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Novel Route for Dyeing of Cotton with Turmeric for Imparting Mosquito Repellent and UV Protection Properties

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ABSTRACT

Nowadays, sustainable approaches for the colouration of textiles are in high demand. Functional textiles remain a top choice for the user as they serve dual functions. The present study focuses on the utilisation of turmeric natural dye for dyeing of cotton using a novel method. A mosquito-repellent ethyl anthranilate was diazotised in the presence of sodium nitrite and hydrochloric acid. The diazotised derivative of ethyl anthranilate was reacted with turmeric-treated fabric by in-situ azoic dyeing method. The developed dyed fabric was evaluated for fastness and colour measurements. Functional effects provided by dyed fabrics (mosquito repellency and UV protection) were also assessed using standard methods. Dyed fabrics indicated excellent UV protection and 100 % mosquito repellency. The introduction of functional groups (azo and aromatic) of the developed dye in the cotton was also confirmed through FTIR analysis. A novel functional finishing-cum-dyeing approach for the functionalization of cellulosic fabrics was explored using turmeric extract and ethyl anthranilate. Prog. Color Colorants Coat. 17 (2024), 289-296© Institute for Color Science and Technology.

1. Introduction

Textile dyes are broadly classified into two categories: synthetic and natural. Natural dyes are obtained from plants, animals, and mineral matter. Synthetic dyes are the compounds synthesised from petrochemical-based raw materials. The world of synthetic dyes has grown rapidly, but some of them may harm the humans and ecology. Even though synthetic dyes are an economically viable option, their production involves the use of harmful chemicals. Synthetic dye effluents, when discharged into the environment, can cause environmental pollution. Thus, the interest in natural dyes has resurfaced in the textile industries.

With the growing need to switch to sustainable development, the harmful effects of synthetic dyes cannot be ignored anymore. In addition to the environmental concern, recent studies have also pointed to the threats of synthetic dyes towards human health [1, 2]. Some azo dyes, which are derivatives of benzidine-based compounds, release harmful amines, allergens, carcinogens, and other toxins that can be a source of cancer, allergies, and other adverse effects on the body [3].

Due to the increasing support for organic materials in all kinds of products, including fashion and lifestyle, the market for natural dyes has started to expand again. An interest in research and development of natural dyeing techniques is increasing. The major limitations of natural dyes are their low fastness to light, washing, and rubbing and their lighter shades. However, these problems can be attributed to the usage of these dyes in their crude form and can be solved by employing various techniques such as mordanting [4-7] for the application of natural dyes, thus allowing a wider range

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of very aesthetic colours as compared to synthetic dyes. Natural dyes, being pre-dominantly non-substantive, are applied to the fabric with the help of mordants, which have an affinity for both the dyestuffs and the substrate. The method of application of dyes depends upon the expected nature of attraction between the substrate and the dye molecule. Based on chemistries, bio [8-10] and metal mordants [11-14] are available. Some of the natural dyes have functional properties such as UV protection and antibacterial properties. Natural-dyed textiles can be used in diverse and highly demanding fields such as sports and tactical wear.

The extraction method of natural dyes plays a significant role in obtaining the desired yield of the principal component. Various techniques for extraction of natural dyes include solvent extraction, microwave-assisted extraction, ultrasound-assisted extraction, enzymatic extraction, fermentation, and aqueous extraction. The ultrasonic-assisted extraction of neem bark dye was performed, and the extract was utilized for the dyeing of silk [15]. Microwave irradiation was utilized to obtain an extract of Arjun Bark [16]. The dyeing of wool using madder natural dye and ethanolic extract of oak extract as a bio-mordant was performed [17]. A pretreatment of cotton using a gamma ray to enhance the natural dye uptake was performed [18]. The dyeing of wool using ultrasound-microwave-assisted extraction of myrobalan as bio-mordant and turmeric as natural dye was done [19]. An aqueous pomegranate extract was explored as a mordant in silk dyeing [20]. The dyeing of nylon using an acidic aqueous extract of Esfand (*P. harmala*) was performed [21]. Adeel et al. explored silk dyeing with coconut coir using ultrasonic-assisted technique [22]. The dyeing of wool with pomegranate peel was performed using an ultrasound-assisted method [23]. Microwave-assisted extraction and dyeing using Arjun Bark was done [16]. The dyeing of silk was performed with *Ficus religiosa* natural dye using a microwave-assisted process [24]. The colouration of cotton using acidified methanolic extract of *Terminalia arjuna* was explored [25]. The present work involves the alkaline extraction of turmeric in an aqueous medium.

Turmeric is obtained from rhizome of turmeric plant (*Curcuma Longa*) and appears yellowish-brown with dull orange interior. It is used as a food preservation agent, household medicine, and dyeing of textiles. Curcumin is the primary chemical constituent of turmeric [26], and it is responsible for yellow colour. Dyeing of cotton using turmeric dye was performed without mordant [27]. A pretreatment of cotton fabric with chitosan followed by dyeing with turmeric dye was performed by Kavitha et al. [28]. The dyeability of turmeric dye on cotton, wool [29], and polyester [30] has been studied. However, diazotised base-assisted fixation of turmeric to cotton has not been explored.

Ethyl anthranilate (EA) was reported as a safe and nontoxic mosquito repellent [31, 32]. Recently, it has been utilised in textiles for the synthesis of mosquito repellent dyes [32-36]. The present work explores a novel technique of dyeing of cotton fabrics with turmeric. The mosquito repellent intermediate diazotised ethyl anthranilate was reacted with turmeric dye *in situ* on fabric. The fastness and functional properties shown by developed dyed fabric were assessed.

2. Experimental

2.1. Materials

Dried turmeric rhizomes were obtained from a local grocery store in Delhi. The cotton fabric having picks/inch of 72, ends/inch of 96, and grams/square meter of 131.25 was utilised for the experiments. The chemicals such as ethyl anthranilate, sodium nitrite, sodium hydroxide, acetic acid, hydrochloric acid, sodium chloride, and sodium acetate were purchased from TCI and Sigma Chemicals.

2.2. Methods

2.2.1. Application of turmeric to cotton fabric

Dried turmeric root was ground thoroughly in a mortar and pestle till a fine powder was obtained. 10 g of turmeric powder and 1.61 g of NaOH were added to 100 mL of water. The solution was heated for 2 h at a temperature of 90 °C under constant stirring. The obtained dye stock solution had a concentration of 10 % (w/v) after making it to 100 mL.

Three different dyebaths were prepared with varying concentrations of turmeric (10, 20 and 30 %, owf). Sodium chloride was added to the dye baths (15 % on fabric weight) to increase the dye uptake to cotton. A material-to-liquor ratio of 1:15 was used. The application was performed in a Julabo water heating bath at 90 °C for 2 h. Dyed fabrics were squeezed gently to remove any excess absorbed liquor.

2.2.2. Diazotisation of ethyl anthranilate

Ethyl anthranilate (5.3 g, 32.07 mmol) was stirred with hydrochloric acid (36 %, 7 mL) at a temperature of 70 °C. The dissolved ethyl anthranilate was then cooled gradually to be brought to a temperature of 0-5 °C. $NaNO_2$ (2.21 g, 32.07 mmol) was added to the solution to perform the diazotization reaction. The reaction temperature was maintained in the 0-5 °C range by keeping it in an ice bath. The completion status of the reaction was checked by TLC (thin-layer chromatography) plate. Urea was added to reduce the leftover oxidising agent. Sodium acetate-acetic acid buffer was added to the solution gradually to neutralise the reaction mixture and to achieve a pH of 4-5. Finally, the solution was bought to a concentration of 4 % (w/v) by adding the required volume of deionised water.

2.2.3. Development of azoic dye based on turmeric

Three different dyebaths were prepared with 5, 10 and 15 % (on the weight of the fabric) concentrations of diazotized ethyl anthranilate. Turmeric-treated samples were immersed in the bath containing diazotised ethyl anthranilate, and the reaction was carried out in a Julabo water heating bath at 40 °C for 1 h. The dyed fabrics were washed thoroughly under tap water to remove excess chemicals and then washed further with dilute hydrochloric acid. The fabric samples were dried in the air. Concentrations of the turmeric and ethyl anthranilate used to prepare various samples are given in Table 1.

2.2.4. Analysis of colour values and colour strength of dyed cotton fabric

Computer colour matching system (Colour-Eye 7000 A) was used to determine the colour strength (K/S) and colour values (L*, a*, b*) of the dyed fabrics.

Table 1: Amount of raw materials used for synthesis of azoic dye.

Sample	Turmeric Dye (% owf)	Ethyl anthranilate (% owf)
1	10	5
2	20	10
3	30	15

2.2.5. Evaluation of fastness of dyed fabrics

ISO 105-C06 and 105-X12:2016 method were used to determine dyed fabrics' washing and rubbing fastness [37]. Lightfastness of dyed fabrics were obtained as per the ISO 105 BO2 method [37].

2.2.6. Evaluation of mosquito repellency and UV protection

The arm-in-cage method was used to evaluate the mosquito repellency of the dyed fabric samples [38]. AS/NZS 4399 method was used to determine the UV protection activity of dyed fabrics [39].

2.2.7. FTIR analysis of fabrics

Fourier transform infrared spectroscopy (Nicolet iS05) of the dyed fabric was performed to confirm the presence of the dye on the fabric.

3. Results and Discussion

3.1. Colour values of colour strength of dyed fabrics

Table 2 shows the results of colour measurements of the dyed fabrics. L* denotes the lightness value of the shade obtained, and as expected, it is the maximum for the sample with the least shade percentage applied (sample 1) and decreased gradually as the higher concentrations of turmeric and EA were used on the fabric. This suggested that the darkness of developed coloured shade on fabric increased with increasing shade (%). Positive a* values denote the redness, and positive b* values represent the yellowness of the shade obtained. Both a* and b* values were positive, and the values increased with increasing the concentration of turmeric and EA in the dyebath, thus denoting that the combination of turmeric and EA produced redder-yellower shade on cotton.

The colour strength (K/S) values also increased with increasing the shade (%), confirming the presence of more dye on the fabric with upgrading shade (%). Images of the dyed samples are also presented in Table 2 for comparison.

3.2. Colour fastness properties of the dyed samples

The fastness properties obtained for the dyed samples against washing and rubbing are summarized in Table 3. Dry / wet rubbing and washing fastness ratings were in the “very good-excellent” range. The light fastness obtained for the samples was in “moderate” to “good” level and increased with increasing shade (%).

Turmeric contains curcumin as a main component. The coupling position for the reaction of the diazotised base is established in the literature [40]. Based on that, Figure 1 records the dye's probable structure based on ethyl anthranilate and curcumin. The fixation can be attributed to the technique of azoic dyeing, as the final, coloured precipitate formed *in situ* in the cotton fibre by the azo coupling reaction between ethyl anthranilate and curcumin. The colour is water insoluble and does not come out easily from fibre, resulting in excellent fastness properties.

The developed azoic dye is water-insoluble and entrapped inside the fibre. Moreover, the synthesized azoic dye can interact with cotton through hydrogen bonding (Figure 2), wherein the hydroxyl groups of curcumin moiety can interact with hydroxyl groups of cotton. The interactions between natural dyes and textile fibres through hydrogen bonding are well-established in the literature [41, 42].

Table 2: Colour measurement results of dyed cotton.

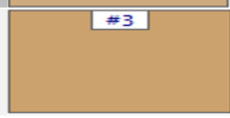
Sample	Colour values			Colour Strength	Colour appearance
	L*	a*	b*	K/S	
1	74.026	4.139	20.482	1.070	
2	71.912	6.810	26.797	1.611	
3	68.951	9.455	32.306	2.496	

Table 3: Fastness of dyed samples.

Sample	Washing Fastness	Rubbing fastness		Lightfastness
		Dry	Wet	
1	4-5	5	4-5	3-4
2	4-5	4-5	4-5	4-5
3	4	4-5	4	5-6

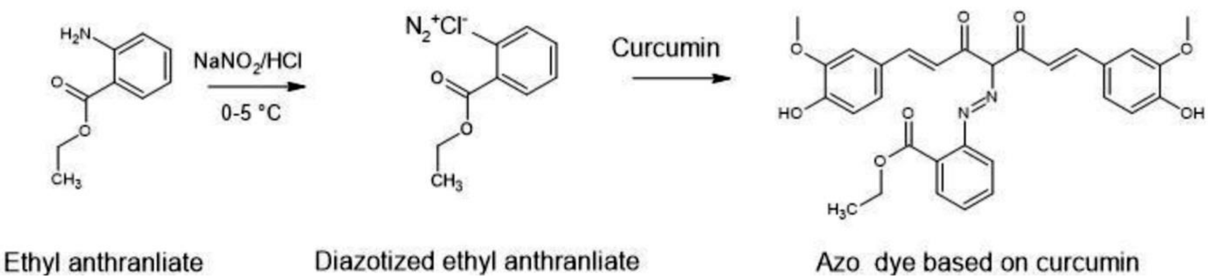


Figure 1: Chemical structure of curcumin-based dye.

3.3. Functional properties of dyed fabrics

The mosquito-repellent action of dyed cotton is recorded in Table 4. A minimum repellency of 98 % and a maximum of 100 % was observed, and an increase in repellency with an increase in shade (%) was also observed. This confirms the mosquito-repellent properties of dyed cotton. Ethyl anthranilate moiety was present on the dyed fabric, and the repellency based on the contact mode of action was obtained. Thus, the dye developed with the combination of turmeric and EA imparted the mosquito repellent protection to cotton.

The mean UPF value and the UPF ratings of dyed fabrics are mentioned in Table 5. A poor UPF rating was confirmed for undyed cotton. The dyed samples were found to have a UPF rating of 50+ i.e., offering maximum protection against UV radiations. Due to the UV-absorbing ability of the dye, UV protection activity was achieved. The dye absorbs the UV rays and prevents it from passing through the fabric.

3.4. FTIR spectroscopy of dyed fabrics

FTIR spectroscopic analysis with the marked major peaks is recorded in Figure 3. The characteristic bands of cotton fabric were obtained for C-H stretching at 2850 cm^{-1}, O-H stretching at 3280 cm^{-1}, and C-H bending at 895 cm^{-1} [38]. OH bending at 1650 cm^{-1} was obtained [38]. The introduction of dye’s functional groups such as N=N stretching at 1540 cm^{-1} [43] and =C-H stretching at 2976 cm^{-1} were also observed. Thus, the presence of dye's functional groups was confirmed on the dyed fabric. Azo group responsible for providing final shade to the *in situ* synthesized azoic dye was confirmed thrugh the FTIR spectrum of the dyed fabric.

Table 4: Mosquito repellency of dyed fabrics.

Samples	Mosquito repellency (%)
1	98
2	100
3	100

$ represents Sample 1-3 are the dyed fabrics prepared as per Table 1.

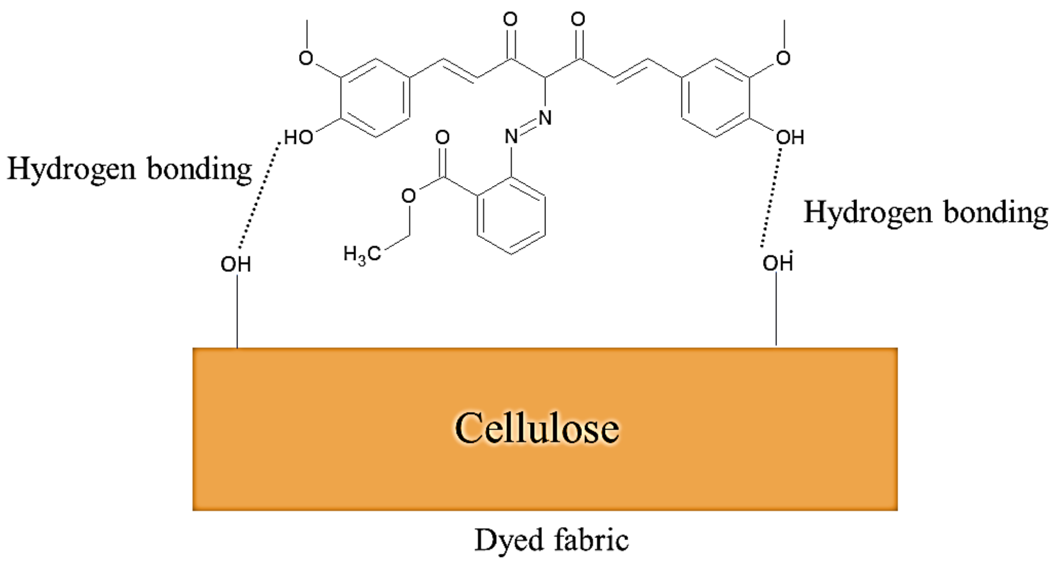


Figure 2: Interaction between turmeric-based dye and cotton.

Table 5: UV protection activity of dyed fabrics.

Sample	Mean UPF Value	UPF Rating
Undyed Cotton	8.96	Poor
1	74.49	Excellent
2	147.85	Excellent

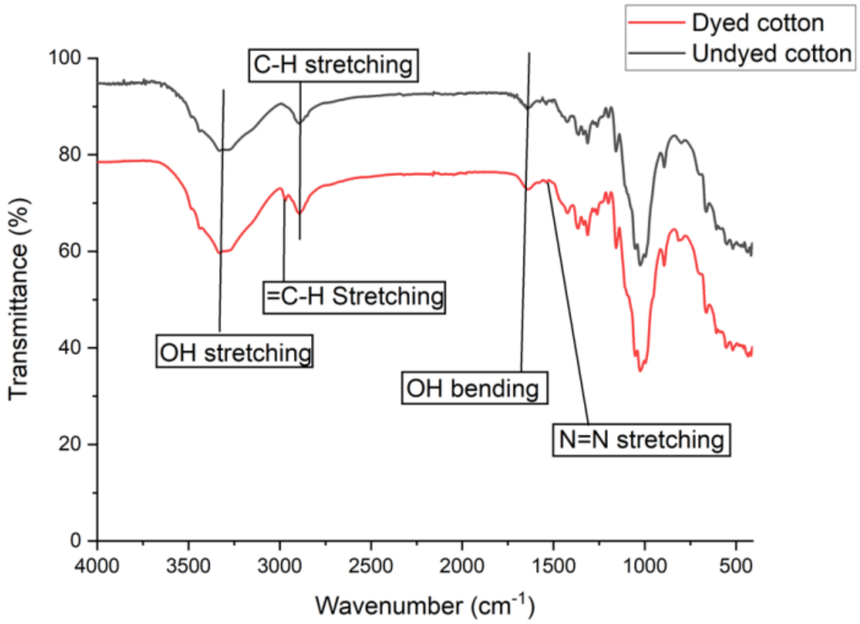


Figure 3: FTIR spectra of undyed and dyed cotton fabrics.

4. Conclusions

The dyeing of cotton fabric with turmeric and ethyl anthranilate was carried out successfully. The obtained colour and fastness properties were found to be excellent. An excellent level of mosquito repellency (100 %) was displayed by dyed fabrics, and an excellent UV protection rating was also confirmed for dyed fabric. FTIR Spectroscopic analysis revealed the presence of N=N bonds in dyed cotton, which was expected due to the presence of azo linkage in the dye molecule.

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