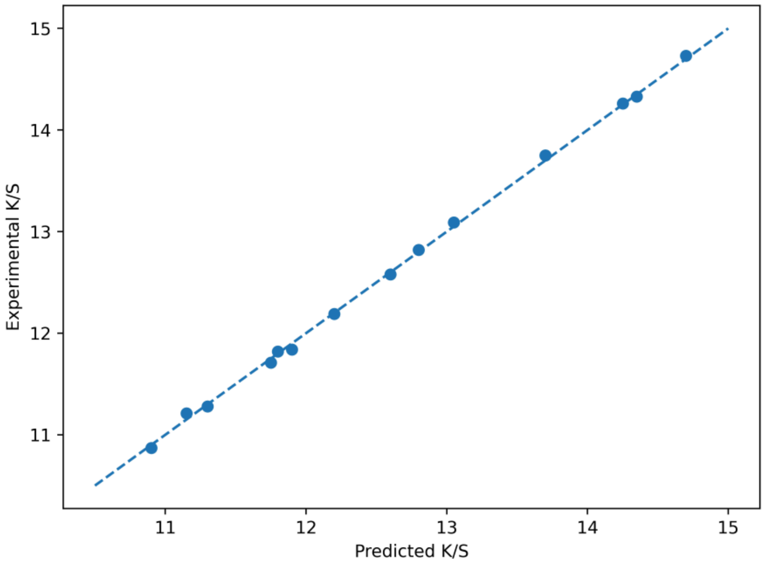

Figure 1. Comparison between predicted and experimental K/S values obtained from the regression model

Analysis of Variance (ANOVA)

Analysis of variance (ANOVA) was conducted to evaluate the statistical significance of the regression model and to determine the relative influence of the investigated parameters. The ANOVA results for the quadratic model are summarized in Table 5.

Table 5. Analysis of variance (ANOVA) for the quadratic regression model

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	23.280	9	2.587	20.36	0.0020
A	5.865	1	5.865	49.94	0.0009
B	2.333	1	2.333	19.86	0.0067
C	1.488	1	1.488	12.67	0.0162
AB	0.036	1	0.036	0.31	0.6032
AC	0.093	1	0.093	0.79	0.4142
BC	0.002	1	0.002	0.01	0.9116
A^2	3.776	1	3.776	32.15	0.0024
B^2	3.720	1	3.720	31.67	0.0025
C^2	5.967	1	5.967	50.81	0.0008
Residual	0.587	5	0.117		
Lack of fit	0.411	3	0.137	2.24	0.204
Pure error	0.176	2	0.088		
Total	23.867	14			

The regression model was found to be statistically significant (p = 0.0020), suggesting that the model effectively describes the relationship between the dyeing parameters and the response variable. The lack-of-fit test was not significant (p = 0.204), indicating that the regression model adequately fits the experimental data.

Among the investigated variables, ultrasonic power (A) exhibited the highest F-value, indicating that it had the most significant effect on dye uptake. This result is consistent with the role of ultrasonic irradiation in enhancing dye diffusion and dye–fiber interaction, as reported in previous studies on ultrasound-assisted dyeing processes [11,12].

Dyeing temperature (B) also showed a significant effect on color strength. Increasing temperature enhances the mobility of dye molecules and promotes swelling of cotton fibers, which facilitates dye penetration into the fiber matrix. In addition, higher temperatures can reduce the viscosity of the dye bath and increase the diffusion coefficient of dye molecules. Similar temperature-dependent dye uptake behavior has been reported for natural dyeing systems in earlier studies [13].

Dyeing time (C) also played an important role in the dyeing process. Increasing dyeing time allows dye molecules sufficient time to diffuse into the fiber structure and reach adsorption equilibrium. However, after a certain period, the increase in color strength becomes less significant because most adsorption sites on the fiber surface are already occupied. Similar adsorption behavior has been reported in previous studies on natural dyeing processes [14].

Because no SEM characterization was performed in the present study, possible microstructural changes of the cotton fiber surface under ultrasonic treatment were not directly evaluated. Therefore, the present discussion focuses on macroscopic dyeing performance and mass-transfer-related effects rather than confirmed surface morphology changes.

These results indicate that ultrasonic power, dyeing temperature, and dyeing time all contribute significantly to dye uptake and should be carefully optimized to achieve the best dyeing performance.

Response Surface Analysis

Response surface methodology was employed to visualize the effects of the dyeing parameters on color strength. Three-dimensional response surface plots and the corresponding contour plots were generated based on the developed regression model.

Figure 2 shows the contour plot describing the combined influence of ultrasonic power and dyeing temperature while the dyeing time was maintained at its center level. As shown in the figure, increasing ultrasonic power generally enhanced the color strength of the dyed fabrics. This behavior can be attributed to the increase in cavitation intensity generated in the dye bath under ultrasonic irradiation. Stronger cavitation effects improve dye dispersion and accelerate mass transfer in the liquid medium, thereby facilitating the diffusion of dye molecules into cotton fibers.

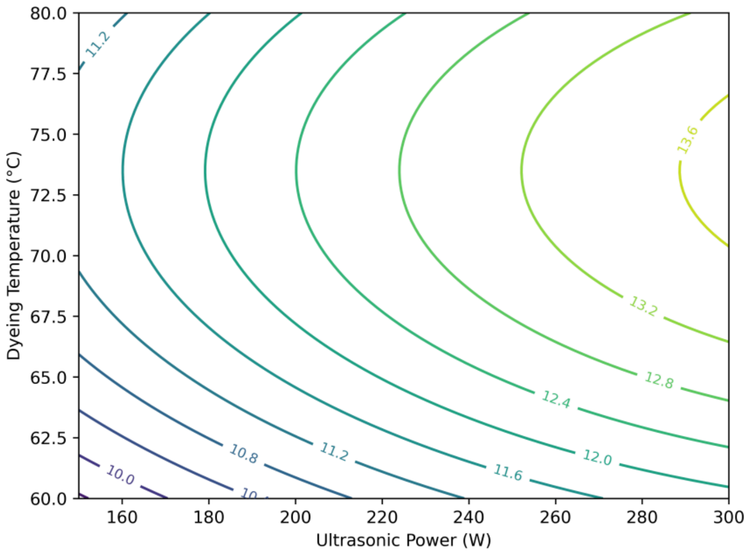

Figure 2. Contour plot showing the interaction effect of ultrasonic power and dyeing temperature on color strength (dyeing time = 60 min)

At the same time, increasing dyeing temperature improved dye uptake due to enhanced molecular mobility and fiber swelling. Higher temperatures allow dye molecules to diffuse more easily into the internal structure of the fiber, which contributes to higher K/S values. Similar effects of ultrasonic irradiation and temperature on dye uptake have been reported in previous ultrasound-assisted dyeing studies [15].

It should also be noted that the natural dye extracted from beetroot mainly contains betalain pigments, which are known to be sensitive to elevated temperatures. Excessively high dyeing temperatures may lead to partial degradation of betalain compounds, which may limit further increases in color strength. Therefore, moderate dyeing temperatures are favorable for achieving a balance between enhanced dye diffusion and pigment stability.

Figure 3 illustrates the effect of dyeing temperature and dyeing time when the ultrasonic power was fixed at its center level. The results indicate that moderate levels of temperature and dyeing time resulted in higher K/S values. When the dyeing time was excessively prolonged, the increase in color strength became less significant. This phenomenon can be attributed to the gradual saturation of available dye adsorption sites on the fiber surface.

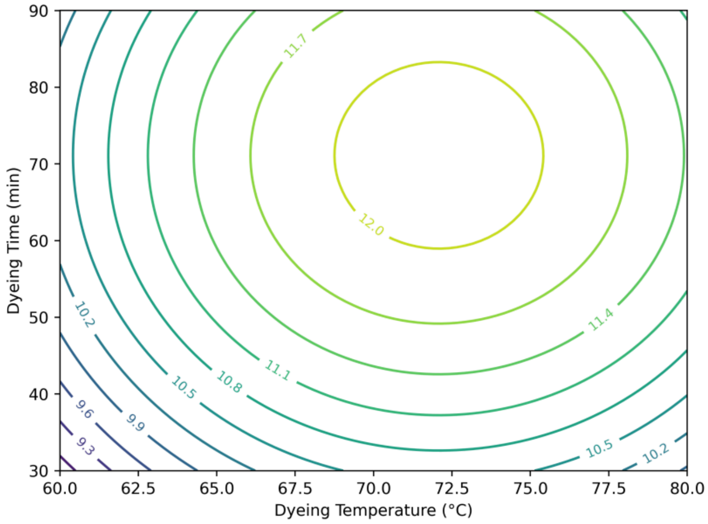

Figure 3. Contour plot illustrating the interaction effect of dyeing temperature and dyeing time on K/S value (ultrasonic power = 225 W)

Overall, the response surface analysis indicates that dyeing performance is mainly governed by the individual effects of ultrasonic power, dyeing temperature, and dyeing time, while the interaction effects among the variables are relatively limited within the investigated experimental range.

Optimization of Dyeing Parameters

Based on the regression model and response surface analysis, the optimal dyeing conditions were predicted using the numerical optimization module of the RSM model. The optimal dyeing parameters were determined as follows: ultrasonic power 230 W, dyeing temperature 70 °C, and dyeing time 60 min. Under these conditions, the predicted K/S value was 14.80, while the experimentally obtained value was 14.72, showing good agreement with the model prediction.

Compared with the lowest parameter combination investigated in this study (ultrasonic power 150 W, dyeing temperature 60 °C, dyeing time 30 min), the optimized conditions increased the K/S value from 10.87 to 14.72, corresponding to an improvement of approximately 35%:

$$Improvement(\%) = \frac{14.72 - 10.87}{10.87} \times 100\% \approx 35\% \tag{4}$$

These results demonstrate that ultrasonic irradiation combined with statistical optimization can significantly enhance dye uptake. Similar improvements in color strength have also been reported in previous studies on ultrasound-assisted natural dyeing processes [16].

Overall, the findings confirm that response surface methodology is an effective tool for identifying optimal operating conditions in ultrasound-assisted natural dyeing. The results also support the observations from the response surface analysis, indicating that ultrasonic power, dyeing temperature, and dyeing time each influence dye uptake and collectively determine the optimum dyeing region.

CONCLUSIONS

In this study, an ultrasound-assisted natural dyeing process using beetroot (Beta vulgaris L.) extract was developed to improve the efficiency of sustainable textile coloration. The effects of key dyeing parameters, including ultrasonic power, dyeing temperature, and dyeing time, on the color strength of cotton fabrics were systematically investigated using response surface methodology based on a Box–Behnken experimental design.

The experimental results showed that the color strength (K/S value) of the dyed fabrics varied from 10.87 to 14.73 within the investigated experimental range. The results showed that, within the investigated ultrasound-assisted dyeing system, ultrasonic power, dyeing temperature, and dyeing time significantly influenced dye uptake and color strength.

Analysis of variance indicated that the regression model was statistically significant and adequately described the relationship between dyeing parameters and color strength. Among the investigated variables, ultrasonic power exhibited the most significant influence on dye uptake, followed by dyeing temperature and dyeing time.

Response surface analysis revealed that moderate levels of dyeing temperature and dyeing time combined with relatively high ultrasonic power produced higher color strength values. Excessively high processing conditions did not further improve dye uptake due to the saturation of dye adsorption sites and possible degradation of betalain pigments at elevated temperatures.

The response surface model predicted that the optimal dyeing conditions within the investigated range were ultrasonic power 230 W, dyeing temperature 70 °C, and dyeing time 60 min, with a predicted K/S value of 14.80. A validation experiment conducted under these conditions yielded a K/S value of 14.72, which was in close agreement with the model prediction and comparable to the highest values observed in the experimental runs. This result supports the reliability of the developed regression model.

Because no SEM characterization was performed in the present study, possible microstructural changes of the cotton fiber surface under ultrasonic treatment were not directly evaluated. Overall, the results demonstrate that ultrasound-assisted dyeing combined with statistical optimization provides an effective approach for optimizing dyeing conditions and improving the performance of sustainable natural dyeing.

Availability of Data and Materials

The datasets used and/or analysed during the current study were available from the corresponding author on reasonable request.

Author Contributions

Baojuan Xin and Yinghui Guo designed the study; all authors conducted the study; Shujun Yan and Yan Yuan collected and analyzed the data. Yan Yuan and Baojuan Xin participated in drafting the manuscript, and all authors contributed to critical revision of the manuscript for important intellectual content. All authors gave final approval of the version to be published. All authors participated fully in the work, took public responsibility for appropriate portions of the content, and agreed to be accountable for all aspects of the work in ensuring that questions related to the accuracy or completeness of any part of the work were appropriately investigated and resolved.

Conflict of Interest

The authors declare no conflict of interest.

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