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Union Dyeing of Cotton-Silk Blended Woven Fabric Using Tanin Bio-mordant and Thyme as a Colourant

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Article

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ABSTRACT

The study explores the application of tea leaf-extracted tannin as a bio-mordant and thyme extract as a natural dye on cationized silk-cotton blend fabric to enhance colour fastness and absorption. The colour strength and depth (shown as DL, Da, and Db values) on silk-cotton blend fabric were investigated in the CIE lab test. The results showed that temperature, shade percentage, and mordanting had a substantial impact on the dyeing process efficacy at DL = 30.61, Da = 11.49 and Db = 12.77. When the temperature was raised to 90 °C and the dye content was increased to 120%, the dyed sample's colour fastness (as measured by wash, rubbing, and perspiration) significantly improved, acquiring a grade of 4 to 5, which indicates good to excellent. There was a slight change in wet rubbing fastness from grade 3 to grade 4, indicating good to fair. The K/S values of the silk and cotton fibres, which ranged from 8.89 to 7.94 on average, respectively, demonstrated that the processes of bio-mordanting and cationization enhanced the absorption of dye. Tensile strength and extension increased in the weft direction to 22.19 N force and 5.21% extension at break, while the warp direction showed a fall to 6.63 N force and a rise in extension of 0.72%. FTIR spectroscopy confirmed the presence of Amino (N-H) bonds, Carbohydrate bonds (CH), Tannins, and Terpenoid phytochemicals of thyme in the dyed sample. The study highlights the potential of thyme extract as a natural dye and its benefits in improving the dyeing properties of silk-cotton blend fabric.

KEYWORDS

union dyeing, cotton, silk, bio-mordant, thyme, tannin, cationization

INTRODUCTION

The textile industry stands at a crossroads where the pursuit of sustainability intersects with the demand for fabrics that meet evolving standards. In this dynamic landscape, natural dyeing methods have emerged as a promising avenue for reducing environmental impact while enhancing textile properties. Cotton and silk, two venerable natural fibres, bring their unique characteristics to textile blends [1,2]. Cotton, known for its breathability and comfort, contrasts with silk, which offers luxurious softness and a natural sheen [3,4]. These inherent differences create challenges and opportunities in the dyeing process, making it imperative to develop techniques that cater to the distinct nature of these fibres since cotton is anionic and silk is cationic when immersed in water [5,6].

The applications of different synthetic chemicals have been a matter of worry as these can hamper nature in a great manner [7]. The method of cationization eliminates the need for electrolytes in the cotton dyeing process by introducing positively charged sites that facilitate the establishment of an electrostatic attraction between the negatively charged dye molecules and the fibre [8]. Cationization is a necessary step for better dyeing and fixing of natural dyes with cotton fabrics. The process is conducted before mordanting [9,10].

Mordants are typically metallic salts or chemical compounds that react with the dye and the fibres to create a stable bond. Commonly used mordants include alum (potassium aluminium sulfate), iron, copper, and tin salts [11]. When textiles are treated with a mordant before dying, it can alter the colour and improve the longevity of the dye, making it more resistant to fading and washing [12]. On the other hand, bio-mordants are derived from natural sources, such as plants and minerals. They are considered more environmentally friendly and sustainable alternatives to traditional metallic mordants [1]. These bio-mordants create a chemical bond between the dye, fibre, and the mordant, like traditional mordants but without the environmental concerns associated with heavy metal mordants [13]. Moreover, natural mordant has shown great antimicrobial and UV protection properties [14].

Thyme (Thymus vulgaris L.), a fragrant herb known for its culinary and medicinal properties, is also a natural dye source. Thyme leaves and stems can yield shades of yellow and green when used as a dye material, making it a sustainable and environmentally friendly option for natural dyeing [13]. Bio mordants derived from natural substances like tannins found in plant extracts or alum sourced from minerals can enhance colour fastness and improve dye uptake [15].

Several studies were conducted on bio-mordants on natural fibres. A study was on thyme, and pomegranate peel to cotton fabrics without metallic mordants [1]. In another paper, the authors used Fuji apple peel extract for silk and cotton fabric dyeing. This study explored the extraction of natural dyes from Fuji apple peel and their application in dyeing silk and cotton fabrics with and without mordants (Mg^{2+}, Ca^{2+}, Al^{3+}, Ti^{4+}, Fe^{3+}, and Cu^{2+}). Two anthocyanins and five quercetin glycosides were identified in the extract. The results indicate that silk achieved deeper shades than cotton, attributed to the higher presence of functional groups and lower crystallinity in silk. Mordant dyeing produced various colours, and Ti^{4+} and Fe^{3+} mordants improved colour fastness. Fuji apple peel dyeing enhanced antibacterial properties and UV shielding in both fabrics, with silk showing sufficient UV protection. The study suggests that natural dyes from Fuji apple peel possess high antibacterial and UV-shielding properties, opening possibilities for their application in textiles [14]. Another study on the use of sage and thyme as natural dyestuffs in the colouring of woollen garments was recently carried out, and the results showed that these plants can be utilized as a natural source of dyes for woollen fabrics [16]. A prior study used bio-mordants based on tannin for the natural dyeing of wool using root extract from madder [17]. The use of tannin as a natural mordant for dyeing cotton fabric was applied successfully and found to enhance colour fastness properties [18]. Individually, several works were conducted on cotton and silk dyeing. To maintain nobility and approach innovatively cotton-silk blend fabric is used in this article. Regarding the presence of flavonoid and phenolic acid compounds, there is a study aimed to prepare crude extracts from Thymus vulgaris collected in Al Jabal Al Akhdar, Nizwa, Sultanate of Oman, using different solvents and assess their total phenol and flavonoid contents, alongside conducting phytochemical screening. Initially, leaves were methanol-extracted, followed by defatting with water and subsequent extraction with solvents of increasing polarity (hexane, chloroform, ethyl acetate, butanol, and methanol). Quantitative determination of total phenol, flavonoids, and phytochemical screening were conducted using established methods. Results indicated a positive presence of flavonoids, saponins, and steroids. Notably, butanol extract exhibited the highest total phenol content, while methanol extract displayed the highest total flavonoid content [19]. And also there is a study that explored shade's impact on flavonoid biosynthesis in tea leaves. It found that shade reduced flavonoid concentration, particularly proanthocyanins and O-glycosylated flavonols, without affecting anthocyanin levels. Shade affected the polymerization of catechins and flavonol glycosylation. Phenolic acid levels increased in shaded leaves, potentially competing with lignins and flavonoids for substrates [20]. In a study, it has been found that the Anatolian acorn (Quercus ithaburensis Decne) offers a sustainable alternative to synthetic dyes for textiles and leather. Its eco-friendly properties, unique colours, and antimicrobial qualities make it valuable for regional trade, job creation, and advancing sustainability goals in Turkey and globally [23]. An analysis of dyeing organic cotton fabrics with Rhamnus petiolaris Boiss, Quercus ithaburansis Decaisne, and a combination of both using microwave irradiation and conventional methods revealed colouring compounds including kaempferol, quercetin, rhamnetin, ellagic acid, and gallic acid derivatives [24].

However, as per the author's knowledge, the application of tannin as a bio-mordant on a cotton-silk blend with thyme as a colourant is still unstudied. Thus, this research project introduces an innovative approach to natural dyeing using thyme as a natural colourant. The project stands out for its comprehensive methodology, including bio-mordanting with tea-derived tannin and cationization of cotton fibres to enhance union dyeing with silk. Tea (*Camellia sinensis*) used in the project contained tannin, gallic acid, epigallocatechin gallate, epicatechin, epicatechin gallate and caffeine acid [18]. These techniques enhance colour quality and align with eco-conscious textile practices. The study goes beyond traditional natural dyeing projects by conducting various tests, such as colourfastness, K/S values, FTIR testing, tensile strength, and spectrophotometry evaluation.

EXPERIMENTAL

Materials and Methods

70% Cotton 30% Silk blended woven bleached fabric has been used for the study. The Ends per inch (EPI) and Picks per inch (PPI) were 106 and 200 respectively. The Grams per square meter (GSM) of the fabric was 61. The wetting agent (collected from Dysin Chem Ltd) was Jingen DT HLF, Switzerland; the Sequestering Agent was Lufibrol 2UD, Switzerland; the Acetic Acid was Acetic Acid (100%) Supelco, Germany; the Cationic Agent was Optifix, Switzerland; Soaping Agent was Cotoblanc SEL, Switzerland.

Tannin Extraction

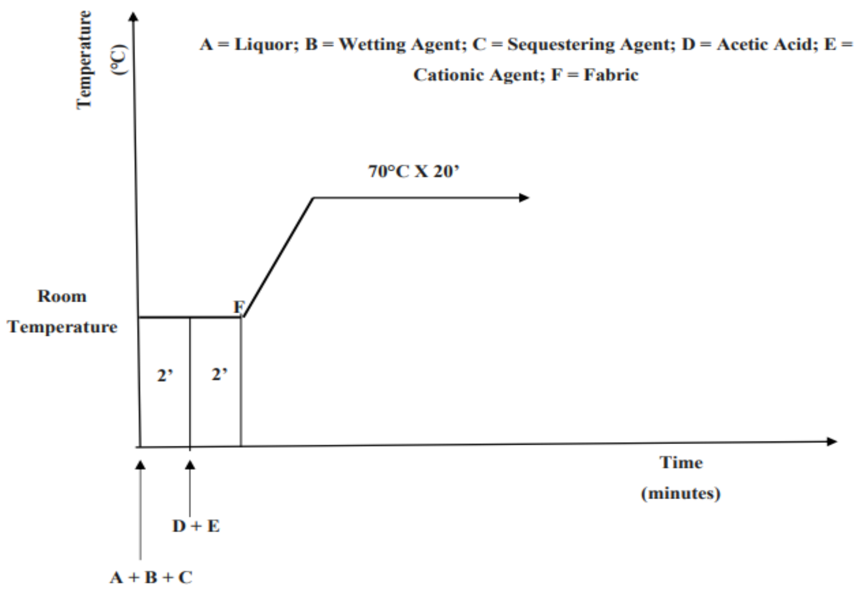
Dried Tea was collected from the source. 10 g of tea was boiled using 120 ml of water for 60 minutes. This process was repeated three times. Tea residue was filtered using filter paper. Three to four droplets of tea water were placed in a test tube to assess tannin. In that test tube, 1 drop of ferric chloride was added. If red became black/blue/brown, Tanin was present. The spot test for tannin analysis was performed accurately. The extracted Tanin Solution was stored in a conical flask. It was ensured that the extracted solution was not in contact with air (Figure 1).

Preparation of Thyme

Dried Thyme leaf was collected from a local shop. It was ground until a fine powder form was obtained. The powder was stored in an airtight container.

Cationization

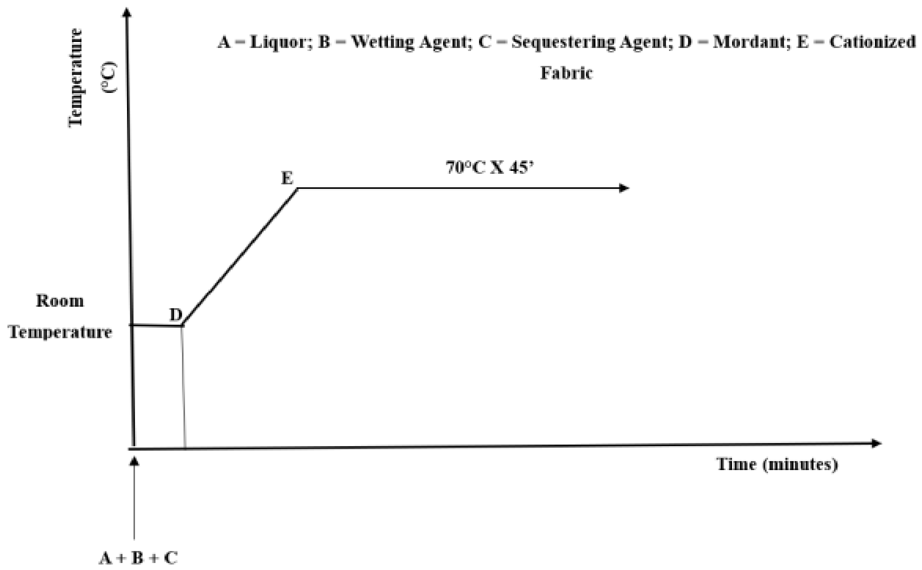
To cationize the fabric, the required amount of liquor (1:20) was taken. To obtain acidic medium acetic acid (2 g/l) was added to the bath. To cationize the cotton part of the fabric, a cationizing agent (50% o.w.f) was added to the bath. The bath was poured into a closed vessel of the lab dyeing machine including the fabric. Then the temperature was raised from room temperature to 70 °C and the machine was run for 20 minutes at 70 °C (Graph 1).



Graph 1. Process diagram of cationization of cotton-silk blended fabric

Mordantation

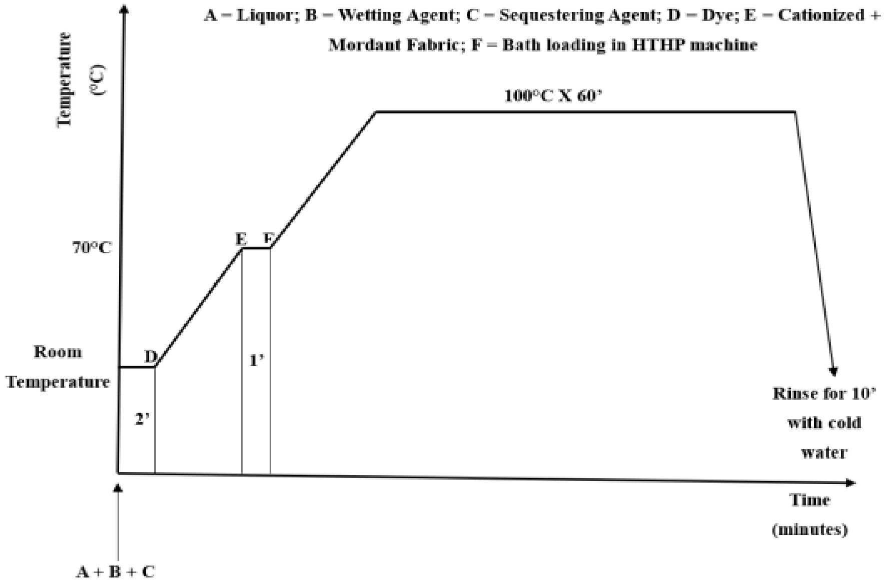
For the application of mordant, the required amount of liquor (1:20) was taken. The temperature was raised to 70 °C. Mordant (50% o.w.f) was added to the liquid. The bath was poured into a closed vessel of a lab dyeing machine including the cationized fabric. Then temperature was raised from room temperature to 70 °C and the machine was run for 45 minutes at 70 °C. This procedure was repeated for 10%, 20%, 30%, 40% mordant variation (Graph 2).



Graph 2. Process diagram of natural mordant (tannin) application on cotton-silk blended fabric

Union dyeing with thyme colourant (HTHP method)

For dyeing, the required amount of liquor was taken. Thyme powder (150% o.w.f) was added to the liquor and the temperature was raised to 70 °C. Pre-treated fabrics were added to the dye bath. The bath was poured into a closed vessel of the lab dyeing machine including the fabric. The temperature was raised from 70 °C to 100 °C. The machine was run for 60 minutes at 100 °C. Cooling was done when the temperature was decreased to 70 °C from 100 °C. After that, the cold rinsing was done for 10 minutes. The above procedure was repeated for the shade variation of 30%, 60%, 90%, 120%. Also, variation was made for temperature 60 °C, 90 °C, 120 °C (Graph 3, Figure 1).

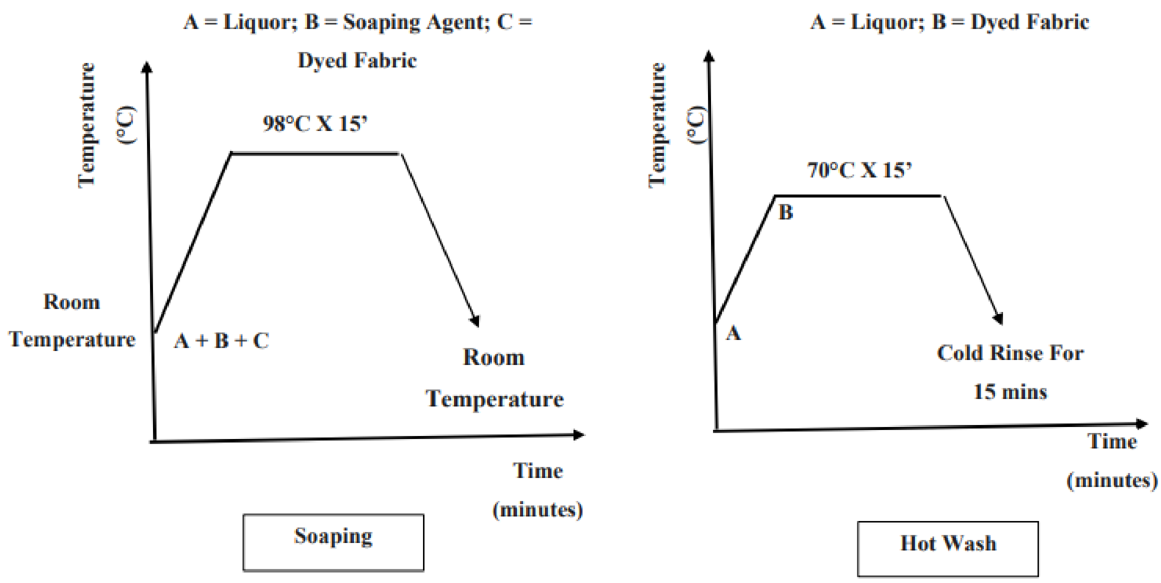


Graph 3. Process diagram of dyeing of cotton-silk blended fabric with natural colourant thyme

Figure 1. Process flow of Dye and Mordant preparation for dyeing of cotton-silk blended fabric

After treatment

To remove unfixed and excess dye from the fabric surface after treatment was performed. Soaping was done for 15 minutes at 98 °C using a Soaping Agent (0.5 g/l). Again, hot wash was done at 70 °C for 15 minutes. Cold rinsing was performed for 15 minutes. A hand dryer was used for drying the dyed fabric (Graph 4).



Graph 4. Process diagram of soaping and hot washing of cotton-silk blended fabric

Evaluation of Colourimetric Properties

Different calorimetric properties of dyed fabric like L*a*b* and K/S value of dyed fabrics were measured in a data colour machine (data colour 650 TM). Since the fabric has two faces, one of which is lustrous and the other less shiny, the K/S value analysis was taken individually. On average, 2 repeats were done for each sample. The measurement of this value is conducted using a spectrometer. The illuminant used standard D65 which is a commonly used daylight illuminant, representing noon daylight (6504 K).

Evaluation of Colour Fastness

Colour fastness to washing, colour Fastness to Perspiration and colour Fastness to Rubbing of the dyed fabrics were measured following the ISO standard respectively: ISO 105-C06:2010 C2S, ISO 105-E04:2013, ISO 105-X12:2016.

Tensile Strength Test

The tensile strength of the dyed samples was compared with undyed samples by tensile strength tester. The ASTM D 5035-11 standard was followed for the analysis strength of the fabric.

Scanning Electron Microscope (SEM)

To ensure that the dyes were successfully deposited on the cloth, treated and untreated fabric were examined under a scanning electron microscope (SEM).

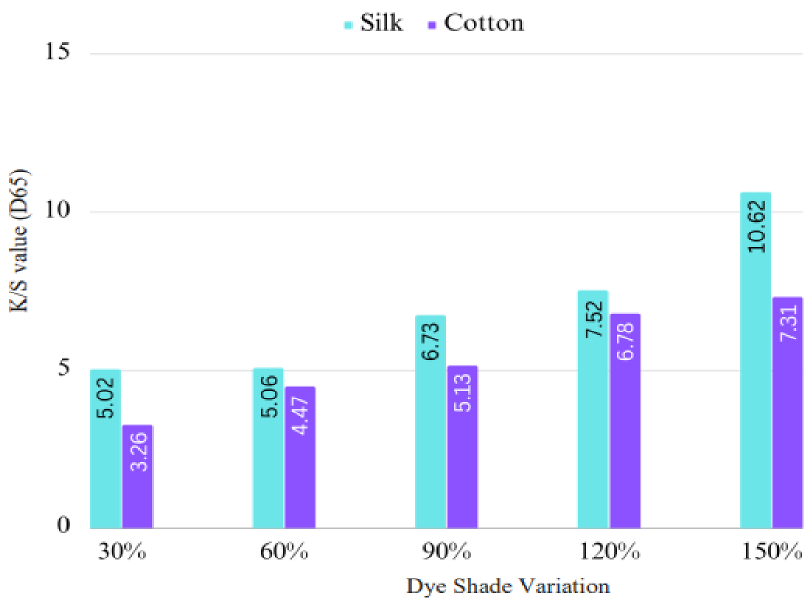
Fourier Transform Infrared Spectroscopy (FTIR) Analysis

FTIR of dyed and undyed samples was conducted by Spectrum two FTIR spectrometer, Perkin Elmer following standard procedure.

RESULTS AND DISCUSSION

Analysis of K/S Value

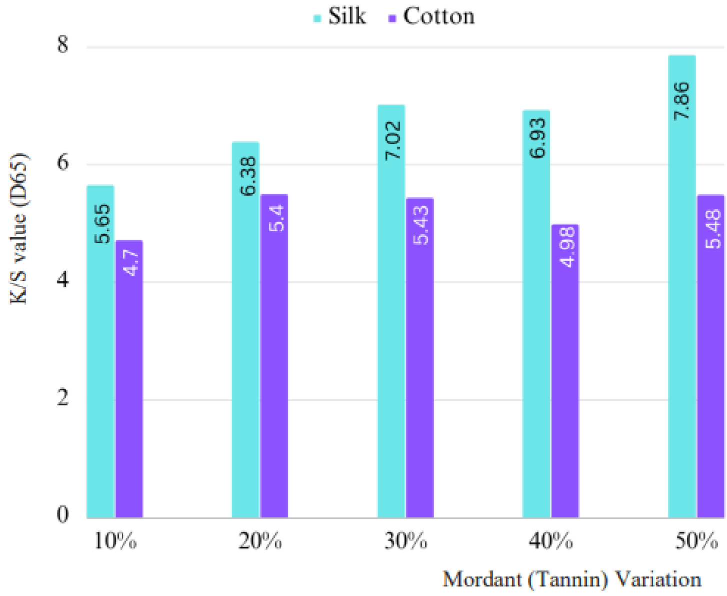
From a theoretical standpoint, the shiny part of the fabric was assumed to be the silk part, and the less lustrous part was assumed to be the cotton part. During the trial-and-error period, the fabric was acid-dyed with cationization to ensure both silk and cotton had the same ionic nature. After dyeing, it was observed that the dye uptake of the shiny side was comparably higher than that of the less lustrous part. The K/S (colour strength) values obtained for the dyed silk-cotton blend fabric at varying shade levels reveal distinctive patterns. In the silk part of the fabric, K/S values increase progressively with higher shade levels, indicating a pronounced affinity of the thyme dye for silk fibres, resulting in more intense colours. From (30-60%), the value showed a very marginal difference of 0.04 on the silk face and the change is proportional up to 120% shade. But when it crossed almost 150% shade of dye; silk face K/S value just jumped near a 3.42 difference change. Conversely, the cotton portion displays lower K/S values at each shade level, suggesting a comparatively milder colouration effect (Graph 5).



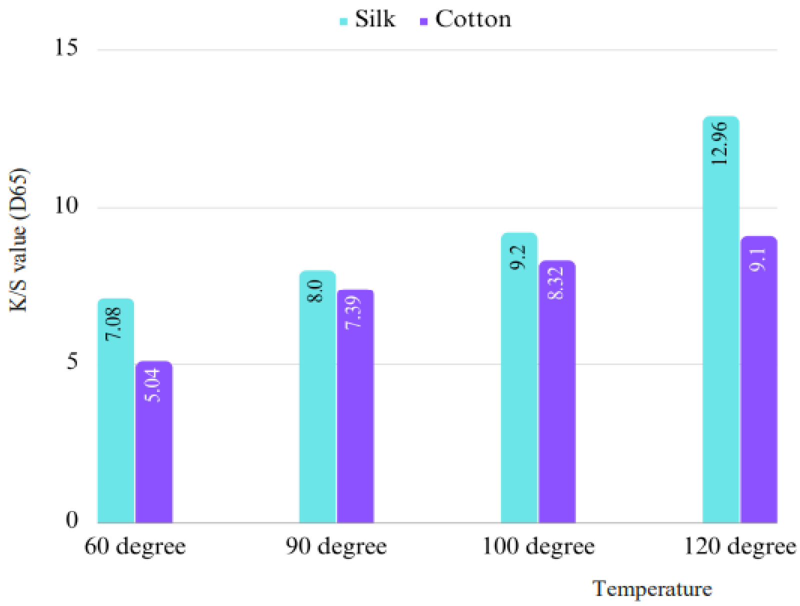
Graph 5. Graphical representation of K/S value analysis on Dye variation

The impact of mordant variation on dyed silk-cotton blend fabric was analyzed by assessing K/S values with varying tannin mordant concentrations. The K/S values showed an upward trend on the silk part as tannin mordant concentration increased, suggesting heightened colour intensity and dye absorption. Similarly, the cotton part exhibited a similar trend, albeit with slightly lower K/S values. Notably, the silk portion consistently demonstrated higher K/S values than the cotton, indicating that the thyme dye had a stronger affinity for silk fibres, resulting in more vivid colours. The optimal tannin mordant concentration was aligned with the desired colour intensity, with higher concentrations yielding deeper colours and lower concentrations producing softer tones, since mordants serve as a link between the fibre and the dyes, being substantive to both. This allows dyes with minimal affinity for textiles to be effectively fixed onto the substrate with the assistance of mordants [22]. These findings underscored the significance of controlling tannin mordant levels to achieve the desired colour outcomes while considering the fabric's intended application (Graph 6).

The K/S (colour strength) values, measured under varying temperature conditions, provided valuable insights into the impact of temperature variation on the dyed silk-cotton blend fabric. K/S values exhibited a consistent pattern of increasing colour intensity as the temperature increased in both the silk and cotton parts of the fabric. Specifically, as the temperature rose from 60 to 120 °C, the K/S values steadily increased, signifying enhanced dye absorption and deeper colouration. Notably, the silk component consistently displayed higher K/S values than the cotton, highlighting the pronounced colouration effect of the thyme dye on silk fibres. The choice of the optimal temperature level was dictated by the desired colour intensity, with higher temperatures resulting in more vibrant colours and lower temperatures producing softer hues. Ensuring uniform colouration across both silk and cotton sections was imperative, potentially necessitating adjustments in the dyeing process for uniformity (Graph 7).



Graph 6. Graphical representation of K/S value analysis on Mordant variation



Graph 7. Graphical representation of K/S value analysis on Temperature variation

Spectrophotometry Evaluation of Dyed Sample (DL, Da, Db, Dc, Dh)

Table 1 shows the values where dye shade percentage (90-150%) was changed respectively while other parameters such as temperature, mordant and cationic agent remained constant. As the dye shade percentage increased, the fabric's silk and cotton parts exhibited consistent colour shifts. The silk part marked this by decreasing DL values, signifying a transition to darker colours. In contrast, the Da and

Db values became more positive, indicating a heightened presence of red and yellow hues and an overall warmth in colour tones. The silk part showed the deepest and warmest hues at the highest dye shade percentage of 150%, with DL at -36.01, Da at 12.65, and Db at 19.75. The cotton part displayed a similar trend, decreasing DL values and shifting towards red and yellow hues along the Da and Db axes as the dye shade percentage increased. At 150% dye shade, cotton exhibited pronounced colour changes, with DL reaching -34.22, Da at 10.90, and Db at 16.36, highlighting the substantial influence of dye shade percentage on enhancing colour depth and warmth. These findings underscore the potential for tailoring colour outcomes in the fabric's silk and cotton portions by adjusting dye shade percentages, which is essential for meeting specific design and aesthetic preferences in textile applications.

Table 1. Effect of Dye variations (Shade percentage) on colourimetric properties

Dye Variations	Lab Values for the face side of the fabric				
(Shade Percentage)	DL	Da	Db	Dc	Dh
30	-26.54	7.33	16.79	18.26	-1.88
60	-28.08	8.23	15.85	19.29	-2.29
90	-29.56	9.88	17.46	21.21	-3.19
120	-31.77	10.94	18.31	22.89	-4.64
150	-36.01	12.65	19.75	23.67	-4.99

The CIE Lab values, representing colour characteristics, were analyzed for the dyed silk-cotton blend fabric at varying mordant variation percentages. These values revealed substantial shifts in colour attributes for the fabric's silk and cotton portions as mordant percentages changed, although Mordants enhance colour stability by facilitating chemical bonding between dye and fibre molecules. But colourimetric properties are minimally affected by mordanting, with slight changes observed in L*, a*, and b* values compared to untreated samples [23]. The trend indicated a transition towards more profound and darker colours as mordant percentages increased, as reflected in the luminance (DL) values. Additionally, changes along the green, red (Da) and blue-yellow (Db) axes indicated alterations in colour tone, with positive values suggesting shifts towards red and yellow hues and negative values indicating shifts towards green and blue hues. Notably, the silk part exhibited more pronounced shifts in CIE Lab values than the cotton part, underscoring the role of fibre type and interactions in the dyeing process. These findings offered valuable control over colour aesthetics for the silk-cotton blend fabric, facilitating the customization of colour outcomes to meet specific design and aesthetic requirements, which were vital considerations for various textile applications. Further assessment of colourfastness and overall fabric performance was recommended (Table 2).

Table 2. Effect of Mordant (percentage %) variations on colourimetric properties

Mordant (percentage %) variations	Lab Values for the face side of the fabric				
	DL	Da	Db	Dc	Dh
30	-28.29	9.27	15.87	16.95	-1.43
60	-31.69	10.83	17.00	17.57	-1.99
90	-33.16	11.17	17.26	18.87	-2.64
120	-32.1	10.83	17.55	19.43	-3.49
150	-34.56	11.72	17.17	21.25	-5.01

Assessment of Colour Fastness to Wash (ISO 105 C6 C2S:2010)

From Table 3-5, colour fastness to wash was overall found to be appreciable loss to slight loss in depth (Mordant variation), and slight loss in depth (Dye variations and temperature variations). In a nutshell, overall colour fastness was found to be good to excellent when the temperature rose from low to high. The higher the temperature went, the staining probability changed from very light to no staining. Quite noticeable was that overall shade didn’t change until it reached 120 °C and a minimum threshold of 40% mordant. Furthermore, higher mordant dosing and high shade percentage have a significant impact on the fixation of the dye but at a certain level of course since primary challenges with natural dyes include limited availability, difficulties in collection, and standardization of application methods. Factors such as cost and poor colour fastness properties also pose significant obstacles [23]. It was found that the increase in the mordant percentage in thyme-dyed cotton-silk blend fabric improved colour fastness. By serving as a mediator, the bio-mordant helped the dye molecules and the fabric interact more strongly. Because the dye was less likely to leak out due to the increased binding, the colour resisted fading during washing.

A higher shade percentage also suggests a more concentrated dye application, which results in a deeper and richer colour. Better colour fastness may result from this saturation since there is more dye available to create strong bonds with the fabric. Colour fastness can also be improved by raising the temperature during the dyeing process. Increased heat can encourage stronger bonding and better dye penetration into the fibres. Since silk fibres are typically harder to dye than cotton, this is particularly important for blends of cotton and silk.

Table 3. Effect of Mordant Variation on Colour fastness to wash of dyed fabric

Mordant variation (%)	Colour Staining						Shade Change
	Acetate	Cotton	Nylon	Polyester	Acrylic	Wool	
30	4/5	4	4/5	4	4/5	4/5	3/4
60	4	4/5	4/5	4/5	4	4	3/4
90	4/5	4	4/5	4/5	4/5	4	4
120	4/5	4	4/5	4/5	4	4/5	4
150	4/5	4/5	4/5	4/5	4/5	4/5	4

Table 4. Effect of Dye Variations (Shade percentage) on Colour fastness to wash

Dye Variations	Colour Staining						Shade Change
(Shade Percentage)	Acetate	Cotton	Nylon	Polyester	Acrylic	Wool	
30	4	4	4	4	4/5	4/5	3/4
60	4	4/5	4	4	4/5	4/5	3/4
90	4/5	4	4/5	4/5	4/5	4	4
120	5	4	5	4	4/5	4/5	4
150	5	5	5	5	5	5	5

Table 5. Effect of temperature variation (°C) on Colour fastness to wash

Temperature Variations	Colour Staining						Shade Change
(°C)	Acetate	Cotton	Nylon	Polyester	Acrylic	Wool	
60	4	4	4	4	4/5	4/5	4/5
90	4	4/5	4	4	4/5	4/5	4/5
100	4/5	4	4/5	4/5	4/5	4	4
120	5	5	5	4/5	4/5	5	5

Assessment of Colour Fastness to Perspiration (Alkali) (ISO 105-E04:2013)

Regarding colour fastness to perspiration test (Table 6-8), overall colour fastness was found to be very slight to no staining. The abrupt change of overall shade became less, and colour staining occurred with minimal effect when the temperature rose (In the temperature table) but the most significant change occurred when the dye variation changes occurred, while in 150% shade, it showed quite a performance while having the minimum colour staining at all. From 120-150% it showed almost the same colour staining as well as shade change. But at a certain level of mordant 30-40%, it showed no further change in improvement of colour staining as well as overall shade change. In the mordant variation table, after reaching approximately 30 per cent mordant threshold, colour staining decreased from very slight staining to no staining at all. Then comes the impact of temperature, where it has a clear impact such as when after crossing 90 °C; colour staining went from very slight staining to no staining and at 120 °C no staining occurred in every fibre component.

Based on the result above, it can be concluded that the primary reason for an increase in colour fastness to perspiration is a stronger bond between the dye molecules and the fibres [22]. The interaction between the dye and the textile is enhanced and stronger bonds are formed when the percentage of bio-mordant is raised [22]. A strong bond between the dye and fibres is essential in the context of perspiration, as it contains salts and other compounds that could cause the dye to fade or leach out when exposed to it. Higher bio-mordant percentages improve binding, which keeps dye molecules firmly attached to the fabric and lessens the chance of colour fading or changing when in contact with perspiration.

Table 6. Effect of mordant variation on Colour fastness to perspiration

Mordant variation (%)	Colour Staining						Shade Change
	Acetate	Cotton	Nylon	Polyester	Acrylic	Wool	
10	4	5	5	5	4/5	4/5	4
20	5	4/5	5	4	4/5	4/5	4
30	4/5	4/5	4/5	4/5	5	4	4
40	5	4	5	4	4/5	5	5
50	5	5	5	5	5	5	5

Table 7. Effect of Dye Variations (Shade percentage) on Colour fastness to perspiration

Dye Variations	Colour Staining						Shade Change
(Shade Percentage)	Acetate	Cotton	Nylon	Polyester	Acrylic	Wool	
30	4	4	4	4	4/5	4/5	4
60	5	4/5	4	5	5	4/5	4
90	4/5	4	5	5	4/5	4/5	5
120	5	4	5	4/5	4/5	4/5	5
150	5	5	5	5	5	5	5

Table 8. Effect of Dye Variations (Shade percentage) on Colour fastness to perspiration

Temperature Variations	Colour Staining						Shade Change
(^{o}C)	Acetate	Cotton	Nylon	Polyester	Acrylic	Wool	
30	4	4	4	4	4/5	4/5	4
60	5	4/5	4	5	5	4/5	4
90	4/5	4	5	5	4/5	4/5	5
120	5	4	5	4/5	4/5	4/5	5

Assessment of Colour fastness to Rubbing (Wet and Dry) (ISO 105-X12:2016)

From Table 9-11, colour fastness of rubbing was found moderate to very slight staining on the cotton part and very slight to no staining on the silk part. Significant staining has been observed in the temperature variation and mordant variation. In dry and wet, mordant change proportionately affected the wet rubbing fastness although it had quite a marginal effect on dry rubbing performance such as moderate to very slight staining. On the other hand, while the temperature goes up, the wet rubbing performance showed quite unstable performance on the cotton part although the silk side stays unchanged in staining. However, in a study by Ramratan et al., the highest treated time 65 minutes achieved a 5 grade and the lowest time 35 minutes had obtained a 4 grade. The brilliant dry rub fastness properties of silk fabrics were obtained when the temperature was 65 °C. The highest dyeing time value of the rubbing fastness according to runs 7, 8, 11 and 12 is 5 grade and the lowest value is 4 grade. According to the study of Barahapurkar et al. using Banana pseudo stem sap as bio-mordant for dyeing silk with celosia fibre, mordant concentration ranging from 8% to 22% was tested at pH 4.0, 70 °C, and 80 minutes. The highest K/S values were achieved at 18% concentration for all dyeing methods, with decreases observed beyond this point possibly due to increased desorption. Mordant concentration was set at 18% for further optimization of pH and temperature while keeping time constant at 80 minutes [21]. But in this test, mordant variation didn’t make any significant impact both on the dry and wet part (Moderate to very slight and very slight staining at 50%) but at 150% dye shade and 120 °C, no staining occurred on both silk and cotton for dry rubbing section.

According to the result above, it can be said that the degree to which the dye adheres to the fabric fibres is directly related to how colour fastness to rubbing is advanced. You can increase the textile's ability to bind dye molecules by increasing the percentage of bio-mordant used. An even more robust and long-lasting colour is produced by this increased binding, which guarantees that the dye stays securely bonded to the fibres. Greater adherence between the dye and the fabric reduces the likelihood of rubbing off or wear indications in the event of abrasion or other stress, and reduced half-dyeing time increases fabric abrasion resistance and breaking force [22]. Improved resistance against rubbing and friction forces is facilitated by the stronger dye-textile interaction made possible by the higher bio-mordant percentage

Table 9. Effect of mordant Variation on Colour fastness to Rubbing

Mordant variation (%)	Dry	Wet
10	4/5	4
20	4	5
30	3	5
40	3	3/4
50	3/4	3

Table 10. Effect of Dye variation (Shade percentage) on Colour fastness to Rubbing

Dye variation (Shade Percentage)	Dry	Wet
30	4/5	4
60	4	4
90	4/5	4
120	5	3/4
150	5	2

Table 11. Effect of temperature variation (°C) on colour Fastness to Rubbing

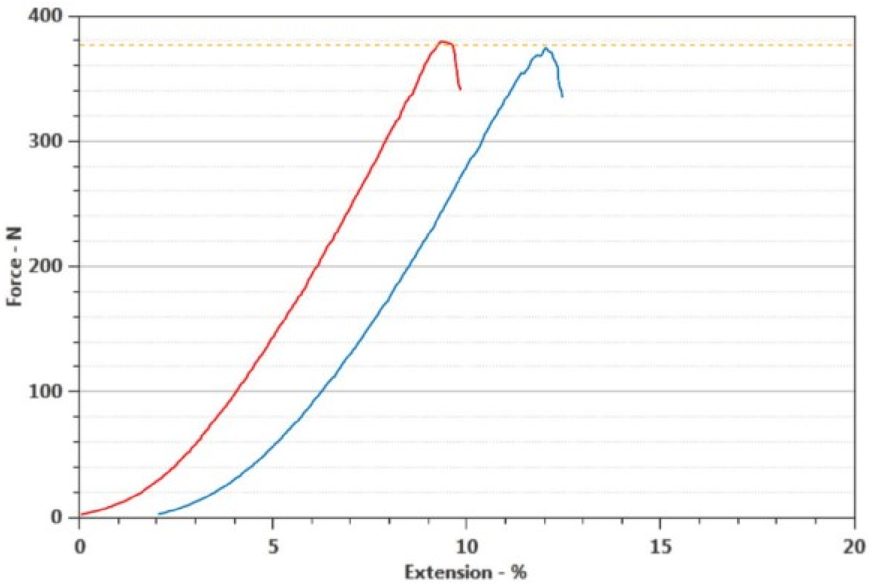
Temperature variation (°C)	Dry	Wet
60	4	3
90	4/5	3
100	5	3/4
120	5	4

Measurement of Tensile Strength

Following the thyme dyeing process, the fabric's tensile properties displayed minor variations. The tensile strength was operated at 24 °C, and humidity control of 66% RH. In Table 12, the dyed fabric demonstrated a slightly lower maximum force to break, measuring 373.98 N while also exhibiting a marginally increased extension percentage of 10.03% and a slightly extended time to break of 0.23 s. These findings suggest that the thyme dyeing process had a limited impact on the fabric's tensile strength and mechanical properties, with the fabric's structural integrity primarily preserved. In the tensile strength test, the initial grey fabric, which contained sizing materials and impurities, exhibited a maximum force to break of 243.14 N, (Table 13) an extension percentage of 13.38%, and a time to break of 0.19 s. On the other hand, the dyed fabric displayed a notable increase in maximum force to break, reaching 265.85 N, indicating improved tensile strength. The extension percentage rose to 18.54%, indicating enhanced fabric extensibility; also, time to break experienced only a slight increase to 0.21 s. These results suggest that the thyme dyeing process positively impacted the fabric's tensile characteristics. When pretreatment chemicals and dyestuffs are applied to cotton textiles, there is a notable reduction in both fabric tear resistance and tensile strength [22]. However, it is crucial to consider that the initial mechanical properties of the grey fabric may have been compromised by impurities, potentially contributing to the observed improvements (Graph 8 and Graph 9).

Table 12. Effect on tensile strength of the fabric in warp direction

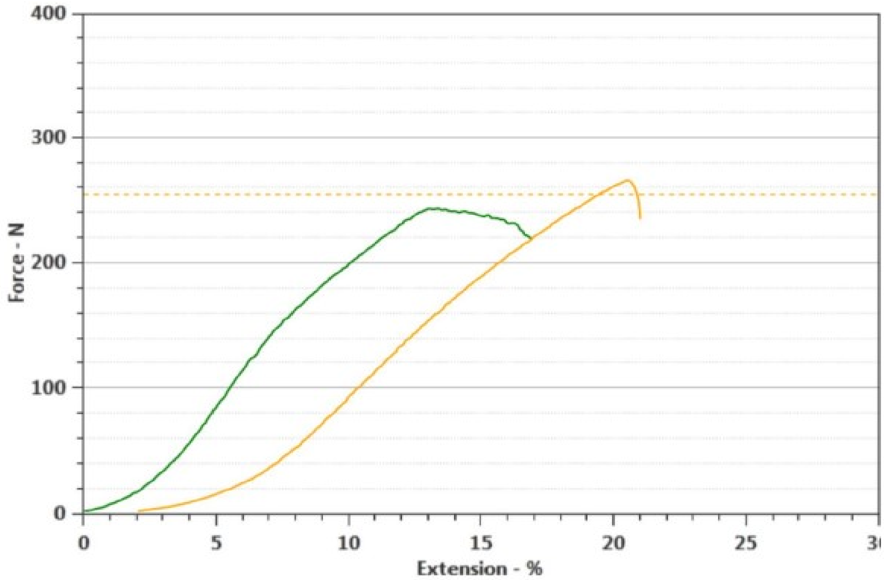
Specimen	Max Force (N)	Extension (%)	Time to break
Grey	379.43	9.35	00:22
Dyed Fabric	373.98	10.03	00:23
Mean	276.71	9.69	00:22
Standard Deviation	3.85	0.4796	00:00
Coefficient of variation	1.02%	4.95%	4.34%



Graph 8. Graphical representation of tensile strength of the fabric in warp direction

Table 13. Effect on tensile strength of the fabric in weft direction

Specimen	Max Force (N)	Extension (%)	Time to break
Grey	243.14	13.38	00:19
Dyed Fabric	265.85	18.54	00:21
Mean	254.50	15.96	00:20
Standard Deviation	16.06	3.65	00:01
Coefficient of variation	6.31%	22.86%	8.32%



Graph 9. Graphical representation of tensile strength of the fabric in weft direction

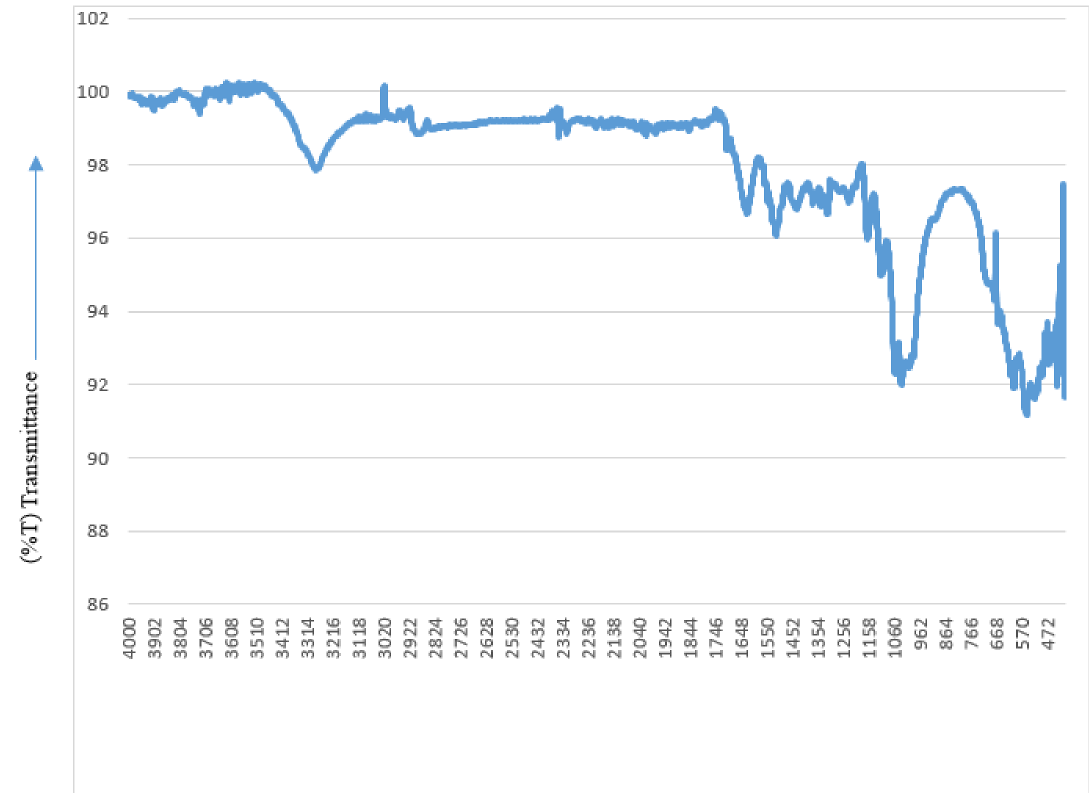
Scanning Electron Microscope Image Analysis

The microstructure of the cotton-silk-coloured fabric is displayed in the Scanning Electron Microscopy (SEM) images in Figure 2 (500x and 2000x). The coloured samples' dye-fibre adhesion and dispersion can be evaluated using SEM images. These details were needed to evaluate the dying process's effectiveness and colour fastness. SEM scans showed that the dye dispersion on the cationized fibre surface was uniform. The utilization of a cationic chemical to generate positive charges on the fibre surface was the root cause since it amplified the allure of negatively charged cotton fibres. The cationized coloured samples exhibited superior adhesion, uniform dye dispersion, and no dye anomalies, as demonstrated by the SEM pictures.

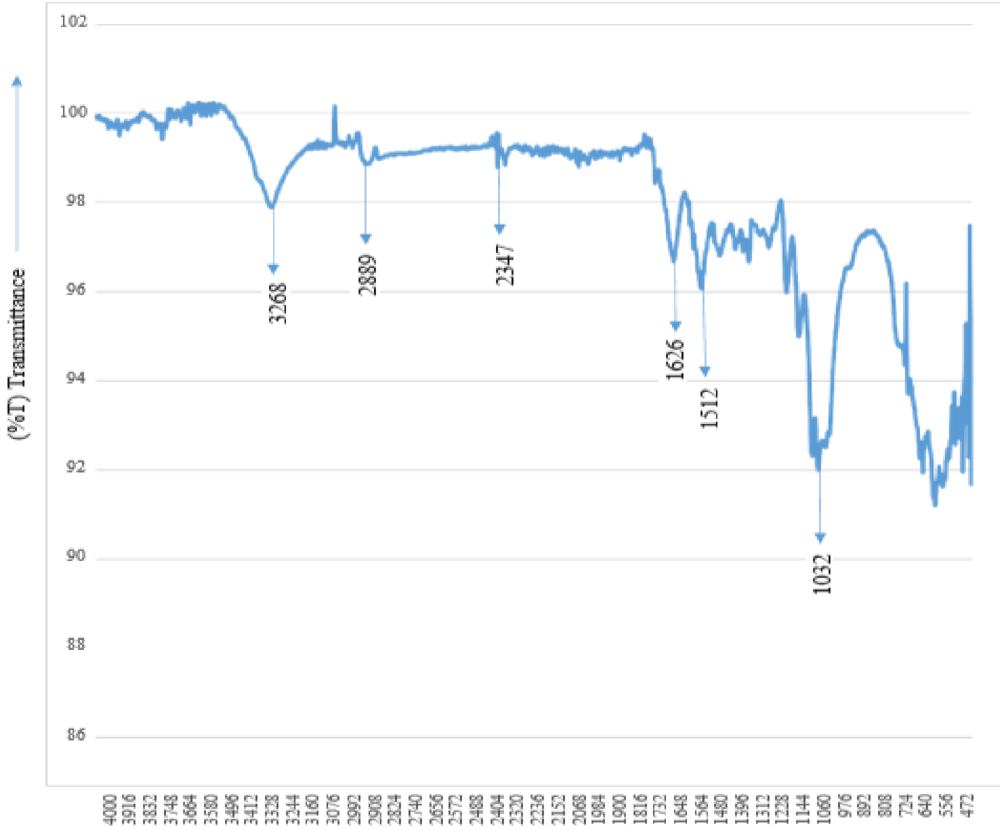
Figure 2. Scanning Electron Microscope Images (500x and 2000x) of cotton-silk blended fabric dyed with thyme extract

FTIR analysis through phytochemical of Thyme

Graph 10. displayed the infrared spectrum of Thyme extract-treated cloth, featuring % transmittance on the vertical axis and wave number on the horizontal axis. This spectrum typically spans from 4000 cm^{-1} to 600 cm^{-1}, encompassing the functional group region (4000 cm^{-1} to 2000 cm^{-1}) and the fingerprint region (2000 cm^{-1} to 600 cm^{-1}). Several notable absorption bands were identified: at 3268 cm^{-1}, N-H stretching bands, indicating the presence of amino acids; at 2889 cm^{-1}, C-H stretching and bending vibrations, suggestive of tannins; a distinct peak at 2347 cm^{-1} confirmed the presence of CO_2; peaks at 1626 cm^{-1} were linked to C=O and C=C carbon backbone stretches, corresponding to terpenoid and phenol compounds; additionally, peaks at 1512 cm^{-1} and 1032 cm^{-1} indicated C=C vibrations and C-O stretching/bending vibrations, respectively, implying the presence of carboxyl groups, commonly found in carbohydrates. The FTIR analysis provides compelling evidence for various phytochemicals from Thyme extract in the cloth sample, encompassing amino acids, tannins, carbon dioxide, terpenoids, phenols, and carbohydrates.



Graph 10. FTIR spectra of raw cotton-silk blended fabric (Grey fabric)



Graph 11. FTIR spectra of thyme-dyed cotton-silk blended fabric

So far, it has been observed that the dye uptake of silk is found to be comparatively better than that of cotton, although in a previous study by Yasukawa et al. at Hirosaki University in Japan, fuji apple peel dyeing of a silk-cotton blend was performed but no natural mordant was used [14]. In that sense, the result is much better. But the catch is that in Yasukawa's study, the effect of using metallic mordants provided much deeper dye penetration, which shows their values: L*: 67.07 ± 0.43, a*: 15.54 ± 0.60, b*: 3.37 ± 0.52, but in this study, the highest penetration was achieved at 150% tannin mordant, which denotes significantly high consumption of mordant. Whereas Yasukawa's study K/S ranged from 0.5 h to 4 h, this study shows significantly higher values, which ranged on average from 5 to 10. Finally, in the colour fastness test, the performance of both mordant and dye wash was quite adequate, which proves the colour strength of the dye was acceptable. Wash fastness received ratings of 4-5, signifying performance midway between very good to excellent. Staining on all three naturally dyed samples demonstrated good fastness with a consistent grade of 5 [14].

CONCLUSION

According to the study's findings, sustainable textile dyeing has advanced significantly. It has been shown that silk-cotton fabric mixes may be made more vibrant and environmentally friendly by using thyme as a natural dye source and cutting-edge methods including cationization of cotton fibres and bio-mordanting with tannin. Apart from dyeing with a copper mordant, all other dyeing processes including bio-mordanting and dye extraction meet the criteria outlined in the NODS [25]. The study revealed that tannin-based bio-mordanting enhances colourfastness, thereby promoting eco-friendliness and addressing the growing demand for sustainable textile practices. The dyeing process affects silk and cotton fibres differently, with thyme's innovative use as a colourant having a more significant effect on silk. Also, it has been observed minutely from the parameter-based data tables that at 120 °C and 150% mordant, both dye penetration and retention are quite excellent, which is both found from K/S analysis tests and post-washing colour fastness tests. The study underscores the potential of natural dyeing, bio-mordanting, and innovative textile techniques in achieving sustainable, high-quality, and customizable fabric outcomes yielding excellent K/S values and demonstrating robust colour fastness, suggesting a potential paradigm shift towards eco-friendly dyeing practices. This innovative approach not only addresses environmental concerns but also opens doors for commercially viable, sustainable textile production. As consumers increasingly prioritize eco-conscious choices, the positive outcomes of this study may influence purchasing decisions, fostering a demand for textiles dyed using natural sources and bio-mordants. The goal of this approach is to inspire further research and adoption of environmentally friendly and aesthetically pleasing textile dyeing techniques, which will ultimately contribute to a more colourful and sustainable future for the fashion and textile industries. It also serves as a significant milestone on the path towards a more vibrant and sustainable textile industry, reflecting the growing global commitment to environmental responsibility and the preservation of natural resources.

Author Contributions

Conceptualization – Ghosh J; Methodology – Khan F, Noor T, Rupanty NS; Formal analysis – Ghosh J, Khan F, Noor T, Rupanty NS; Investigation – Ghosh J, Khan F Noor T, Rupanty NS; Resources – Ghosh J, Khan F, Noor T, Rupanty NS; Writing-original draft preparation – Ghosh J, Khan F, Noor T, Rupanty NS, Das SC; Writing-review and editing – Ghosh J, Khan F, Noor T, Rupanty NS, Das SC; Visualization – Ghosh J, Khan F, Noor T, Rupanty NS; Supervision – Ghosh J. All authors have read and agreed to the published version of the manuscript.

Conflicts of Interest

The authors declare no conflict of interest.

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Hazards

Research involving chemicals, procedures or equipment has no unusual hazards.

Human research subjects

The research was conducted in accordance with relevant guidelines and regulations for experiments involving humans or affirmation that informed consent was obtained from all human research subjects.

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