



Original Research Article

Sustainable Cotton Dyeing with Banana Floral Stem Extract using Microwave Irradiation and Chitosan Optimised by A Response Surface Methodology

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ABSTRACT

Natural dyes offer a sustainable alternative to synthetic dyes, addressing environmental concerns in textile production. Currently, the dyeing and finishing stages typically consume large amounts of water, energy, and chemicals. To enhance sustainability, a microwave-assisted dyeing process using chitosan and banana floral stem extract has been developed for cotton fabrics. This process was optimised using Response Surface Methodology and Box-Behnken Design to minimise lightness (L^*) and maximise colour difference (ΔE). The optimal conditions include a radiant power of 554.12 W, a radiation time of 7.10 min, and a chitosan concentration of 0.80%. These conditions result in a lightness value of 62.04, a colour difference of 35.36, and a desirability coefficient of 0.856. Cotton samples treated under these conditions exhibited superior colourfastness compared to conventional methods, with ratings of 4 (good) for washing, 4-5 (good to excellent) for lightfastness, and 4-5 (good to excellent) for perspiration. The colourfastness scale ranges from 1 (very poor) to 5 (excellent), indicating the level of resistance to fading or colour change. This study demonstrates the eco-friendly and efficient potential of microwave-assisted dyeing with chitosan, promoting sustainable textile practices.

KEYWORDS

Microwave irradiation, Chitosan, Banana floral stem, Box-Behnken Design, Lightness, Colour difference.

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INTRODUCTION

The utilisation of natural dyes as replacements for synthetic counterparts in the textile sector is progressively burgeoning, driven by the goal of mitigating environmental consequences by adopting secure and suitable alternative materials [1]. Natural dyes derived from renewable sources, like plants, algae, and fungi, are valued for their biodegradability, lack of toxicity and carcinogenicity, and environmental compatibility. For example, *Lawsonia* from henna offers an environmentally sustainable alternative to synthetic dyes, as it does not require costly effluent treatment systems [2]. Similarly, a dye derived from green algae is biodegradable and free from harmful chemicals, making it a sustainable choice for textile industries [3]. The fungus *Talaromyces verruculosus* also produces a natural pigment that is non-cytotoxic and safe for use, providing an eco-friendly dyeing solution [4]. Furthermore, methylating brazilin from second wood with dimethyl carbonate (DMC) enhances its colour stability and resistance to pH changes, while DMC's environmentally benign properties align with sustainable chemistry principles [5]. Other source of natural dyes are banana plants, which are classified as biowaste following the harvest of the fruit. Phytochemical analysis indicates that the aqueous extract of the banana floral stem (BFS) from the Ambon variety (*Musa sapientum*) comprises chromophore compounds potential as natural dyes, including condensed tannins, flavonoids, anthraquinones, and anthocyanins, rendering it a viable option for multifunctional textile applications [6]. The findings further validated that BFS can yield significant colour strength ($K/S = 0.71$) in cotton fabric [7].

Another concern for natural dyes is the weak adherence to the fabrics. For example, the most famous natural fibre, cotton, has a high density of negatively charged groups from exclusively hydroxyl groups and ether bonds, which makes it difficult to dye with natural dyes [8]. Natural dyes in aqueous solutions are repelled by these charges, resulting in weak colourfastness and poor colour absorption [9]. Numerous techniques, including surface modification using metal and biological mordant, have been carried out to enhance cotton fabric dyeing performance [8]. Amongst these, biological mordants, such as chitosan, are used to modify the surface of cotton to increase its affinity for anionic dyes. Chitosan polysaccharides are highly recommended among natural materials due to their distinctive chemical and biological features, as well as their accessibility to industrial applications [10]. Silk fabrics coloured with natural *Rubia cordifolia* L. colours using the pad-dry method and treated with chitosan have shown better colourfastness of 5 (outstanding) to wash, rub, and light [11]. This cationisation technique will be applied in the dyeing process for BFS on cotton fabrics.

Furthermore, research on applying safe radiation technology to the environment is being developed for dyeing. Gamma radiation at 30 kGy enhances the dyeing affinity of cotton fabric with lutein from marigold, improving colour strength and fastness, particularly when tannic acid is used as a pre-mordant and copper as a post-mordant [12]. Similarly, electron beam irradiation efficiently modifies synthetic fabrics like polypropylene and polyester by introducing functional groups that enhance natural dye absorption and colour retention [13]. Ultraviolet radiation also improves lutein uptake, increasing colour strength and fade resistance, especially when applied for 90 min under optimal conditions [14]. Additionally, plasma treatment enhances fabric properties, including antibacterial activity, while reducing the use of hazardous chemicals, contributing to more sustainable dyeing processes [15]. Furthermore, microwave radiation in linen dyeing with natural lac optimises processing by reducing both time and energy consumption, offering a safer and more eco-friendly alternative to conventional heating methods [16]. Among these techniques, microwave irradiation, not only for surface modification but also for the dyeing process simultaneously, provides numerous benefits owing to its uniform heating, low cost, short processing time, and low energy consumption with mass transfer dynamics [17]. Recent research indicates that combining natural dye from the insect *Laccifer lacca* with microwave heating is effective for

linen dyeing, lowering dyeing time to approximately 7 min while ensuring sufficient colourfastness and conserving energy [16]. Stimulation of dyeing with microwave assistance for 4 min yields excellent colour strength [18]. Another study used microwave heating to dye reactive dye and get a fixing duration of 3 min yielded superior results [19]. Furthermore, the microwave method completes the fixation stage in just 3 min, unlike the conventional technique, which takes 45 min to complete the process. Additionally, the colour strength of the samples dyed using the microwave method was greater than that of those dyed via the conventional method [19]. The prospective microwave dyeing combined with chitosan cationisation should enhance the adherence of the BFS on cotton fabrics. The process will be optimised by a statistical approach, specifically the Design of Experiments (DoE) approach with Response Surface Methodology (RSM).

Response Surface Methodology is instrumental in constructing empirical models, optimising processes, and analysing interactions among numerous factors to enhance process outcomes [20]. With Box-Behnken Design (BBD), RSM enhances efficiency by enabling the concurrent optimisation of many process variables, thus minimising the number of experimental trials needed when compared with other RSM techniques, such as Central Composite Design (CCD) [21]. The absence of corner points in BBD avoids extreme conditions, especially in the dyeing process using the microwave. This technique has been successfully implemented in industrial-scale optimisation of various process parameters [22].

To the best of our knowledge, there have been no reports on using microwave and chitosan on cotton dyeing with banana (*Musa paradisiaca* var. Nipah) floral extract. In the present study, the BBD was employed to refine the cotton dyeing process, in which the variables of radiant power, radiation time, and chitosan concentration were optimised to attain optimal lightness levels and colour differences. Furthermore, a comparative assessment of treated and untreated cotton samples was performed to evaluate differences in colour strength and fastness.

MATERIALS AND METHODS

This section provides a comprehensive description of the methodology employed in this study. The process begins with preparing natural dyes, followed by applying chitosan to fabric samples. The dyeing process, along with its optimization, is then carried out. Afterwards, the samples are analysed using Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy (SEM) to characterize their properties. Finally, the colour evaluation of the samples is conducted, providing a thorough assessment of the effects of the treatments applied.

Materials

Plain 100% cotton fabric (96 g/m²) was supplied by PT. Primiissima, a Special Region of Yogyakarta, was purchased from the local online market. Banana (*Musa paradisiaca* var. Nipah) floral stems were selected as a natural dye source and were gathered from Sungai Kakap, Kuburaya, Indonesia, indigenous habitat. Chitosan (C₅₆H₁₀₃N₉O₃₉) with medium molecular weight (GR for analysis, CAS number 9012-76-4), acetic acid (CH₃COOH) (Merck for analysis EMSURE®, CAS number 64-19-7), and non-ionic detergent Triton X-100 (C₁₆H₂₆O₂) (GR for analysis, CAS number 9036-19-5).

Dye extraction

A crude bio-source extract was formulated by boiling 50 g of banana (*Musa paradisiaca* var. Nipah) floral stem powder with 1000 mL of distilled water. The extract was then heated at 60 °C for 60 min, followed by filtration, and the resulting extracted material was preserved for subsequent dyeing procedures.

Chitosan treatment

Before dyeing process, a clean cotton fabric (5 cm × 10 cm) was washed using non-ionic detergents was impregnated for 1 h in a solution containing 0.5–1% (w/v) of chitosan and 1% (v/v) of acetic acid in distilled water, then padded 100% wet pick up with a padder by a pad-dry-cure method, dried at 80 °C for 5 min, and cured at 120 °C for 3 min.

Dyeing process

The dyeing process was conducted in a laboratory-based domestic microwave oven (Electrolux, model EMM2308X, with an output power of 800 W operating at 2450 Hz). The dyeing process in variable factor settings using hard liquor with a fabric ratio of 40:1 and the microwave's radiant power and radiation time were adapted based on the experimental design. For comparison purposes, the conventional dyeing technique involved treating cotton by heating the dye solution to a temperature of 60 °C for 1 h, utilising a material-to-liquor ratio of 1:40.

Fourier Transform Infrared Analysis

The functional groups present in irradiated cotton, non-irradiated cotton, chitosan, and chitosan-treated cotton were identified using Attenuated Total Reflectance-Fourier Transform Infrared (ATR-FTIR) spectroscopy using Thermo Scientific Nicolet iS10 with a resolution of 4 cm⁻¹ and a spectral range of 4000–550 cm⁻¹.

Scanning Electron Microscopy

The surface morphology of irradiated and non-irradiated cotton was analysed using a scanning electron microscope (SEM) JEOL JSM-6510LA operating at 10 kV.

Experimental design

The experimental design and statistical response analysis were conducted using the statistical software Design Expert 13.0 (Stat-Ease, Minneapolis, USA) to obtain optimal radiant power, radiation time, and chitosan concentration variables that yield the lowest lightness and highest colour difference. The Box Behnken Design, an RSM tool, optimised those three process variables. The range of experimental factors represents the independent input parameters, their coded factors, and actual factors are tabulated in Table 1. The statistical software IBM SPSS Statistics (IBM Co., Ltd., America, United States) was utilised to evaluate the difference between the predicted values and the actual or experimental values by an independent-sample t-test for lightness and colour difference. Lightness and colour difference were designated as the corresponding output responses to the input parameters and expressed by second-order polynomial equations as shown in Eq. (1):

$$Y = \beta_0 + \sum_{i=1}^n \beta_i X_i + \sum_{i=1}^n \beta_{ii} X_i^2 + \sum_{1 \leq i < j}^n \beta_{ij} X_i X_j + \varepsilon \quad (1)$$

Here, β_0 , β_i , β_{ii} , and β_{ij} represent regression coefficients; X_i and X_j are coded independent variables; Y is the response of lightness and colour difference, and ε is the model error.

Table 1. Experimental factor range

Code Factors	Actual Factors	Unit	Low level	High Level
A	Radiant power	Watt	100	800
B	Radiation time	min	1	12

C Chitosan concentration % 0.5 1

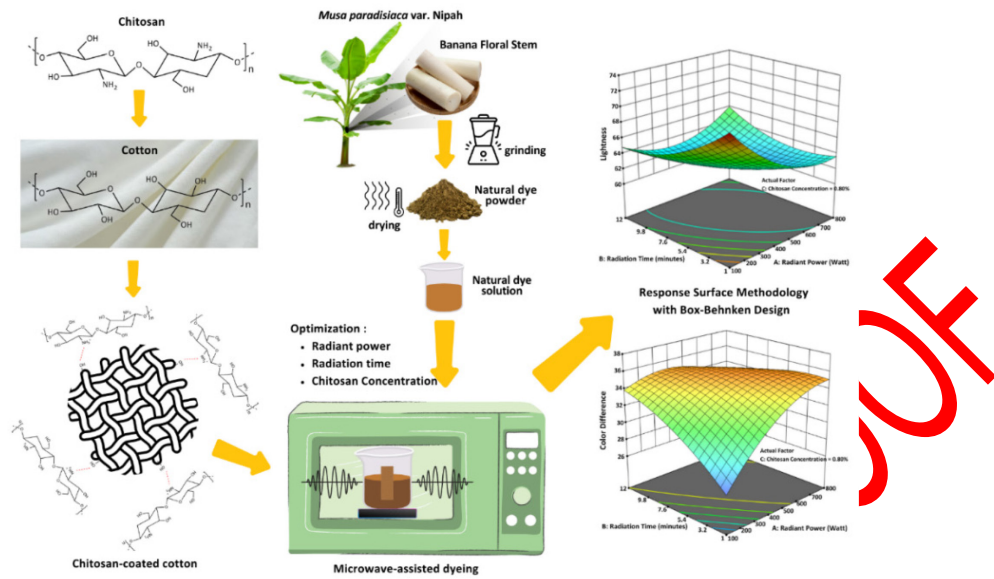


Figure 1. Schematic illustration of microwave-assisted dyeing of chitosan-coated cotton using banana (*Musa paradisiaca* var. Nipah) floral stem extract to optimise the dyeing process utilised RSM with BBD

Dyeing assessment

The colour analysis of the cotton fabric samples at each stage was conducted. The optimisation of three parameters of dyeing (radiant power, radiation time, and chitosan concentration) used lightness values from colour coordinate measurement, especially lightness (L^*) and colour difference (ΔE) as responses. The Colourimeter HFBTE AMT516 was used to measure CIE L^* , a^* , and b^* parameters. L^* represents the level of lightness, a^* ranges from redness to greenness, and b^* ranges from yellowness to blueness. The values of ΔE mean the colour difference between the sample and reference, which are the treated cotton and untreated cotton fabric. The colour difference (ΔE) was obtained using Eq. (2):

$$\Delta E = \sqrt{(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2} \quad (2)$$

The absorbance (Abs) and colour strength (K/S) of some selected dyed cottons were measured utilising a Shimadzu UV-2401-PC Spectrophotometer. The Kubelka-Munk in Eq. (3) was employed to compute the colour strength value. Further, the colour fastness of the dyed cotton is evaluated by International Standards, utilising the grey scale for colour change and staining, rated on a scale of 1 to 5. A grey scale value of 1 indicates very poor fastness, while a rating of 5 signifies excellent fastness [23]. The dyed cotton samples were tested under ISO 105-C10:2006, ISO 105-B01:2014, and ISO 105-B07:2009 to assess wash fastness, light fastness, and perspiration fastness levels with colour changes assessed. The schematic illustration of the methodology is expressed in Figure 1. The colour strength (K/S) was obtained using Eq. (3):

$$\frac{K}{S} = \frac{(1-R)^2}{2R} \quad (3)$$

where K/S is the ratio of diffusion coefficient and reflectance coefficient and R is the reflectance coefficient.

RESULTS AND DISCUSSION

This section begins by demonstrating the effects of chitosan and the dyeing process assisted by microwave radiation on the colour coordinate values ($L^*a\ b$), colour difference (ΔE), colour strength (K/S), and absorbance (Abs) of dyed samples. Subsequently, optimization is conducted to determine the optimal values of radiation power, radiation time, and concentration used in the dyeing process, along with its mathematical model, followed by experimental validation. The section concludes with the evaluation of the colour fastness of both treated and untreated samples towards wash, light, and perspiration.

Effect of chitosan and microwave-assisted dyeing treatment

The cotton fabrics were subjected to chitosan treatment through the pad-dry-cure method to impart a cationic charge, facilitating dyeing with banana (*Musa paradisiaca* var. Nipah) floral stem dye without requiring metallic mordants. The infrared spectrophotometer was employed to determine the functional groups of the cotton samples and confirm the modification of the cotton surface with chitosan. The spectra of cotton fabrics, chitosan, and chitosan-coated cotton fabrics are presented in Figure A1. After the dyeing process, the cotton fabrics were evaluated and compared between microwave dyeing and conventional dyeing. Figure 2 shows the absorbance spectra of dyed fabrics with and without chitosan dyed using microwave-assisted and conventional dyeing methods. Chitosan coating as a bio-mordant on cotton before dyeing enhances the cotton's ability to absorb dyes compared to untreated cotton. This is indicated by the high absorbance value in Figure 2. Greater dye absorption occurs in cotton fabrics with higher absorbance levels [24]. Conversely, dyeing treatment using microwave radiation influences the absorbance value. In microwave-assisted dyeing, the absorbance value is higher compared to conventional dyeing. The data in Table 2 further support this observation.

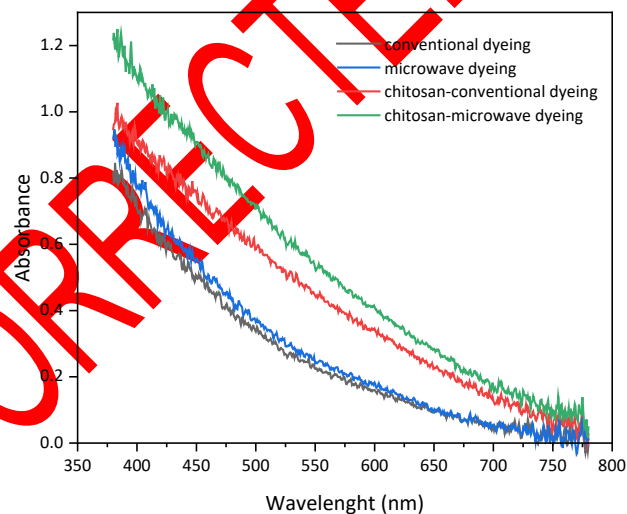


Figure 2. Absorbance spectra of the dyed cotton with conventional dyeing, microwave dyeing, conventional dyeing with chitosan-coated, and microwave dyeing with chitosan-coated

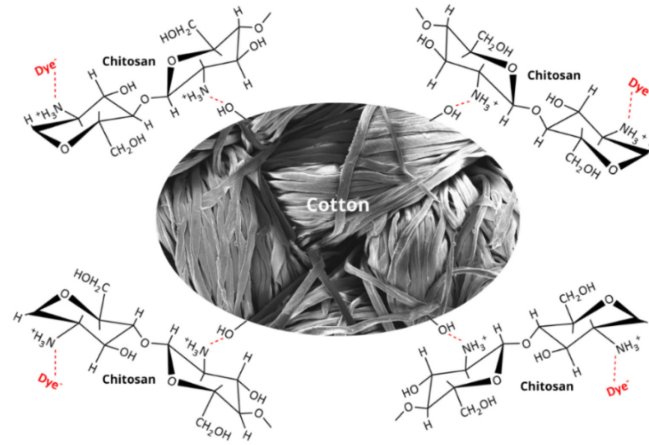


Figure 3. Possible mechanisms that generate electrostatic attraction between cotton, chitosan, and dye

Table 2. Effect of microwave irradiation and chitosan treatment on the colour coordinate ($L^*a^*b^*$), colour difference (ΔE), colour strength (K/S), and absorbance (Abs) of dyed samples






Sample	L^*	a^*	b^*	ΔE	K/S	Abs	Coloured images
Cotton fabrics	92.84	0.77	-2.28	—	—	—	
Cotton dyeing with conventional dyeing	71.29	4.52	10.06	25.11	0.36	0.25	
Cotton dyeing with microwave dyeing	70.35	5.33	14.55	28.46	0.44	0.28	
Chitosan-coated with conventional dyeing	61.46	8.65	15.14	36.74	0.86	0.42	
Chitosan-coated with microwave dyeing	59.12	8.77	14.77	38.62	1.43	0.52	

Table 2 shows that the dyeing of cotton fabrics treated with chitosan shows better colour difference, colour strength, and colour absorption ability than untreated cotton fabrics. This indicates that chitosan aids in absorbing and retaining the colours of banana flower stem extracts on cotton fabrics. Lower L^* values suggest that the sample darkens relative to the control sample. Table 2 revealed that the presence of chitosan in dyed fabrics reduces lightness (L^*) while simultaneously increasing colour difference (ΔE), colour strength (K/S), and absorbance (Abs) values. The colour strength (K/S) value of the dyed material exhibits a direct relationship with the dye concentration within the material [23]. Chitosan-coated cotton fabrics exhibit a higher K/S value than uncoated cotton. Amino groups of chitosan that are slightly protonated in acidic environments may enhance the immobilization of chitosan molecules onto cotton via electrostatic attraction [25]. Figure 3 illustrates the possible mechanism of dye adherence to cotton coated with chitosan. The diverse functional groups in the cotton, chitosan, and dye structures allowed an electrostatic force among the three [26].

Table 2 also demonstrates that chitosan-coated cotton dyeing assisted by microwave irradiation yields superior results, with the lowest lightness value and the highest colour difference and colour strength (K/S). Microwave-assisted heating is a more efficient method for chemical reactions, significantly reducing reaction times. This method directly heats the reaction mixture, providing uniform temperature throughout the system [27]. Unlike conventional heating, microwave irradiation heats materials volumetrically, which only affects the surface. Polarised molecules, such as water, reorient with the changing electromagnetic field, generating heat through friction and facilitating dye penetration into the fabric. The microwave heating rate and dye fixation reach equilibrium at a specific point, optimising dye adherence to the fabric and producing superior colour depth in a shorter time than conventional methods [16]. The enhancement in colour immersion and dye absorption on textile materials can be attributed to the efficient mass transfer kinetics promoted by microwave-assisted extraction, which accelerates the process while preserving the integrity of the cotton structure [28]. Additionally, microwave radiation plays a dual role by altering fibre surfaces, which further enhances dye penetration and improves colour fastness, ensuring better retention without damaging the cotton material [29]. As seen in the FTIR spectra presented in Figure A2, microwave irradiation did not exert a notable impact on the chemical structure of cotton fibres as also observed by [30]. The surface morphology of cotton fabrics also displays no differences, as examined using Scanning Electron Microscopy (SEM) in Figure A3.

Microwave-assisted dyeing provides significant environmental and economic advantages, including reductions in water, chemical, and energy consumption, while promoting eco-friendly dyes [31]. When compared to other radiation-based dyeing methods, such as ultrasonic and UV radiation, microwave-assisted dyeing offers superior efficiency and environmental benefits. This method reduces dyeing time, lowers chemical consumption, and enhances colour strength and durability [32]. Microwave-assisted dyeing is more time-efficient than ultrasonic dyeing, while UV radiation is typically restricted to post-dyeing treatments [33]. However, the industrial-scale implementation of microwave dyeing faces challenges related to high initial investment, the necessity for specialized equipment, and the need for uniform heating across large volumes of fabric [34]. Process optimization is crucial to maintaining operational efficiency and product quality.

Model fitting and statistical analysis

Significant processes of variables (A) radiant power, (B) radiation time, and (C) chitosan concentration were chosen for dyeing process optimisation of the operating conditions using the Box-Behnken approach for minimising lightness (L^*) and maximising colour difference (ΔE) of dyed samples (Table 3). Box Behnken Design generated a total of 27 experiments. All experiments lightness (L^*) and colour difference (ΔE) are presented in Table 4.

Table 3. Independent variables and levels according to Box-Behnken

Variables	Levels		
	-1	0	1
Radiant power	100	450	800
Radiation time	1.0	6.5	12.0
Chitosan concentration	0.50	0.75	1.00

Analysis of variance (ANOVA) was employed to assess the significance of the factors and their interactions with the response. P-values below 0.05 indicate the statistical significance of the

model and its terms. Insignificant interactions were eliminated through a model reduction in this study to enhance the model. Table 5 demonstrates that the model is adequate for exploring the design space. The significance of the interaction terms (AB, AC, BC) indicates whether a meaningful interaction effect exists between the two dependent variables. In this case, the ANOVA model results indicate that A, B, C, AB, A², B², and C² are significant model terms as the "Prob > F" values are less than 0.05. This table also confirmed that only the interaction between A and B is significant. This implies that the relationship between term A and the responses L* or ΔE is influenced by term B, or remains constant across different levels of the other term B. Similarly, the significance of the quadratic terms (A², B², C²) suggests the presence or absence of a significant quadratic relationship between the variables (A, B, C) and the responses variables. This indicates that the relationship between the dependent variables and the response variables is non-linear, exhibiting a curved pattern [35]. The sum of the square model provided the most significant model (p < 0.0001) with an insignificant lack of fit for both L* and ΔE, which were 0.4562 and 0.3386, respectively. The lack of significance in lack-of-fit testing is insignificant due to pure error, and if this term is insignificant, then the model is accurate. The quadratic model in terms of coded factors and actual factors is provided in Eq. (4) and (5), respectively (after removing insignificant terms).

Table 4. Experimental design of the process and responses

Sample	A: Radiant power [W]	B: Radiation time [min]	C: Chitosan concentration [%]	Response 1: Lightness	Response 2: Colour difference
1	450	6.5	0.75	61.04	36.52
2	450	6.5	0.75	61.71	35.26
3	100	6.5	0.5	69.28	28.17
4	450	1.0	0.5	69.44	28.22
5	800	12.0	0.75	66.28	32.01
6	100	6.5	1.0	68.64	29.41
7	450	6.5	0.75	60.31	36.57
8	800	1.0	0.75	63.17	35.85
9	800	6.5	0.5	68.63	28.30
10	450	6.5	0.75	62.17	34.68
11	450	6.5	0.75	62.32	34.60
12	450	6.5	0.75	63.86	33.20
13	450	6.5	0.75	63.39	33.83
14	450	12.0	0.5	65.34	32.23
15	100	12.0	0.75	65.56	32.87
16	450	6.5	0.75	63.92	33.41
17	100	1.0	0.75	71.22	26.06
18	450	1.0	1.0	66.68	30.87
19	450	6.5	0.75	62.71	35.84
20	450	6.5	0.75	61.24	35.96
21	450	12.0	1.0	65.30	32.23
22	450	6.5	0.75	63.85	33.73
23	450	6.5	0.75	60.87	36.73
24	800	6.5	1.0	63.70	33.68
25	450	6.5	0.75	61.36	35.87
26	450	6.5	0.75	62.74	35.05
27	450	6.5	0.75	64.79	32.52

$$\text{Lightness (L}^*) = 62.42 - 1.62A - 1.00B - 1.05C + 2.19AB + 2.51A^2 + 1.63B^2 + 2.64C^2 \quad (4)$$

$$\begin{aligned} & \text{Colour difference } (\Delta E) & (5) \\ & = 34.92 + 1.67A + 1.04B + 1.16C - 2.67AB - 2.11A^2 - 1.11B^2 - 2.92C^2 \end{aligned}$$

where A is the radiant power of the microwave, B is radiation time, C is the chitosan concentration, and AB is the interaction between the radiant power and the radiation time of the microwave.

The regression coefficients (R^2) for the two responses (lightness and colour difference) are 0.8524 and 0.8374, respectively, indicating that only 0.1476% (lightness) and 0.1626% (colour difference) are not explained by the model due to errors. The predicted R^2 and adjusted R^2 values for lightness and colour difference are 0.7981 and 0.6823 and 0.7776 and 0.5830, respectively (Table 6). The predicted R^2 agrees with the adjusted R^2 for lightness and colour difference since the difference is less than 0.2. These high R^2 values are quite consistent with each other and enhance the significance of the model. After plotting normal percentage probability plots, data points that deviate from the model are scattered along the straight line, and very few points are outside the line (Figure 4). A strong correlation between the dependent variables and responses was observed from a simplified quadratic model, which was validated through a normal probability plot of the residuals. The assumption of normality was confirmed, as the residual plots closely followed a linear correlation for both lightness and colour difference, indicating that the simplified quadratic model is reliable for the selection process [36]. The same observation is seen in the actual versus predicted lightness (Figure 5a) and colour difference values (Figure 5b). Therefore, this relationship between experimental responses and model outputs is significant.

Table 5. ANOVA results of the established model

Source	Mean square		F-value		p-value		
	L*	ΔE	L*	ΔE	L*	ΔE	
Model	27.91	26.91	15.68	13.98	< 0.0001	< 0.0001	Significance
A-Radiant power	20.87	22.26	11.72	11.57	0.0028	0.0030	
B-Radiation Time	8.06	8.71	4.53	4.53	0.0467	0.0467	
C-Chitosan Concentration	8.76	10.74	4.92	5.58	0.0390	0.0290	
AB	19.23	28.42	10.80	14.77	0.0039	0.0011	
A ²	30.75	21.75	17.27	11.30	0.0005	0.0033	
B ²	13.06	6.03	7.34	3.14	0.0139	0.0927	
C ²	34.09	41.81	19.15	21.72	0.0003	0.0002	
Lack of Fit	1.77	2.26	0.99	1.25	0.4562	0.3386	Insignificance

Table 6. ANOVA results of fit statistics

Measuring item	Lightness	ΔE
Std. Dev.	1.33	1.39
Mean	64.43	33.10
C.V. %	2.07	4.19
R ²	0.8524	0.8374
Adjusted R ²	0.7981	0.7776
Predicted R ²	0.6823	0.5830

Effects of dyeing parameters on lightness and colour difference

Figure 6 illustrates how both radiant power (A) and radiation time (B) influence the lightness and colour difference of dyed samples. Eq. (4) and (5) were employed to simplify the plotting of response surfaces, with two variables represented simultaneously while the third variable remained constant at a specified value within each graph. The figure demonstrates that higher

radiant power and longer radiation times directly correlate with increased colour differences and decreased lightness. An increase in radiant power and radiation time decreases the lightness value and increases the colour difference value until reaching the optimum power of 554.12 W and radiation time of 7.10 min. This relationship is attributed to the rise in system temperature as radiation time extends. The elevated temperature enhances the diffusion of dye substances, leading to more significant fibre swelling in cotton [37]. In low temperatures, the quantity of dye absorbed is small, leading to a diminished value of colour difference. At elevated temperatures, more dye molecules rapidly migrate to the surface from the dye bath and permeate the fabric through the open pores of the cellulose material, resulting in a higher colour difference value. The solubility of the dye, the rate of dye absorption, and the absorption of the dye are all improved at elevated temperatures, leading to accelerated diffusion of dye molecules into the fabric through its open pores, yielding a higher colour difference value [23].

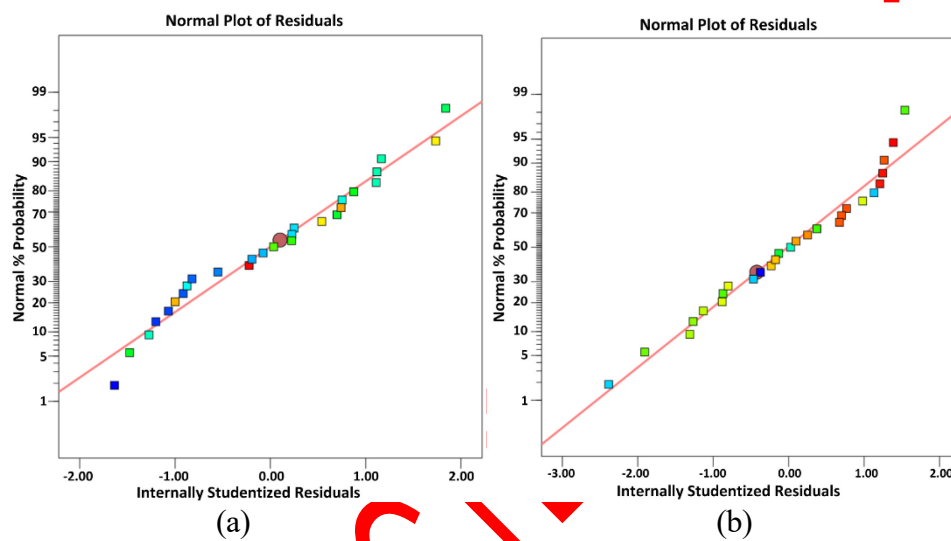


Figure 4. The studentised residuals vs. normal percentage probability plot for (a) Lightness responses and (b) ΔE responses

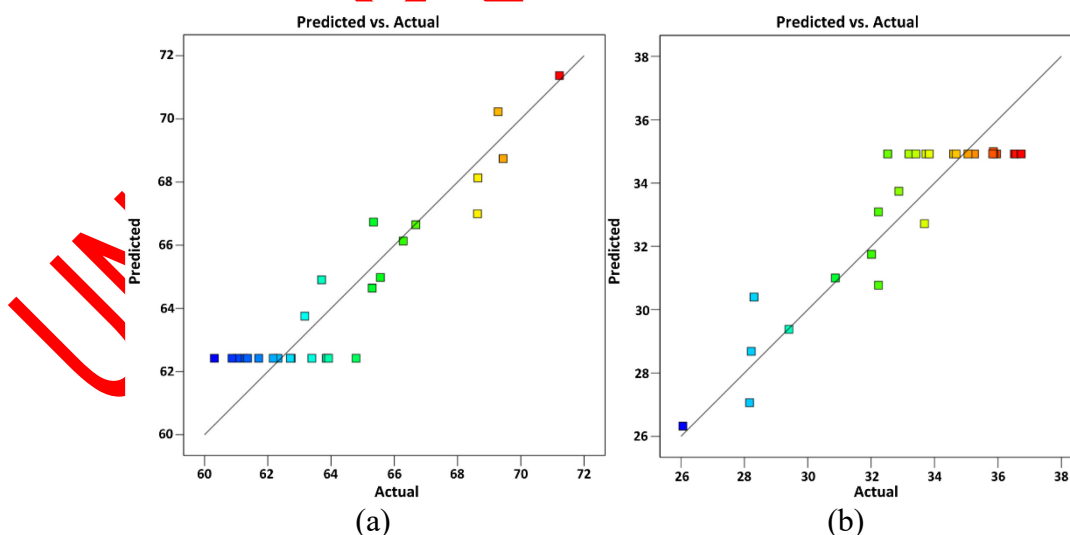


Figure 5. The actual versus predicted plot for (a) Lightness responses and (b) ΔE responses

On the other hand, further increases in power (>554.12 W) and radiation time (>7.10 min) have the opposite effect. Increased microwave power and radiation time cause more significant damage to the natural dye matrix, and intense absorption of microwave energy leads to a swift

temperature rise within the dyeing vessel [38]. Increasing radiant power and radiation time accelerates temperature rise, leading to dye molecule degradation. The decline in colour difference values beyond a specific dyeing temperature (more than 80 °C) may be attributed to the desorption of dye molecules upon attaining equilibrium at this temperature, leading to diminished dye absorption and, consequently, reduced colour difference values [23]. The chromophore compound from banana floral stem extract is significantly retained within the cellulose matrix at 80 °C, which expands the cotton pores, facilitated by the attractive interactions between the cellulose of cotton and the chromophore anchoring sites. Nonetheless, elevating the temperature beyond 80 results in the hydrolytic destruction of the chromophore compound of banana floral stem extract [7]. Insufficient heating levels fail to expedite the movement of dye molecules toward the changed fabric surface. In contrast, excessive heating induces the desorption of dye molecules or leads to dye degradation, culminating in diminished colour strength [16]. Moreover, the higher kinetic energy of the dye substances and the breakdown of dye aggregates occur at higher temperatures [39]. In the microwave mechanism, internal electric fields are produced through the interaction of energy with substances at the molecular level. This ability converts electromagnetic energy into thermal energy within the irradiated material [40].

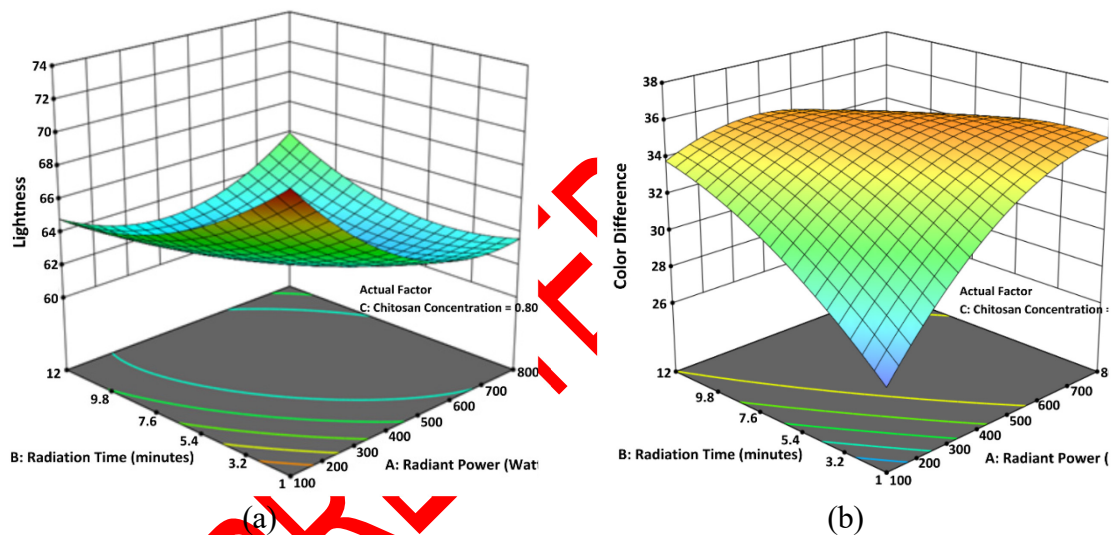


Figure 6. Response surface plots showing the effect of each significant parameter on (a) Lightness and (b) ΔE responses of dyed samples

Optimisation and validation of the model

The optimal conditions for dyeing cotton fabrics with banana floral stem extract were predicted using Design Expert on the optimisation function. The study aims to discern the variance between observed outcomes and projected results based on one or more criteria of the dyeing process. This investigation aimed to achieve the lowest lightness and the highest colour difference, considering microwave dyeing radiant power, radiation time, and chitosan concentration within a specified range. The findings are outlined in Table 7. The optimisation results indicated that the optimal conditions for dyeing were achieved at a radiant power of 554.12 W, radiation time of 7.10 min, and chitosan concentration of 0.80% with 0.856 desirability. According to Design-Expert 13.0, the ideal expected values for minimum lightness are approximately 62.04, and the maximum colour difference is approximately 35.36.

Table 7. Optimised conditions for obtaining the lowest lightness (L^*) and the highest colour difference (ΔE)

Name	Goal	Lower Limit	Upper Limit	Optimum
A: Radiant power	in range	100	800	554.12

B: Radiation time	in range	1	12	7.10
C: Chitosan concentration	in range	0.5	1	0.80
Lightness (L*)	minimise	60.31	71.22	62.04
Colour difference (ΔE)	maximise	26.06	36.73	35.36

Cotton fabric samples were arranged under the optimised conditions to validate the experimental model. Table 8 shows that the predicted lightness and colour difference are closely aligned with the experimental or exact values, further validating the experimental model's accuracy. The predicted values exhibit significant agreement with the exact (experimental) values and are deemed statistically insignificant, with $p > 0.05$. The values for prediction data exhibit negligible variance from the experimental data. This implies that there are no significant deviations from the objective distribution, implying that the applied model is appropriate and sufficiently adaptable for accurate optimisation [41].

Table 8. Predicted and exact values of the dependent variable under optimum conditions of microwave-assisted dyeing. ns denotes an insignificant difference between the predicted and the exact value ($p > 0.05$)

Dependent variable	Microwave-assisted dyeing		Sig.
	Predicted	Exact	
Lightness (L*)	62.04	61.54	0.575 ^{ns}
Colour difference (ΔE)	35.36	36.46	0.474 ^{ns}

Colourfastness properties

This study assessed the quality of dyeing on the cotton fabric via a colourfastness test for wash, light, and perspiration. The evaluation compares the colour change with a colour change standard. Proven standards include those published by the International Standards Organisation (ISO), including a grey scale for evaluating colour change in cotton samples. Table 9 presents the colourfastness values of cotton fabrics dyed using conventional and microwave methods, both with and without chitosan treatment, using an aqueous extract from a banana (*Musa paradisiaca* var. Nipah) floral stem. The coloured image of colourfastness properties is shown in Table A1, Table A2, and Table A3. Cotton fabrics treated with chitosan exhibit good wash, light, and perspiration fastness properties, scoring between 3–4, 4–5, and 4–5 on the grey scale, respectively, with 5 being the highest rating [42]. In contrast, cotton samples without chitosan treatment exhibit poor wash and light fastness. The highest colourfastness is achieved when the cotton fabric is coated with chitosan, possibly owing to the ionic interaction between chitosan's amine groups and the natural dye molecules' hydroxyl groups [43]. Additionally, there are slight differences in wash fastness between conventional dyeing methods and microwave-assisted dyeing on chitosan-coated cotton, with microwave assistance resulting in slightly better results. This improvement can be ascribed to the ability of microwave radiation to modify the fabric surface, enhancing the sorption of colourants without altering their chemical nature [44]. Thus, suggest the application of a microwave-assisted process for cotton dyeing. The results highlight the effectiveness of microwave irradiation and chitosan in improving colour difference, colour strength, and the colourfastness of cotton fabrics dyed with banana floral stem extract.

Table 9. The colourfastness properties of dyed cotton

Samples	Wash fastness (ISO 105-C10:2006)	Light fastness (ISO 105-B01:2014)	Perspiration (ISO 105-B07:2009)
Conventional dyeing	1–2	4–5	2
Microwave dyeing	1–2	4–5	2

Conventional dyeing with chitosan-coated	3–4	4–5	4–5
Microwave dyeing with chitosan-coated	4	4–5	4–5

Note. 1–2 (very poor to poor), 2 (poor), 3–4 (fair to good), 4 (good), 4–5 (good to excellent)

Based on the literature, the aqueous extract of BFS (*Musa sapientum*) contains several compounds, including condensed tannin, flavone, anthraquinone, and anthocyanin, contributing to its dyeing properties. Among these, flavone and anthraquinone showed stronger exhaustion and fixation on cotton than tannin and anthocyanin. Tannin, with its higher molecular weight, exhibited lower fixation after dyeing and washing. The better fixation of flavones and anthraquinone is likely due to their ability to form stronger interactions with cotton fabrics through hydrogen bonding and van der Waals forces [7]. Adding chitosan further enhances colourfastness by introducing functional groups (–OH, –NH₂), which improve the binding of these compounds to the cotton matrix [45].

Microwave-assisted dyeing technology offers significant advantages in terms of dye uptake, colour strength, and fastness, making it a versatile solution for various fibre types [46]. While the technology provides notable energy savings and operational cost reductions through faster and more uniform heating, the high initial capital investment remains a substantial barrier to large-scale implementation [46]. Despite this, long-term cost savings in water, chemicals, energy, and labour can offset these upfront expenses [47]. However, challenges persist, including the need for specialized technical expertise and difficulties in scaling the process for industrial use, particularly for small-scale manufacturers [48]. Furthermore, while the technology reduces the need for additional chemicals and water, operational costs, such as electricity consumption and maintenance, may partially diminish the anticipated energy savings. Therefore, it is crucial to carefully evaluate the economic feasibility of adopting microwave-assisted dyeing on a large scale, considering both initial and ongoing operational costs [49].

CONCLUSION

The growing demand for eco-friendly products has led to their widespread adoption. This study optimises microwave-assisted dyeing of cotton fabric using chitosan and banana floral stem extract. The method was validated and optimised using Response Surface Methodology (RSM) with Box-Behnken Design (BBD). Results show banana floral stems are an excellent natural dye source, enhancing cost-effectiveness, energy efficiency, and time savings. Optimal conditions were 554.12 W radiant power, 7.10 min radiation time, and 0.80% chitosan concentration. Applying these tools to other botanical sources is recommended. Compared to conventional methods, microwave dyeing significantly reduces dyeing time. Chitosan as a bio-mordant improves colour strength, and colour fastness, thus reducing environmental impact. A combined chitosan-treated cotton and microwave dyeing technique shall pave the way for green dyeing of natural dyes on cotton fabrics.

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STATEMENT AND DECLARATION

During the preparation of this manuscript, the first author utilised ChatGPT to conduct grammar and style checks, enhancing the clarity and readability of the text. After using this tool/service, the authors reviewed and edited the content as needed and took full responsibility for the content.

NOMENCLATURE

Symbols

L^*	lightness	
ΔE	colour difference	
W	Watts	
K/S	colour strength	
Y	response of lightness and colour difference	
X_i and X_j	coded independent variables	
A	radiant power	Watts
B	radiation time	min
C	chitosan concentration	%
a^*	ranges from redness to greenness	
b^*	ranges from yellowness to blueness	
Abs	absorbance	
AB	the interaction between the radiant power and the radiation time of the microwave	
R^2	regression coefficients	
p-value	probability value	

Greek letters

$\beta_0, \beta_i, \beta_{ii},$ and β_{ij}	regression coefficients
E	the model error

Subscripts and superscripts

ns	not significance
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Abbreviations

BSF	Banana Floral Stem
DoE	Design of Experiments
RSM	Response Surface Methodology
BBD	Box-Behnken Design
CCD	Central Composite Design
ATR-FTIR	Attenuated Total Reflectance-Fourier Transform Infrared
SEM	Scanning Electron Microscopy
ANOVA	Analysis of variance
Sig.	significant
min	minutes
Std. Dev.	Standard Deviation
C.V.	Coefficient of Variation

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APPENDIX

This appendix provides additional data on the effect of chitosan and microwave-assisted dyeing treatment, as well as the optimization of the dyeing process using Box-Behnken Design (BBD). It includes infrared spectra of untreated, chitosan-coated, and dyed cotton, alongside surface morphology images obtained through scanning electron microscopy (SEM). The appendix also presents the optimal dyeing conditions derived from BBD, showing the best parameters for microwave-assisted dyeing. Additionally, the colourfastness properties, including wash, light, and perspiration fastness, of the dyed cotton samples are summarized with detailed grading results.

Effect of chitosan and microwave-assisted dyeing treatment

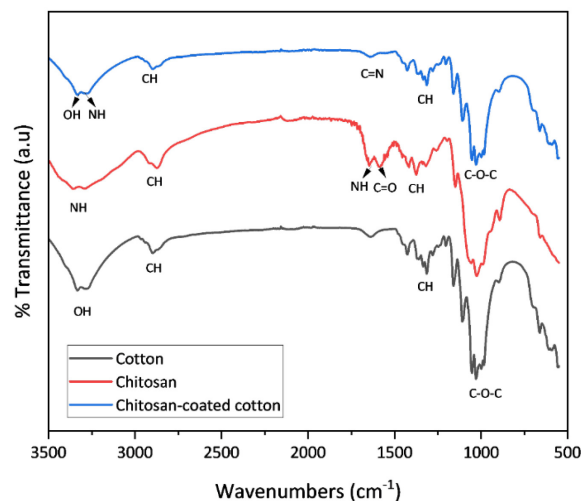


Figure A1. Infrared spectra of cotton (a) chitosan (b) and chitosan coated cotton (c)

The distinctive peaks of chitosan were challenging to differentiate from those of cotton in the Infrared spectra of chitosan-coated cotton due to similarities in their chemical structures. The similarity between the chitosan backbone and cotton has been suggested as the basis due to their mutual affinity. Chitosan has a free amine group at the C₂ position of its glucosamine residue that is protonated in acidic conditions, resulting in a positively charged chitosan molecule [50]. In Figure A1 (a), the broad spectrum observed within the interval 3500–3000 cm⁻¹ indicates the –OH stretching vibration of cotton, while the asymmetric C–H stretching is evident around 2900 cm⁻¹. There is a spectrum corresponding to the CH range of wagging at wavenumber 1314 cm⁻¹. The complex absorption observed around 1029 cm⁻¹ is linked with the cotton's C–O–C stretching groups [9]. This indicates that cotton contains a cellulose functional group. In Figure A1 (b), the absorption band between 3500–3100 cm⁻¹ represents NH₂ groups of chitosan, with amino groups (N–H) appearing between 1650–1580 cm⁻¹, which is a characteristic absorbance band of chitosan, depending on the groups it connects [51]. The C=O stretching vibration exhibits an absorption band around 1646 cm⁻¹. Furthermore, the spectrum depicted in Figure A1 (c) reveals a broad spectrum at 3500–3100 cm⁻¹ assignable to NH₂ and –OH groups, with a peak at 1641 cm⁻¹ corresponding to forming a C=N group between the aldehydic group and chitosan. There are peaks at wave numbers 1161 cm⁻¹ and two peaks at wave numbers 1054 cm⁻¹ and 1031 cm⁻¹, which are the peaks of the C–O–C stretching group and C–O stretching group respectively. This suggests successful binding between chitosan and cotton, affirming the effective coating of the cotton [42].

Infrared spectra were obtained to observe the chemical properties. Figure A2 shows the infrared spectra of dyed cotton using conventional and microwave-assisted methods. Microwave irradiation did not exert a notable impact on the chemical structure of cotton fibres [30]. Even after radiation exposure, the intensity of the functional peaks of –OH groups and bonds remained unchanged, suggesting that microwave irradiation remains unchanged in the chemical composition of the fabric. This underscores another noteworthy benefit of microwave radiation, as it preserves the fabric's chemical properties while offering energy, time, and cost savings [20]. The distinctive peak associated with hydrogen bonding within the cellulose structure of cotton fabric remains unchanged following microwave treatment lasting up to four min. Infrared spectral analysis conducted on treated and non-treated cotton fabric reveals that the peaks corresponding to –OH at 3300 cm⁻¹, CH at 1200 cm⁻¹, and C=O at 1075 cm⁻¹ exhibit no alteration. The persistence of the characteristic hydroxyl linkage peak in the cellulosic fabric suggests that the chemical composition remains unaffected by these rays, offering significant advantages within the textile processing sector [52].

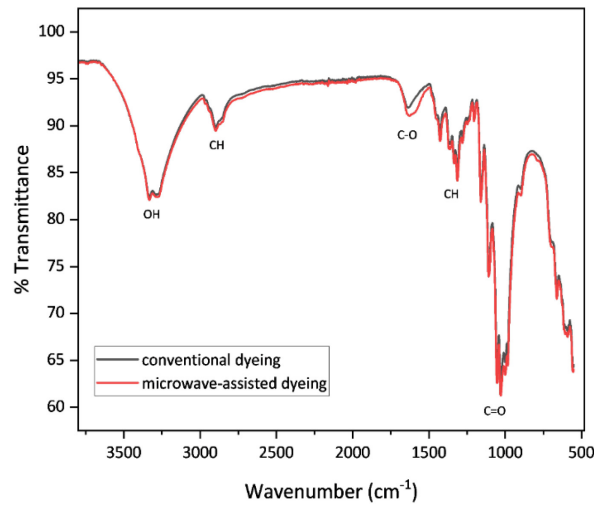


Figure A2. Infrared spectra of dyed cotton with different dyeing methods (a) conventional dyeing (b) microwave-assisted dyeing

The surface morphology of cotton fabrics was examined using Scanning Electron Microscopy (SEM). The sample was coated with a thin layer of gold (Au) to ensure proper conductivity and reduce charging effects during SEM imaging. This allowed for high-quality images with minimal distortion. Figure A3 presents images of cotton fabrics in three conditions, including untreated, treated with chitosan, and treated with both chitosan and natural dye.

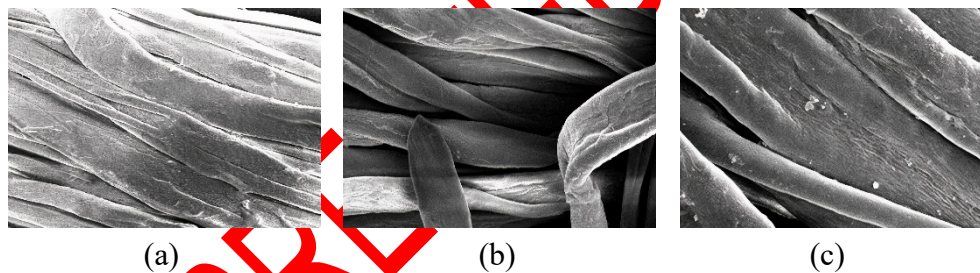
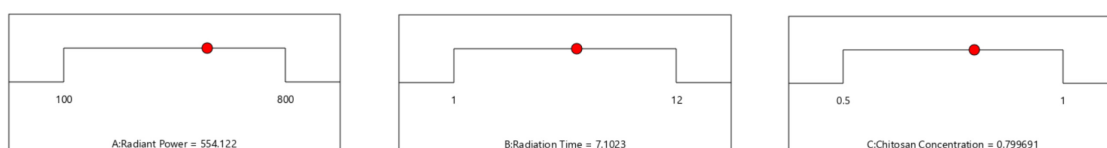


Figure A3. Scanning Electron Microscope Images of (a) undyed cotton (b) chitosan-coated cotton (c) chitosan coated-dyed cotton

Profiles of optimisation dyeing process use Box-Behnken Design

Figure A4 is a ramp function graph illustrating the main dependent variables (red dot) and the independent variables or their responses (blue dot). The height's spot relative to the point is the criterion anticipated to be attained during the optimisation procedure. The peak response value is predicted to emerge through a positive ramp gradient (lightness and colour difference), whereas a flat ramp exhibits a uniform desired value (radiant power, radiation time, and chitosan concentration). A desirability achievement of 85.6% was attained under the specified criteria when the cotton dyeing process employed a microwave with a radiant power of 554.12 W, a radiation duration of 7.10 min, and a chitosan concentration of 0.80%, yielding the lowest lightness (62.04) and the highest colour difference (35.36).



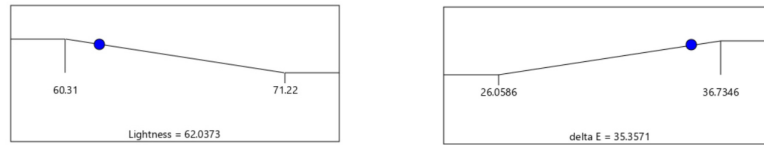

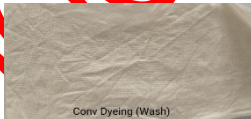
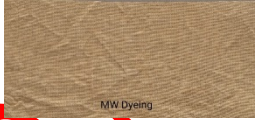
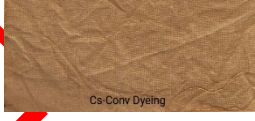



Figure A4. Desirability ramps for optimisation variable—the optimal values of the design variables (red points—optimal dependent variables values; blue points—optimal responses the predicted values)




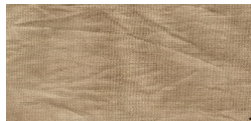
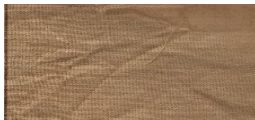
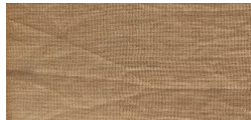
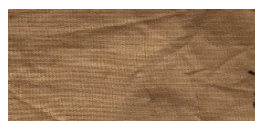
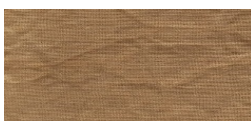
Colourfastness properties of dyed cotton

Table A1. The colourfastness properties to wash

Samples	Greyscale	Before	After
Conventional dyeing	1–2		
Microwave dyeing	1–2		
Conventional dyeing with chitosan-coated	3–4		
Microwave dyeing with chitosan-coated	4		

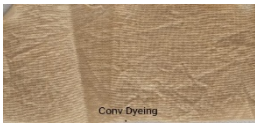
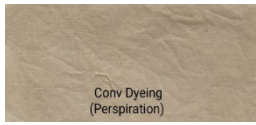
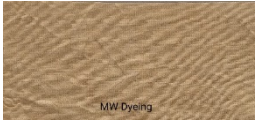
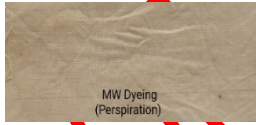

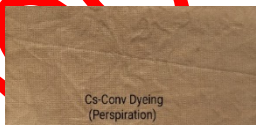

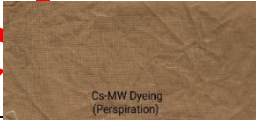
Note. 1–2 (very poor to poor), 3–4 (fair to good), 4 (good)

Table A2. The colourfastness properties to light

Samples	Greyscale	Before	After
Conventional dyeing	4–5		
Microwave dyeing	4–5		
Conventional dyeing with chitosan-coated	4–5		
Microwave dyeing with chitosan-coated	4–5		

Note. 4–5 (good to excellent)

Table A3. The colourfastness properties of perspiration

Samples	Greyscale	Before	After
Conventional dyeing	2		
Microwave dyeing	2		
Conventional dyeing with chitosan-coated	4–5		
Microwave dyeing with chitosan-coated	4–5		

Note. 2 (poor), 4–5 (good to excellent)

UNCORRECTED