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Optimized Dyeing of Cotton with semi-synthetic Embelin Ninhydrin Dye Obtained by Chemical Modification of Embelin from *Embelia Schimperi*

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ABSTRACT

Chemical modification is a promising path to address the limitations of a natural dye, such as limited shades and inadequate fastness properties. This study investigated the modification of embelin (2, 5-dihydroxy-3-undecyl-1, 4-benzoquinone) a plant-based benzoquinone compound from *Embelia schimperi* (Myrisinaceae family) with ninhydrin to get a semi-synthetic dye. The modified dye was applied to cotton fabric along with mordants to provide different shades. Optimum dyeing conditions were determined using Central Composite Design which showed optimum conditions of pH at 9, time of 60 min and temp of 80°C. The color fastness ratings were in the range of 4–5 evaluated on the Gray scale.

摘要

化学改性是解决天然染料的局限性 (如色度有限和牢度不足) 的一条很有前景的途径. 本研究探讨了用茚三酮对来自杨梅科的植物基苯醌化合物 embelin (2, 5-二羟基-3-十一烷基-1, 4-苯醌) 进行改性以获得半合成染料. 改性染料与媒染剂一起应用于棉织物, 以提供不同的色度. 采用中心复合设计法确定了最佳染色条件, 结果表明, 最佳染色条件为pH值为9, 时间为60分钟, 温度为80°C. 根据灰度评估, 色牢度等级在4-5范围内.

KEYWORDS
Chemical modification; embelin ninhydrin; optimization; colourfastness; response surface; color strength

关键词
化学改性; 恩贝林茚三酮; 优化; 色牢度; 响应面; 颜色强度

Introduction

In the modern age, synthetic dyes have taken a center stage in textile dyeing, however, they have shown carcinogenic and toxic effects. (Haji 2019) These harmful effects have caused society to shift to green dyeing of textile fibers using natural dyes, which are eco-safe and biodegradable (Adeel et al. 2022). Natural colorants contain different chromophores or auxochromes responsible for color. These colorants belong to different classes of natural products that include carotenoids, anthocyanins, chlorophylls, betalains and quinonoid and are produced by plants or animals (Haji and Rahimi 2020). Quinones found in nature have been of interest to humanity mainly because of the variable color properties they possess. Quinones such as carthamine and anthraquinone including alizarin and purpurin have bright colors, they have been isolated from *Rubia tinctorum* and are known for dyeing (Dulo et al. 2021), however, their solubility in water is low (Adeel et al. 2018) and on application to textile fibers, they have low affinity and poor fastness properties (Naveed et al. 2020).

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The application of natural dyes to textile fibers has been successful for proteinous fibers, however, application to cellulosic fibers needs further studies (Haji and Naebe 2020). Several techniques and methods have been employed to enhance its dyeability that include plasma treatment techniques (Haji 2020) functionalizing with cationic dyes (Haji, Mansour Bidoki, and Gholami 2020), mordanting whose role is to give color to textile fabric (Habib et al. 2021), microwave irradiation (Buyukakinci, Karadag, and Torgan Guzel 2021) and chemical modification (Ioannis, Blackburn, and Rayner 2011). Few studies have been carried out on chemical modification to enhance applicability to textile fibers, however, plenty of plant-based polyphenolic compounds are available in the literature that can serve as substituents for synthetic phenols (Pawar, More, and Adivarekar 2018).

In this study, embelin (a reported hair dye) isolated from *Embelia schimperi* and *Embelia ribes* (Myrisinaceae family), an alkyl substituted hydroxyl benzoquinone (Singh et al. 2019) is chemically modified with ninhydrin to overcome its limitations such as poor solubility and poor retention of its color. Semi-synthetic embelin ninhydrin has been considered a dye for the first time to dye the cotton fiber. The effect of three different parameters on the color strength of the dyed cotton fabric was investigated using response surface methodology and Central Composite Design. The optimum values for the highest color strength were obtained using the established model

Materials and methods

Copper Sulfate ($CuSO_4 \cdot 5H_2O$), Alum ($KAl\ (SO_4)_2 \cdot 12H_2O$), Ferrous Sulfate, ($FeSO_4 \cdot 7H_2O$) and Nickel Chloride ($NiCl_6$) were the used mordants. Ninhydrin and acetic acid were sourced from Sigma Aldrich, woven unbleached commercial cotton fabric was used.

Dye preparation

Embelia schimperi berries samples were collected from the . . . .County with coordinates (0°20′13.3"S 35°22′43.4"E). They were dried at room temperature for one week and ground into powder form using a grinder (Nutribullet, NB-101B model). Acetone was used for extraction and embelin was isolated using ethyl acetate and hexane in a 6:4 ratio. Semi-synthetic dye was synthesized as per the modified method of Mahendran (Mahendran et al. 2011). The resultant product was purified using classical column chromatography, yielding an embelin-ninhydrin (ENn) dye which was red-brown in color (Yield: 42%).

Mordanting of cotton fibers

Mordanting of cotton was done with, alum, copper sulfate, ferrous sulfate, and nickel sulfate. The mordanting process was carried out at optimal conditions at 80°C for 60 min and pH 9 with the 4% of weight fabric (o.w.f). The mordant solutions were used in a 1:50 material-to-liquor ratio. The pre-mordanting, cotton fabric was immersed in a mordant solution before dyeing with embelin ninhydrin dye. In simultaneous mordanting, the mordant solution was adopted together with embelin ninhydrin dye and in post-mordanting, the already mordanted cotton fabric was dyed (Chakraborty, Pandit, and Roy Maulik 2020)

Dyeing process and color measurements

The cotton fabrics were dyed using the laboratory type-dyeing machine (PARAMOUNT Digi wash i2). The cotton fabric was wetted with a nonionic surfactant, 30 minutes before dyeing (Wang et al. 2020). Dyeing experiments were carried out according to the procedure designed in Table 1. The dyeing pH was adjusted using 0.1 M hydrochloric acid and 0.1 M sodium hydroxide to the levels in the experimental design. The dye bath was at a 1:50 material-to-liquor ratio, upon completion of the dyeing process, the fabrics were washed with adequate water to remove the unfixed dyes and allowed

Table 1. Coded variables and the CCD surface design with responses.

Run	X_1	X_2	X_3	K/S
1	−1	−1	−1	1.56
2	1	−1	−1	3.11
3	−1	1	−1	2.31
4	1	1	−1	4.71
5	−1	−1	1	4.6
6	1	−1	1	4.01
7	−1	1	1	2.98
8	1	1	1	4.71
9	−1.672	0	0	1.98
10	1.672	0	0	4.06
11	0	−1.672	0	1.48
12	0	1.672	0	4.72
13	0	0	−1.672	3.61
14	0	0	1.672	4.23
15	0	0	0	4.63
16	0	0	0	4.67
17	0	0	0	4.61
18	0	0	0	4.6
19	0	0	0	4.64

to dry before measurements of color strength and fastness properties. The evaluation was based on the Gray scale for monitoring change (ISO 105-C02:1989, ISO 105 A02:1993, ISO 105-X12:2000). The dyed cotton reflectance values to its CIELAB color space (L*, a* and b*) values were evaluated using SpectroFlash X-rite SP62 spectrophotometer under illuminant, D65 (Manyim et al. 2021). The blank was undyed cotton fabric, calculations were done by applying Kubelka–Munk equation, at a given wavelength.

Optimization of dyeing process using response surface methodology (RSM)

Initial preliminary dyeing conditions were determined which informed the feasible experimental range. Response Surface Methodology (RSM), Central Composite Design experiments were designed using R. To determine the effect of three variables (temperature, time, and pH) on the color strength of the dyed fabric. The design yielded,19 experiments, and the color strength K/S was the respondent (dependent variable).

Results and discussion

Statistical modeling and analysis

Table 1 shows the experimental matrix as well as the response (K/S) after dyeing with the semi-synthetic dye. Table 3 shows the ANOVA results for the fitting of various models to the response data (K/S). R-squared is considered a measure of the quality of fit (Thi Huong, Thi Hong Khanh, and Pham Duy Linh 2021) according to the p-values (< 0.05) (Haji and Rahimi 2020) the values greater than 0.05 were considered to be of insignificant. The quadratic model was chosen as the best fit. The F-Test value was found to be 6.93 which concludes that the model was statistically significant and p values less than 0.05 were observed in (X1, X2, X3, $X1^2$, $X2^2$). The model Table 2, recommends R-squared (87.39%) which indicates that there is good predictability between the data obtained and the model established together with the response (K/S). The predicted R^2 is an indication of the accuracy.

Table 2. Model summary.

S	R-sq	R-sq(adj)
0.579408	87.39%	74.77%

Table 3. Analysis of variance.

Source	DF	AdjSS	Adj MS	F-Value	P-Value
Model	9	20.0318	2.32576	6.93	0.004
Linear	3	11.1967	3.73223	11.12	0.002
X1	1	5.4010	5.40105	16.09	0.003
X2	1	3.4497	3.44968	10.28	0.011
X3	1	2.3460	2.34598	6.99	0.027
Square	3	6.1554	2.05179	6.11	0.015
X1*X1	1	3.6626	3.66264	10.91	0.009
X2*X2	1	3.2724	3.27235	9.75	0.012
2-Way Interaction	3	3.5797	1.19325	3.55	0.061
X1*X2	1	1.2561	1.25611	3.74	0.085
X2*X3	1	1.3366	0.33661	3.98	0.077
Error	9	3.0214	0.33571		
Lack-of-Fit	5	3.0184	0.60369	804.91	0.000
Pure Error	4	0.0030	0.00075		
Total	18	23.9533			

The predicted R-squared of (87.39%) was in reasonable agreement with the adjusted R-squared of 74.77%."Adequate precision" shows the extent of the predicted response regarding the associated signal-to-noise ratio. The data obtained showed that this model can be used to navigate over the design space.

Equation 1 shows the relationship between the coded values and the response factor (K/S).

$$K/S = 4.619 + 0.3770X_1 + 0.3013X_2 + 0.2485X_3 - 0.1869X_1{}^2 - 0.1766X_2{}^2 - 0.0717X_3{}^2 + 0.1417X_1 * X_2 - 0.1256X_1 * X_3 - 0.1462X_2 * X_3$$

(equation1)

The effect of parameters on color strength

Figure 1 shows the simultaneous effect of dyeing variables on the color strength of embelin ninhydrin-dyed cotton fibers. Figure 1(a) shows the simultaneous effect of dyeing pH and temperature on color strength. It can be observed that color strength has increased when pH increases from 6 to 9 and at a temperature of up to 80°C. The relationship between the pH and the color strength can be attributed to the correlation between the embelin ninhydrin dye molecule and the cellulosic structure at varied pH values. As observed in Figure 2, the dye molecule possesses -C = O and – OH, the hydroxyl groups are converted to alkoxide and which results in a molecule and the cellulosic structure at varied pH values with increased solubility and better diffusion to the cellulosic fibers, on the other hand, the carbonyl groups convert to negative charge at alkaline pH leading to increased color strength. Secondly, covalent bonds are also formed on the interaction of the quinone functional groups present in embelin ninhydrin dye with appropriate functional groups in the cellulosic polymer of the fabric. Thus, at high alkaline conditions, the embelin ninhydrin dye gets activated on interaction with O-nucleophile in the cellulose fabric, and a covalent bond forms according to Michael's addition mechanisms (Lewis 2014).Figure 1(b) shows the simultaneous effect of temperature and time on color strength. At a temperature of up to 80 C and a time of 60 min, the color strength is optimal. It shows that the optimum time for swelling the cotton fabric is 60 min and is sufficient to transfer the Embelin ninhydrin to the outer surface of the cellulosic fabric. This is due to dye molecules movement and pore sizes of the cellulosic fiber increasing with increased temperature enhancing the dye exhaustion enabled by kinetic energy which breaks the aggregated dye facilitating higher color strengths. It was found by Ayda et al., which increasing temperature favors cotton fiber swelling, leading to a higher dye uptake (Ayda 2019). At elevated temperatures further, increase in temperature leads to low color strength due to reduced affinity of dye to fiber. Figure 1(c), shows the simultaneous effect of pH and time on the color strength and is observed Increasing both factors up to a pH of 9 and time of 60 min there are optimal color strengths

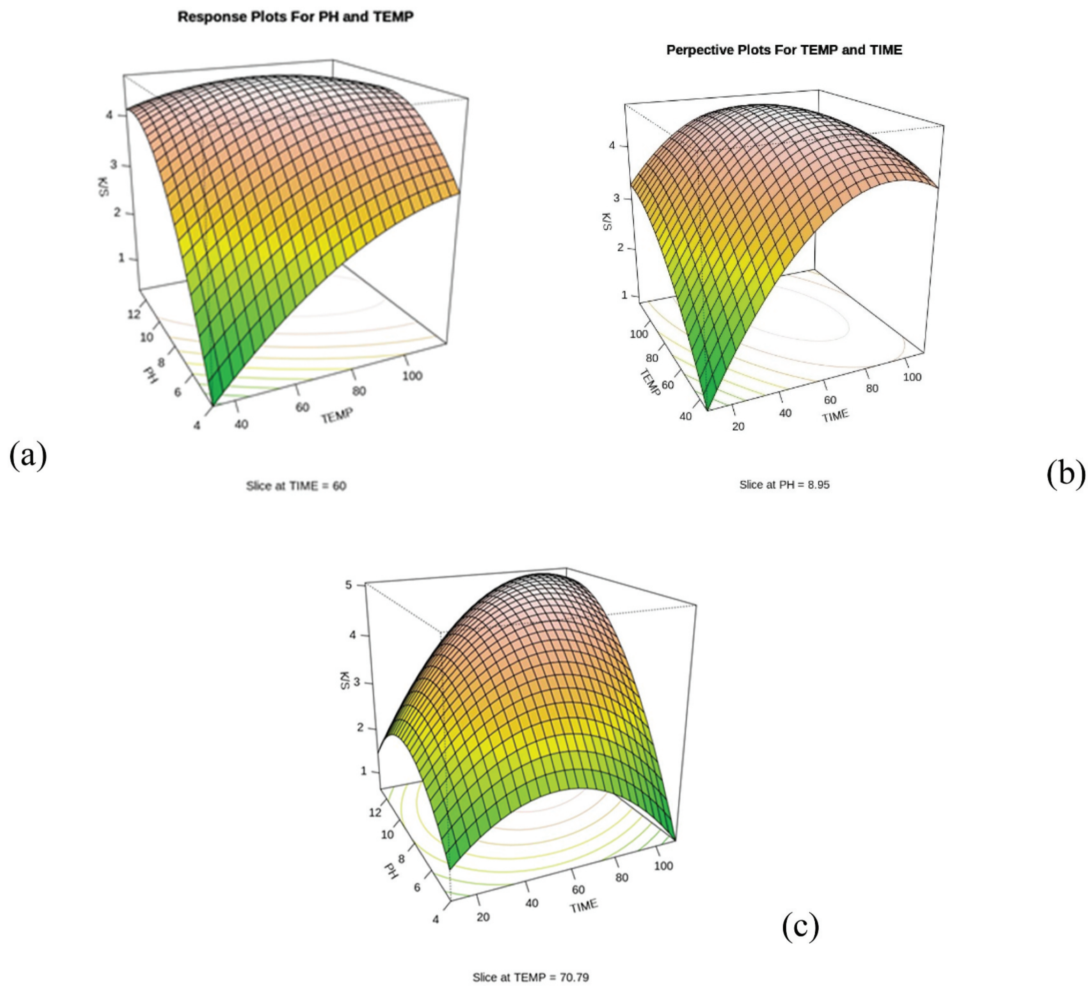

Figure 1. Plot of 3-D (a)- Temp vs Time (b) pH vs time (c) pH vs temp.

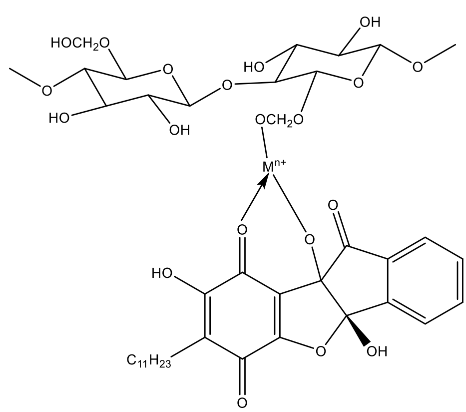

Figure 2. Interaction between embelin ninhydrin dye and mordanted cellulose (cotton) fiber.

Table 4. Color measurements of the dyed fabric using different methods of mordanting.

Method	Mordant	L*	a*	b*	ΔE	C*	H°	K/S	Shades
ENn dye	NO	57.70	+13.97	+9.65	27.40	16.97	55.36	4.02	
Embelin	NO	77.4	-2.47	+18.30	12.66	18.46	7.79	0.18	
Sim.	Copper	55.68	+10.24	+10.74	27.88	14.84	43.64	2.30	
	Ferrous	53.19	+4.32	+9.10	27.82	10.07	25.39	2.74	
	Alum	57.74	+9.88	+8.63	25.12	13.12	48.86	2.31	
	Nickel	36.94	-0.16	+10.26	43.20	10.26	0.89	2.51	
Post	Ferrous	46.22	+2.89	+15.04	36.49	15.32	10.88	4.04	
	Nickel	65.96	-1.31	+21.57	26.73	21.61	3.48	2.14	
	Copper	67.98	-0.10	+13.48	18.69	13.48	0.43	2.68	
	Alum	62.80	+12.77	+10.01	23.73	16.23	51.91	3.73	
Pre	Alum	70.71	+2.34	+6.69	11.80	7.09	19.28	2.46	
	Ferrous	56.11	+0.05	+12.67	27.78	12.67	0.23	4.09	
	Copper	76.94	-2.36	+5.73	8.34	6.20	22.39	2.27	
	Nickel	74.11	-0.74	+10.38	13.18	10.41	4.08	2.32	

Effect of mordants on Color characteristics

The addition of mordants to dyes was to create affinity between the dyes and fabrics resulting in different shades (Adeel et al. 2021). The results are given in Table 4 showing that the ferrous mordant, under pre, post, and simultaneous mordanting gave the highest color strength with K/S values of 4.09,4.04 and 2.74, respectively, with dark brown shades. Alum gave the second-best color strength in post-mordanting with color strength K/S 3.73 in violet shades and copper gave the best color strength of 2.68 in post-mordanting with violet shades. Nickel gave the lowest color strength with pre, post, and simultaneous values of 2.32, 2.14, and 2.51, respectively. The high color strength (K/S) values observed in ferrous mordanted cotton fabrics could be due to the chelate effect of d-block elements and the availability of free orbitals of ferrous metal that allow for the formation of bonds on interaction with dye and cotton fabric resulting in the formation of hydrogen bonds between cotton fibers, dye and mordants (Adeel et al. 2018). The Embelin ninhydrin-dyed cotton fabric without mordants gave a color strength of K/S 4.02. This color strength was greater than embelin-dyed cotton fabric with color strength of K/S 0.18. Compared with embelin ninhydrin mordanted cotton fabrics, the color strength was similar to ferrous mordanted. All the mordanted embelin ninhydrin dyed cotton fabric gave the excellent color fastness of 4–5 Table 5.

Model validation

Optimized dyeing conditions were validated by performing the experiments using values from optimized conditions. The optimized conditions gave a color strength of 4.71 whereas the dyeing carried out at a pH of 9, time of 60 mins and temperature of 80 C gave a color strength of 4.02 experimental. These showed no significant variation between the predicted and actual values. These optimum conditions were used in the evaluation of color fastness properties

Table 5. The color fastness of dyed fabric with different methods of mordant used.

		Wash fastness		Rubbing fastness		
Method	Mordant	C.C	C.S	Dry	Wet	Lightfastness
Embelin	NO	2–3	2	2	2	1
Embelin ninhydrin	NO	4	5	5	3	4
Simultaneous	Alum	5	5	5	4	4
	Copper	5	5	5	5	4
	Ferrous	4	5	5	4	5
	Nickel	5	5	4	4	4
Pre	Alum	5	5	4	4	4
	Copper	5	5	4	4	4
	Ferrous	5	5	5	4	4
	Nickel	5	5	5	4	4
Post	Alum	4	5	5	4	5
	Copper	4	5	5	4	4
	Ferrous	5	5	5	4	5
	Nickel	5	4	5	4	5

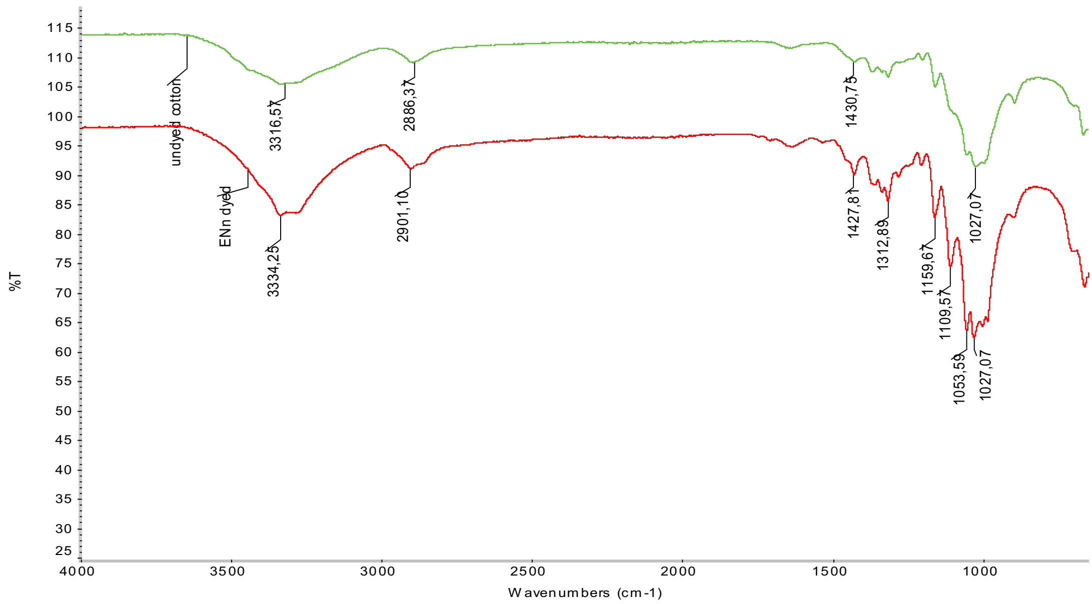

Figure 3. FTIR Spectra showing, embelin ninhydrin dyed cotton fiber and undyed cotton fiber.

FTIR analysis of blank cotton and embelin ninhydrin dyed cotton

The embelin ninhydrin dyed cotton fabric Figure 3 shows the characteristic cellulose structure fingerprints at 1027 cm^{-1} and an additional signal for the dyed fabric at 1110 cm^{-1}, this is due to the interaction between the dye auxochromes and functional groups present in the cellulose fabric as a result of C-O-C stretch according to spectra in Figure 2. Figure 3 is blank cotton fiber showing characteristic cellulose fingerprints at 1027 cm^{-1}.

Conclusion

A chemical modification of embelin to form embelin ninhydrin dye has resulted in a compound confirmed through various analytical techniques, such as FTIR. Its application as a dye has shown that it is an effective colorant with good fastness properties and higher color strength compared to most of its mordanted fabrics and the precursor embelin dye. The Response surface methodology established a model significant above 74.77%, which is statistically acceptable.

Research highlights

- The study introduced semi-synthetic embelin ninhydrin as a dye for the first time and its application as a dye to cotton fiber for the first time.
- The study investigated use of different mordants with embelin ninhydrin dye which gave different shades with cotton fabric and had good fastness properties (4–5) on the Grey scale for the first time
- The study came up with an optimized model of dyeing cotton fabrics with embelin ninhydrin dye at optimum values of pH 9, temperature of 80 ^{o}C and time of 60 min for the first time.

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Disclosure statement

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