UV-Vis spectroscopical and colorimetric analysis of natural dyed woollen fabrics employing a combination of chemical and botanical mordants

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Abstract

The current study aims to evaluate the colorant fastness grades and color strength on woven fabrics that were dyed by botanical dye derived from Symplocos racemosa leaves. A variety of botanical and chemical mordants, such as lime extract + $CuSO_4$, lime extract + $K_2Cr_2O_7$, lime extract + $FeSO_4$ and lime extract + $SnCl_2$, were used in the experiments, with corresponding ratios of 1:1, 1:2 and 1:3. Here, the dyeing process was combined with a mordant technique such as simultaneous mordanting technique. This study also includes fastness testing of dyed materials. Under UV-Vis Spectroscopic investigation, variation in the absorbance values and a wide range of color shades were detected with varying mordant ratios and their combinations.

The lime extract + $K_2Cr_2O_7$ of bio- and chemical mordants showed the highest value of absorption. The fastness grades of the dyed fabrics to light, perspiration, wash and rub have also been evaluated and found to be fair to good. Use of natural dyes in large scale in the textile industries may benefit from this observation with this species as Symplocos racemosa.

Keywords: Botanical dye, woven woollen fabrics, *Symplocos racemosa*, bio- and chemical mordants, fastness and color strength.

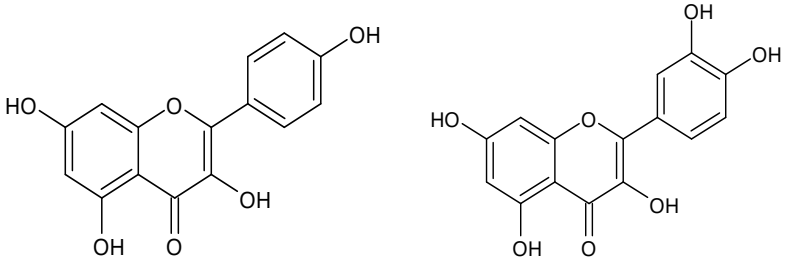
Introduction

The majority of people in the past relied on natural resources such as flowers, fruits, leaves, seeds, bark and plant roots to color textiles[1-3] and so on. Around the world, plants colorants have been used to dye fabrics such as wool, silk, cotton[5,8,10,11] and many types of biofibers. These colorants have been used to color textiles[7,9,12] because wool and other biofibers are used by different human societies world-wide and fastness grades vary depending on the weather and soil type. Their remarkable color diversity and environmentally friendly nature[6] create a non-polluted environment. Chemical and spectroscopic analysis were used to extract the color components that cause dyeing and to determine the chemical components that make them up.

The two types of mordants[18] that are commonly used, are botanical (such as an extract of leaves, flowers, stems and roots etc.) and chemical (such as metallic salts that can create chemistry between the fabrics and botanical dye). The locust species is currently producing botanical dyes with qualities[21,23,24] and it provides us with excellent color grades in relation to the grey scale. The aqueous extract is used in this study to try coloring woollen cloths at various stages of refined dyeing.

This is followed by the use of both chemical and botanical mordants and the fastness of dyed materials was assessed by washing, rubbing, perspiration and light exposure. Several natural resources are used in the industrial dyeing processes[25,26]. The non-recreatable nature of botanical or natural dyes[27,28], their multiform hues, mild color grade and the lack of adequate scientific information on the chemistry for coloring with appropriate finishing or dyeing conditions are, however, are some of the limits.

A review of the literature on the application of natural dyes using chemical and biological mordants or their combinations[13-17] with varying color characteristics followed by simultaneous mordanting provided a wealth of information about the finishing of textiles with botanical dyes like quinones, flavonoids and anthocyanins. The out of pre-isolated dye bearing colorants, there are only two colorants such as quercetin and kaempferol from *Symplocos racemosa*[19,20] leaves are listed below (Figure 1).



Kaempferol **Quercetin**

Figure 1: Two major pre-isolated colorants from the leaves of *Symplocos racemosa*

Indian status of plants yielding dyes and environmentally beneficial natural dyes: The research states that the primary application of natural materials has been their adaptability in investigating applications such as textile finishing, medicine polishing, cosmetic products coloring, antibacterial and antimicrobial properties. Moreover, food goods are colored using plant dyes because they are non-toxic. According to published research, over 450 different plants can yield dyes in India[22]. Sadly, there are insufficient data resources available to determine whether natural dyes can be used for extraction and dyeing processes and there is also a lack of awareness in the public about the advantages of using natural dyes over synthetic ones.

Material and Methods

Symplocos racemosa is commonly known as Lodhra or Lodh which is a member of family Symplocaceae and can be found in a variety of locations in the Garhwal region of Uttarakhand at an elevation of 1500-2000 meters. In this study, bleached plain weaved woollen fabrics were acquired from the Gopeshwar, District Chamoli of Uttarakhand and the leaves of this plant were used as plant material for dyeing. This process was followed by aqueous extraction. Chemical reagents as mordants such as $K_2Cr_2O_7$, $CuSO_4$, $SnCl_2$ and $FeSO_4$ (Analytical reagents grade) and certain laboratory reagents like sodium carbonate, acetic acid and common salt were also utilized as the same and a bio-mordant as 'lemon extract' was used for this investigation.

Aqueous botanical dye extracted from *Symplocos racemosa* leaves was used to dye fabrics. The color shades of the dyed fabrics were affected by the lime extract when it was mixed with a standard volume of distilled water and heated at 80 ^oC for 30 minutes. This was done in the order to simulate mordanting. The solution was then cooled and filtered and the filtrate gave rise to the appropriate mordant condition. Now, take a known amount of shade dried *Symplocos racemosa* leaves, soak them in lukewarm water for the entire night and then boil the extract with distilled water, cool, filter and lastly dye it to weaved woollen fabrics.

Optimal conditions were used for the dyeing of fabrics including 60 minutes dye extraction period, a 1:20 M-L ratio and 50 minutes dyeing time. The following combinations of chemical and botanical mordants were utilized in the following ratios such as 1:1, 1:2 and 1:3 respectively: lime extract + $CuSO_4$, lime extract + $K_2Cr_2O_7$, lime extract + $FeSO_4$ and lime extract + $SnCl_2$. Each two mordant combination's total quantity was 5% of the woollen fabric's weight or 5 g of mordant of every 100 g of fabric. These four combinations of mordants were subjected to a variety of condition including simultaneous mordanting for dyeing. Therefore, after the fabrics were dyed, the solution was cooled and the absorbance was measured using UV-Vis spectrophotometer at a specific wavelength of 500 nm.

A Shirley-MBTF Microsal Fade-O-meter (ISO: 2454-1984) was used to test the fastness grades towards light and the blue wool standards (BS1006: BOI: 1978) were used to compare the fade nature of the dyed materials: Sasmira Launder-O-meter IS-3 for wash fastness followed with ISO: 105 A03 (% of staining) and ISO: 05 A02 (loss of shade depth). The Macbeth 2020 plus computer-ACM system was used with AATCC manually operated Crock meter and a grey scale followed with ISO: 105 A03 (% of staining) as per IS:766-1984 technique used to test the rub fastness.

The M:L ratio was used to measure the perspiration fastness grades in the acidic and alkaline media for 30 minutes at 25 ^oC using a perspirometer that was loaded with 4.5 kg (10 lbs). Finally, the samples were removed and allowed to dry in the air at a temperature no higher than 60 ^oC. Grey scale was then used to stain and alter the color of the samples.

Features of the dyed fabric colors: Using four combinations of chemical and botanical mordants simultaneously, the CIEL*a*b* values determined the various parameters including hue saturation (h^o), lightness (L*), redness-greenness value (a*), yellowness-blueness value (b*) and chromaticity (c*).

Strength of color: The Kubelka-Munk equation (equation 1) was utilized to calculate the *K/S* value of the dyed woollen fabrics. The surface reflectance of the samples was measured using a computer-aided Macbeth 2020 plus reflectance spectrophotometer:

$$\text{Color strength } (K/S) = (1-R_{\lambda max})^2/2\, R_{\lambda max} \quad (1)$$

where $R_{\lambda max}$ is the sample's surface reflectance value at a specific wavelength where maximum absorption for given dye/color components occurs, S is the scattering co-efficient and K is the absorption co-efficient.

The interaction between natural dyes and fabrics, particularly the role of mordants: The chemistry used to dye the various fabrics is primarily dependent on the nature of the dye components and how well it bonds with the functional groups present in the fabric to form the complex that gives the fabric its color.

However, if mordanting is necessary to change the color of the fabric to a different shade, three conditions must be met: pre-mordanting, simultaneous mordanting and post mordanting. In this scenario, if the pre-mordanting condition are met, the mordant, whether natural or metallic, will react with the fabric first, forming a metallic complex with them.

Dyeing will take place in the presence of a dye component that was separated from the dye bearing plant or other natural resources, resulting in the formation of another complex between the fabrics, mordant and dye component. A reactive scheme for above mechanism is given in figure 2. For instance, if wool is fabric, mordant will be a metallic salt and quercetin will be a dye component.

Results and Discussion

Mordant Combination - Lime extract + Stannous Chloride: In this case, the assessed fastness to light remained constant (4) for two ratios of mordant combinations such as 1:1 and 1:3 except 1:2 ratio of mordant combination (4-5), although observed fastness during washing under concurrent mordanting ranged from good to excellent (4-5) in the change color whereas excellent (5) during color staining. Almost excellent (5) color change was seen for both wet and dry rubbing fastness conditions. For perspiration fastness grades, it was observed excellent (5) in color staining and good (4) in color change under both acidic and alkaline media (Table 1).

Mordant Combination - Lime extract + Copper Sulphate: In this case, the assessed fastness to light remained constant (4) for two ratios of mordant combinations such as 1:1 and 1:2 except 1:3 ratio of mordant combinations (4-5).

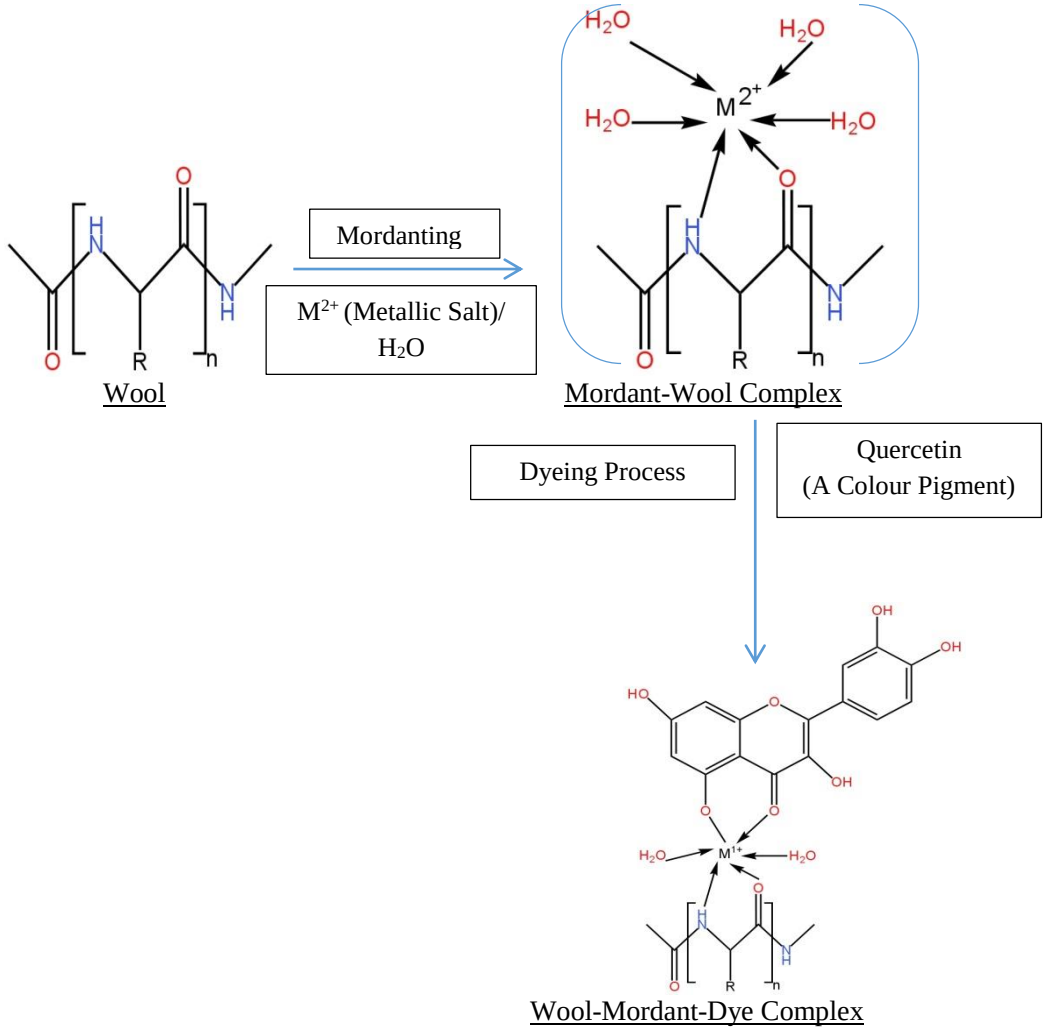


Figure 2: A general scheme for Mordanting and Dyeing processes with Wool

Table 1
Color fastness grades of dyed weaved woollen fabrics with *Symplocos racemosa* dye at some refined conditions using botanical and chemical mordant combination as LJ: SC under simultaneous mordanting condition

Mordanting method	Mordant proportions	Fastness grades to light	Fastness grades to washing		Fastness grades to rubbing			Fastness grades to Perspiration			
			CC	CS	Dry		Wet	Acidic		Alkaline	
					CC	CS	CC	CS	CC	CS	CC
Simultaneous Mordanting	1:1	4	4-5	5	5	5	5	5	4	5	4
	1:2	4-5	4-5	5	5	5	5	5	4	5	4
	1:3	4	4-5	5	5	5	5	5	4	5	4

LJ: SC – Lime extract: $SnCl_2$, CC –Change in color, CS – Staining of color.

On the other hand, fastness during washing under mordanting combinations ranged from good in the change color whereas almost excellent (5) during color staining except 1:3 ratio of mordant combination (4-5). Almost excellent (5) color change was observed for both wet and dry rubbing fastness conditions. For perspiration fastness grades, it was observed almost excellent (5) in color staining and almost fair (3) in color change under both acidic and alkaline media (Table 2).

Mordant Combination - Lime extract + Potassium Dichromate: Under this instance, fastness grades to light good to excellent (4-5) for two ratios of mordant combinations such as 1:1 and 1:3 except 1:2 ratio of mordant combination which was good (4). On the other hand, fastness during washing under 1:2 and 1:3 mordanting combinations was observed good (4) except 1:1 which was poor to fair (2-3) in the change color whereas excellent (5) during color staining for all mordant combinations. Almost excellent (5) color change was observed for wet and dry rubbing fastness during color staining condition and good to excellent (4-5) for two ratios of mordant combinations such as 1:2 and 1:3 except, 1:1 ratio of mordant combination which was an excellent (5) one. For perspiration fastness grades, it was observed that under acidic medium, almost good to excellent (4-5) in color staining, good in color change and excellent (5) in color staining and good (4) in color change under alkaline media (Table 3).

Mordant Combination - Lime extract + Ferrous Sulphate: In this case, the assessed fastness to light all ratios remained constant as good (4) for all mordant combinations, although observed fastness during washing under all mordant combinations ranged from fair to good (3-4) during color change and good to excellent (4-5) during color staining. Almost excellent (5) color change was seen for both wet and dry rubbing fastness conditions. For perspiration fastness grades, it was observed excellent (5) in color staining and good (4) in color change under both acidic and alkaline media (Table 4).

The environmental friendly botanical dye that was aqueously extracted from *Symplocos racemosa* leaves had a brown hue. Table 5 indicates that when we extracted botanical dye from this species without mordant, we saw less beautiful color shade. However, when we used different conditions with some chemical mordants such as $K_2Cr_2O_7$, $CuSO_4$, $SnCl_2$ and $FeSO_4$, we observed wonderful shades of colors.

UV-Vis Spectroscopical analysis: Using a UV-Vis Spectrophotometer, the absorbance values of dyeing solutions were analysed at a wavelength of 500 nm. The mordant combination of lime extract + $K_2Cr_2O_7$, produced the best result with a value of 2.98; the other three combinations are shown in the figures 3-6 and table 6, producing observed values of 1.1274, 1.5044 and 0.7485 for lime extract + $SnCl_2$, lime extract + $FeSO_4$ and lime extract + $CuSO_4$ respectively.

Table 2
Color fastness grades of dyed weaved woollen fabrics with *Symplocos racemosa* dye at some refined conditions using botanical and chemical mordant combination as LJ: CS under simultaneous mordanting condition

Mordanting method	Mordant proportions	Fastness grades to light	Fastness grades to washing		Fastness grades to rubbing			Fastness grades to Perspiration			
					Dry		Wet	Acidic		Alkaline	
			CC	CS	CC	CS	CC	CS	CC	CS	CC
Simultaneous Mordanting	1:1	4	4	5	5	5	5	5	3	5	4
	1:2	4	4	5	5	4-5	5	4-5	3	5	3
	1:3	4-5	4	4-5	5	5	5	5	4	5	3

LJ: CS – Lime extract: $CuSO_4$, CC –Change in color, CS – Staining of color

Table 3
Color fastness grades of dyed weaved woollen fabrics with *Symplocos racemosa* dye at some refined conditions using botanical and chemical mordant combination as LJ: PD under simultaneous mordanting condition

Mordanting method	Mordant proportions	Fastness grades to light	Fastness grades to washing		Fastness grades to rubbing			Fastness grades to Perspiration			
					Dry		Wet	Acidic		Alkaline	
			CC	CS	CC	CS	CC	CS	CC	CS	CC
Simultaneous Mordanting	1:1	4-5	2-3	5	5	5	5	4-5	4	5	3-4
	1:2	4	4	5	4-5	5	5	5	4	5	4
	1:3	4-5	4	5	4-5	5	5	4-5	4	5	4

LJ: PD – Lime extract: $K_2Cr_2O_7$, CC –Change in color, CS – Staining of color

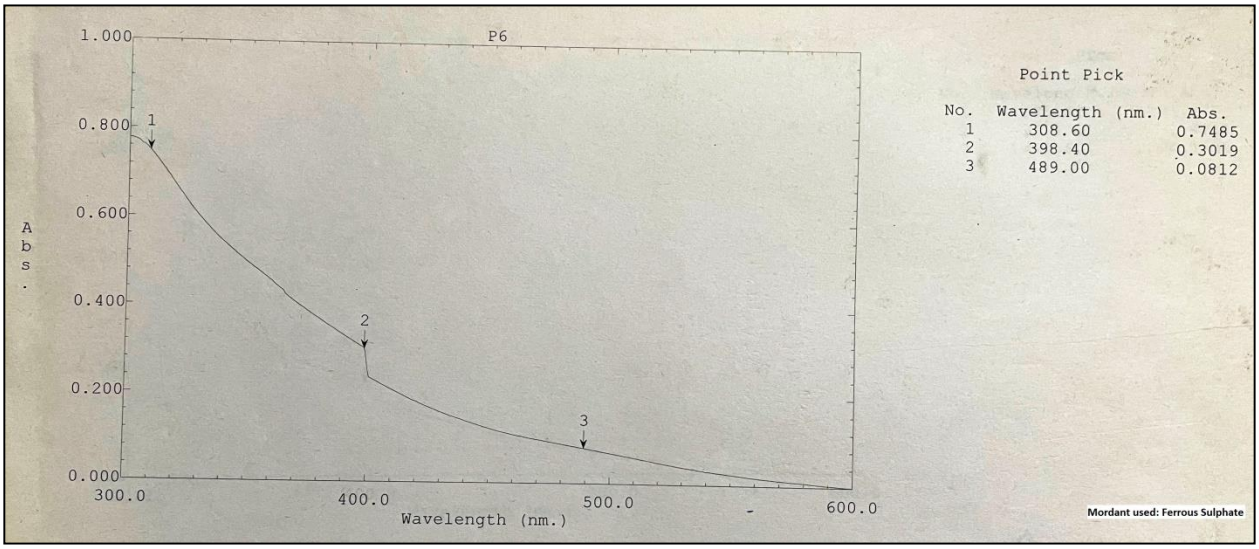


Figure 3: UV-Vis Spectra of the extracted dye for dyeing using combination of mordants as Lime extract and Ferrous Sulphate under simultaneous condition

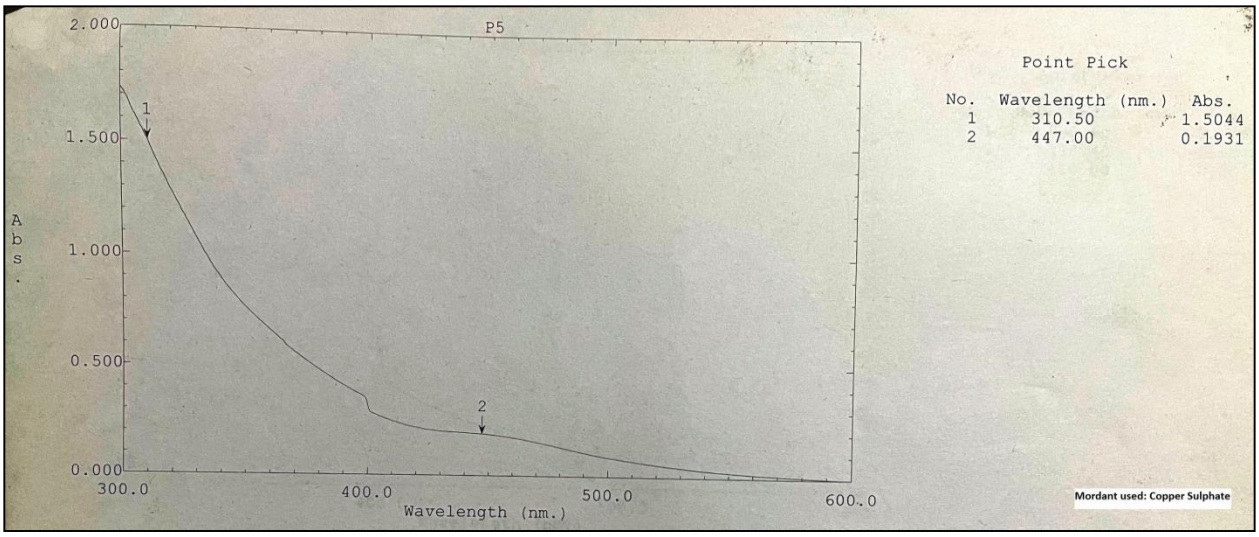


Figure 4: UV-Vis Spectra of the extracted dye for dyeing using combination of mordants as Lime extract and Copper Sulphate under simultaneous condition

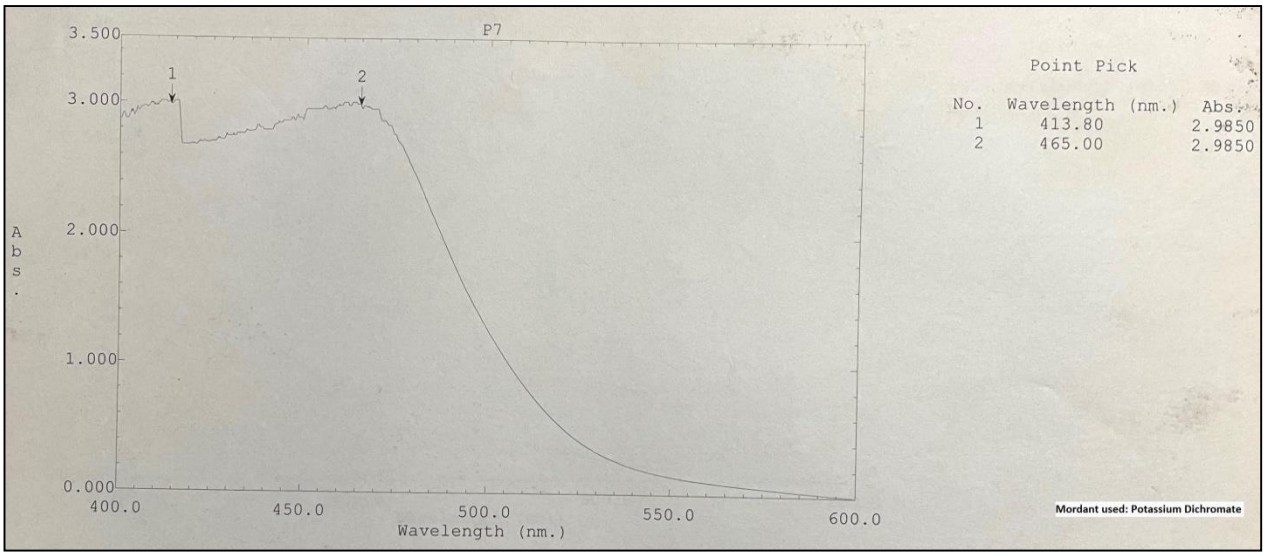


Figure 5: UV-Vis Spectra of the extracted dye for dyeing using combination of mordants as Lime extract and Potassium Dichromate under simultaneous condition

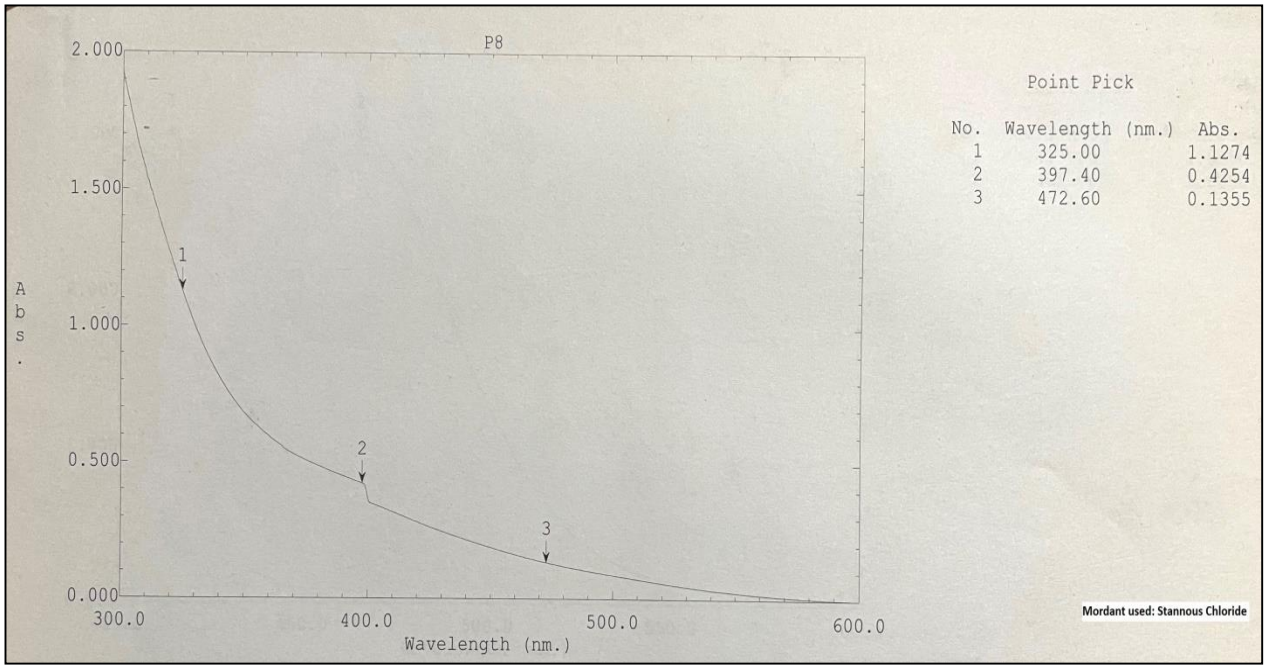


Figure 6: UV-Vis Spectra of the extracted dye for dyeing using combination of mordants as Lime extract and Stannous Chloride under simultaneous condition

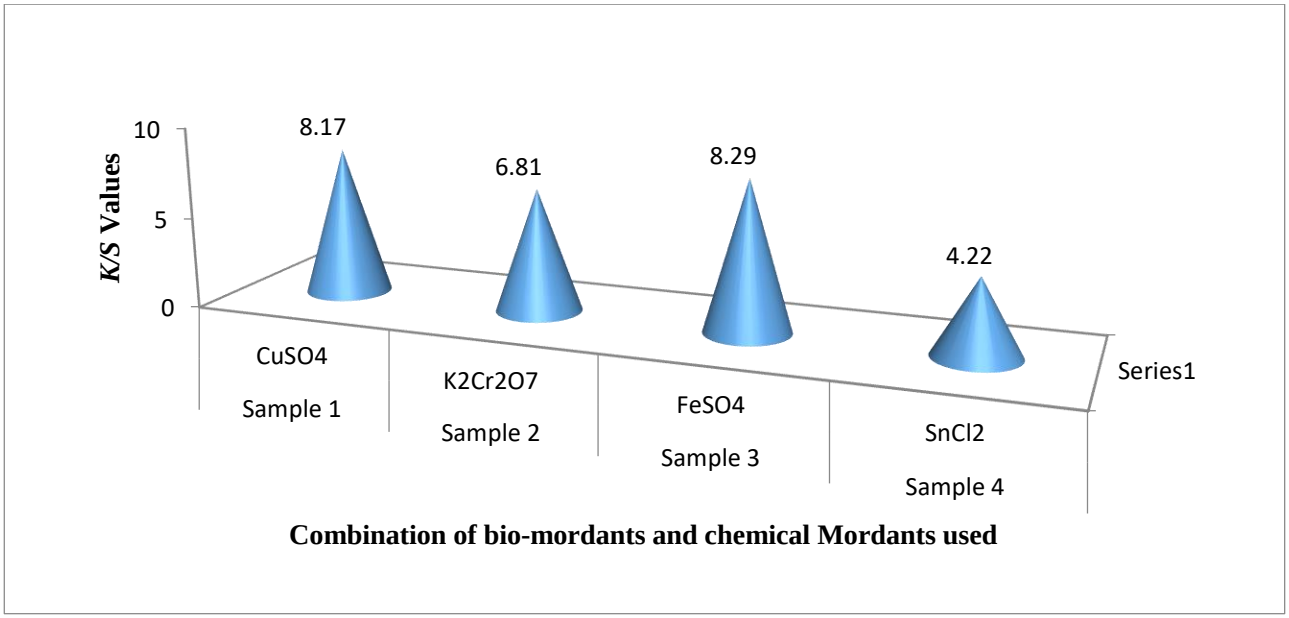


Figure 7: K/S values of different dyed wool samples using selected combination of bio mordants and chemical mordants

Table 4
Color fastness grades of dyed weaved woollen fabrics with *Symplocos racemosa* dye at some refined conditions using botanical and chemical mordant combination as LJ: FS under simultaneous mordanting condition

Mordanting method	Mordant proportions	Fastness grades to light	Fastness grades to washing		Fastness grades to rubbing			Fastness grades to Perspiration			
					Dry		Wet	Acidic		Alkaline	
			CC	CS	CC	CS	CC	CS	CC	CS	CC
Simultaneous Mordanting	1:1	4	3-4	4-5	5	4-5	5	4-5	5	5	5
	1:2	4	3-4	4-5	5	5	5	5	5	5	5
	1:3	4	3-4	4-5	5	5	4-5	5	5	5	4-5

LJ: FS – Lime extract: $FeSO_4$, CC –Change in color, CS – Staining of color

Table 5
Observed color shades of dyed weaved woollen fabrics with dye from the leaves of *Symplocos racemosa* using combination of chemical and bio-mordants
(With and without mordant dyeing)

Mordant condition Used	Dyeing (without mordants)	Dyeing with the combination of chemical and bio-mordants (lime extract)
Simultaneous Mordanting		
		
		
		

Table 6
UV-Vis-Spectral analysis (values of absorbance) of the extracted dye for dyeing using four combinations of bio and chemical mordants under simultaneous condition

S. N.	Observed wavelength about 500 nm	Observed Absorbance	Combination of mordants used	Observed shades of color of dyed fabrics
1.	325 nm	1.1274	LE+SC	Light Yellow
	397.40 nm	0.4254		
	472.60 nm	0.1355		
2.	310.50 nm	1.5044	LE+CS	Light Green
	447 nm	0.1931		
3.	413.80 nm	2.98	LE+PD	Pale Yellow
	465 nm			
4.	308.60 nm	0.7485	LE+FS	Light Brown
	398.40 nm	0.3019		
	489 nm	0.0812		

LE+SC: Lime Extract + Stannous Chloride, LE+CS: Lime Extract + Copper Sulphate, LE+PD: Lime Extract + Potassium Dichromate and LE+FS: Lime Extract + Ferrous Sulphate

Table 7
CIEL*a*b* values using four combinations of bio and chemical mordants under simultaneous condition

Sample	Mordant Combinations	*L**	*a**	*b**	*C**	h^0
1	LE+SC	55.93	7.63	24.65	25.81	72.80
2	LE+CS	58.96	-7.28	21.30	22.51	108.86
3	LE+PD	57.32	6.32	31.59	32.21	78.72
4	LE+FS	70.93	2.18	26.51	26.60	85.31

LE+SC: Lime Extract + Stannous Chloride, LE+CS: Lime Extract + Copper Sulphate, LE+PD: Lime Extract + Potassium Dichromate and LE+FS: Lime Extract + Ferrous Sulphate

Analysis of color characteristics and color strength: Table 7 illustrates the effects of combining botanical and chemical mordants on wool fabrics with the variation in the values for colorimetric parameters (*L**, *a**, *b**, *c** and h^o). Of the remaining combinations, LE+CS and LE+FS gave the best results in the term of lightness and hue saturation, coming in at 58.96 and 70.93 and 108.86 and 85.31 respectively while the two combinations of LE+SC, 7.63, 24.65 and 25.81 respectively showed good results for the values of redness-greenness value (a*), yellowness-blueness value (b*) and chromaticity (c*).

However, the best results for yellowness-blueness value (b*) and chromaticity (c*) in the LE+PD combination were found to be 31.59 and 32.21. A graphical figure 7 revealed the color strength (K/S) of the dyed woollen fabrics treated with mordants and the color strength values vary when different mordants combinations were used. For instance, the combination of LE+FS and LE+CS revealed the best results among the other combinations with the values 8.29 and 8.11 respectively.

Conclusion

It was concluded that the *Symplocos racemosa* plant provides the best dyeing properties on woollen fabrics such as light fastness which was observed almost good to excellent for all the cases. Washing fastness grades were observed as an excellent in color staining and good to excellent for LE+SC than rest whereas almost excellent (5) color change was seen for both wet and dry rubbing fastness conditions for all the cases. For perspirations, fastness grades were almost excellent (5) in color staining and good (4) in color change in both acidic and alkaline media for all the combinations under simultaneous mordanting treatment.

The highest value of absorbance was observed for LE+PD combination of botanical and chemical mordants under UV-Vis spectral analysis. Out of all the combinations, combination of LE+CS and LE+FS produced best results in the term of lightness and hue saturation and highest or best value of b* and c* observed in the case of the combination LE+PD while LE+FS mordants combination has the highest value of color strength of K/S value respectively.

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