

Investigation of Color, and Fastness Properties of Wool Yarn Dyed with Ternary Plant Combination: A New Dyeing Method for the Production of Eco-Friendly Textiles

Menderes KOYUNCU*¹ ORCID 0000-0002-4900-9327

¹Department of Textile, Van Vocational School, Van Yuzuncu Yil University, Van

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Abstract

Lately, the utilization of natural colorants in textile dyeing and printing has come to the forefront of the manufacturing of textile goods due to increased environmental troubles. In this study, wool yarns were dyed with ternary dye combinations (Scutellaria Orientalis L, Rubia Tinctorium L, and Punica Granatum L). The color coordinates and washing and light fastness were investigated. Washing fastness and light fastness are fair to good. Dyed wool yarns were worked calorimetrically and their color coordinates CIELAB (L*, a*, and b*) and color strength (K/S), and ΔE^* values were given. Fourier transform Infrared Spectrophotometer (FTIR), and Scanning Electron Microscopy (SEM) morphological studies have also been carried out on dyed and undyed yarns. From the results, it could be said that the natural dye extracted from ternary dye combinations plants has good potential in wool yarns dyeing and can be exploited further.

Keywords: Natural dyeing, Wool yarn, Coloring compound, Fastness, FTIR, SEM

Çevre Dostu Tekstil Üretimi için Yeni bir Boyama Yöntemi olarak Üçlü Boya Kombinasyonu ile Boyanmış Yün İpliklerinin Renk ve Hask Özelliklerinin Araştırılması

Öz

Son zamanlarda artan çevre sorunları nedeniyle tekstil boyama ve baskıda doğal renklendiricilerin kullanımı tekstil ürünleri imalatında ön plana çıkmıştır. Bu çalışmada, yün iplikleri üçlü boya (Scutellaria Orientalis L, Rubia Tinctorium L, and Punica Granatum L) kombinasyonları ile boyama işlemi yapılmıştır. Boyanmış ipliklerin, yıkama ve ışık haslıkları yapılarak orta - iyi ve iyi değerlerde olduğu görülmüştür. CIELAB (L*, a*, and b*) renk koordinatları, and K/S, ve ΔE^* değerleri incelenmiştir. Ayrıca FTIR ve SEM analizleri yapılarak boyamadan sonra değişimler incelenmiştir. Sonuçlardan, üçlü

*Corresponding author (Sorumlu yazar): Menderes KOYUNCU, menderes@yyu.edu.tr

boya kombinasyonlu bitkilerden ekstrakte edilen doğal boyanın, yün iplikleri için iyi bir potansiyele sahip olduğu ve daha fazla yararlanılabileceği söylenebilir.

Anahtar Kelimeler: Doğal boyama, Yün ipliği, Renk bileşimi, Haslık, FTIR, SEM

1. INTRODUCTION

Rubia tinctorium L. is manufactured from rhizomes of *Rubia tinctorium*. It is known as Turkish red. It has been used for hundreds of years to color carpet and kilims yarns. Rhizomes from three- or four-year-old plants are collected for dyeing. It gives different colors with different mordants. Such as, when CaCO_3 is used, the color becomes brick red. $\text{K}_2\text{Cr}_2\text{O}_7$, the color dark orange; CuSO_4 , the color light brown, etc. [1].

It is also known by different local names such as *Punica granatum* anar, and pomegranate. It belongs to the family of *Punicaceae*. *Punica granatum* is commonly found in the Mediterranean basin and Southern Asia in warm, temperate climates. The parts that give dyestuff are the bark and flowers [2]. *Scutellaria orientalis* L is a type of flowering plants belonging to the *Lamiaceae* family. They are known commonly as skullcaps and include about 350–400 species. The main flavonoids of this kind are baicalein, wogonin, chrysin [3].

Wool has historically played an important role in various human cultures due to its unique properties. The behavior of wool differs from one fiber to another because each wool fiber has its own structure; diameter crystalline microfibrils (degree of crystallinity) and amino acids that directly affect their behavior [4]. Protein-based materials such as wool and cashmere are highly valuable renewable natural resources. The total world consumption of products made from such materials has increased steadily over the past fifty years [5]. Natural dyes are known for their use in the coloring of food dyes, leather, and textiles as well as natural protein fibers like wool, silk, mohair, etc. as major areas of application since prehistoric times. The use of natural dyes with poor to moderate wash and light fastness properties has declined drastically since the industrial revolution in the 19th century introduced

widely available and less expensive synthetic dyes with moderate to excellent color fastness properties [6]. However, there has recently been a renewed interest in the application of natural dyes on natural fibers due to worldwide environmental factors. Today, natural dyes have little or no economic importance and are used in limited quantities by craftsmen. However, due to the increasing interest of consumers in environmentally friendly clothing, the use of natural dyes has been reconsidered [7]. Natural dyeing of different and textile materials have been continued mainly in the decentralized sector for specialty products long with the use of synthetic dyes in the large scale sector for general textiles-garments owing to the specific advantages and limitations of natural dyes and synthetic dyes. Although synthetic dyes have performed better recently, the use of natural dyes on textile materials is attracting more scientists to research this issue due to the non-allergic, non-toxic and environmentally friendly properties of natural dyes. In dyeing studies of textile products with natural dyestuffs (Seeds, leaves, flowers, and bark etc.) and using different analytical devices, and different mordants, and, ternary combinations, etc. with high fastness values are carried out [8]. Gharari et al. Studied the simultaneous determination of baicalein, chrysin, and wogonin in four Iranian *Scutellaria* species by high-performance liquid chromatography [9-10]. Different mordants and other complexing agents, mordanting kinds, and dyeing methods have been extensively studied in the literature. [11-13]. However, not much has been reported in the literature about the dyeing of wool yarns with three different plant solution. Therefore, to gain this dyeing method understanding, in the present work, the dyes have been extracted from *Rubia Tinctorium* L. and *Scutellaria Orientalis* L and *Punica Granatum* L. and the application of the extracted dye on wool yarns without mordants. The effect of three different plant solutions mixed

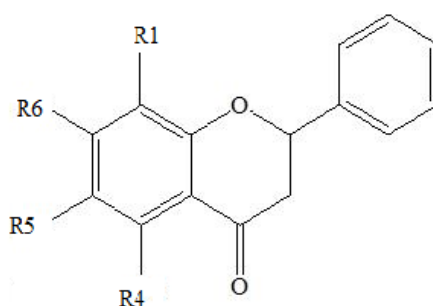
on dyeing quality was also examined, and the color properties of dyed wool yarns have been assessed.

2. METHOD

2.1. Wool Yarns and the Natural Dyes

In the study, 100% wool with a weight of 1.0 g, and 2.5 Nm strayhgarn yarns were used for experiments. Yarns were also obtained from wool

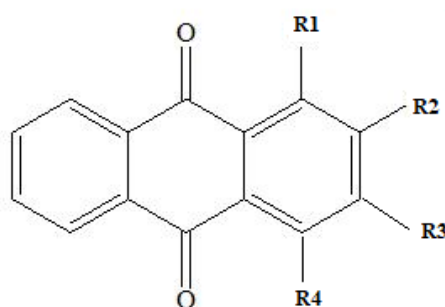
yarn factories in Turkey. Natural plants were collected in the season in Van-Turkey. Plants collected were dried under shady and airy conditions and the flower of *Scutellaria orientalist L.*, roots of *Rubia Tinctorium L.*, and bark of *Punica Granatum L.* was ground to powder form using an electrically operated grinder. The molecular structures and molecular weight of the three dyes are shown in Figures 1, 2, and 3.



Color compounds in *Scutellaria orientalist L.*

Compounds	R ₁	R ₂	R ₃	R ₄	R ₅	R ₆	The molecular formula, and Mol.Wt.
Baicalein	-	-	-	OH	OH	OH	C ₁₅ H ₁₂ O ₅ ,272,25
Wogonin	CH ₃	-	-	OH	-	OH	C ₁₆ H ₁₄ O ₄ ,270,28
Chrysin	-	-	-	OH	-	OH	C ₁₅ H ₁₂ O ₄ ,256,25

Figure 1. Chemical structure of *Scutellaria Orientalist L.* [3]



Color compounds in *Rubia Tinctorium L.*

Compounds	R ₁	R ₂	R ₃	R ₄	The molecular formula, and Mol.Wt
Alizarin	OH	OH	-	-	C ₁₄ H ₈ O ₄ ,240,21
Purpurin	OH	OH	H	OH	C ₁₄ H ₈ O ₅ ,256,21
Pseudopurpurin	OH	COOH	OH	OH	C ₁₅ H ₈ O ₇ ,300,22
Rubiadin	OH	CH ₃	OH	H	C ₁₅ H ₁₀ O ₄ ,254,24
Munjistin	H	OH	COOH	OH	C ₁₅ H ₈ O ₆ , 284,22

Figure 2. Chemical structure of the *Rubia Tinctorium L.* [1]

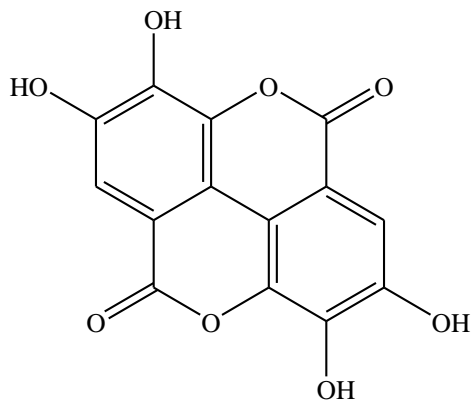


Figure 3. Chemical structure of Punica Granatum L. [2]

The molecular formula is $C_{14}H_6O_8$ and molecular weight is Mol.Wt.: 301,01 m/e: 302,01 (100,0%), 303,01 (15,5%), 304,01 (2,8%)

2.2. Dye Extraction

The extraction temperature is an important element acting on extraction efficiency. Nevertheless, high temperatures can cause various chemical structure degradations or undesirable components may be in the extraction.

As a general rule, the extraction temperature range varies to a large extent, from 50 to 220 °C, depending on the extraction technique [14-17]. In addition, small particle sizes are generally suitable for high extraction efficiency. However, the particle size is too small can result in the formation of agglomerates that reduce extraction, and also the solvent-feed ratio must be determined in the extraction. This study used the solid-liquid extraction method, and due to the evaporation of the solvent in the extraction process, the temperature was not increased above the boiling point of the solvent. About 1 g of dried plants (Punica Granatum L, Rubia Tinctorium L, and Scutellaria Orientalist L.) were weighed respectively, taken in the 250 ml beaker, and dissolved in 100 ml of solvent (H_2O). After heating the beaker at 70-80 °C for 1h, the extract was filtered, and stored and stored in liquid form after cooling at room temperature, and used for dyeing.

2.3. The Procedure of the Dyeing

The dyeing process was done in a laboratory type dyeing machine. (Atac YK-12). The wool yarns were dyed without mordants. The wool yarns were immersed in the dye solution in the dyeing machine and according to the dyeing prescription as given below. The procedure of the dyeing is shown in table 1. The colored samples were washed with distillate water and the samples were dried at room temperature. In this study, the pH value of the triple dye mixture was kept between 3-5 with acetic acid. In previous studies, it was found that low pH values increase dye uptake on textile materials. It is known that the alkaline solution, a reaction with hydroxide ions (OH^-) converts the ammonium ion (NH_3^+) to amino (NH_2) groups and the fiber contains more carboxylate ion (COO^-) Thus, electrostatic repulsion between the anionic colorants and the protein fibers occurs, which leads to a decrease in dye absorption [14-17]. In general, color uptake of natural dyes temperature increases between 60-90 °C. While the maximum dye uptake is in the temperature range of 60-80 °C for some natural dyes, it is at a temperature of 90 °C for some natural dyes. Similarly, Mongkhlorattanasit et al. [15]. Reported that the maximum hue obtained for natural dyes at 90 °C found to be suitable for wool dyeing. It causes less aggregation and more expansion in wool fiber at high temperatures. This allows dye molecules to penetrate the wool yarn.

Table 1. The process of the dyeing

Dyeing no	Dyeing prescription	Temperature	Time	pH Values
2	Rubiatic: 10 mL	90 ±2 °C	45 min	up to pH : 3-3.5
	Punica granatum: 70 mL			
	Scutellaria: 20 mL			
3	Rubia tinc: 25 mL	90 ±2 °C	45 min	up to pH: 4.5-5
	Punica granatum: 18 mL			
	Scutellaria: 50 mL			
4	Rubia tinc :12 mL	90 ±2 °C	45 min	up to pH: 5.0
	Punica granatum: 6 mL			
	Scutellaria: 75 mL			

2.4. Colour Measurement and Analysis

The dyed samples were evaluated in terms of color coordinates using a Konica Minolta CM-3600d color data software CM-S100w Spectra Magic NX (D65 illuminant, 100 standards observed) spectrophotometer in terms of CIE Lab is the lightness (L), (+a) red, and (-a) green coordinates, (+b) yellow, and (-b) blue coordinates shows. Color Strength (K/S) values were calculated by on Kubelka- Munk (Equation 1) [16].

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

Where K is the absorption coefficient, S is the scattering coefficient and R is the reflectance of dyed samples.

2.5. Fastness Testing

The dyed wool yarns were tested according to ISO standard test methods. washing fastness was assessed as per ISO 105 C03, and light fastness was assessed as per ISO 105 B02.

2.6. Fourier Transform Infrared (FTIR) Analysis

Fourier transform infrared (Bruker ALPHA model) dyed and undyed wool yarns were analyzed for

yarn-dye interactions (with a resolution of 4 cm⁻¹). Bands in the FTIR spectra were analyzed in accordance with the literature data.

2.7. Scanning Electron Microscopy (SEM) Analysis

The morphologies of dyed wool yarns were characterized using a scanning electron microscope (Carl Zeiss Sigma 300 Field Emission). The operating voltage is 10 kV.





3. RESULTS AND DISCUSSION

3.1. Colour Analysis

In terms of the color change observed in triple dye combinations at different pH and different dyeing prescriptions of dyed yarns were tested for changes in the color of shades. The results of color measurements of shades obtained are shown in Table 2. The L* takes a value between 0 and 100, where where the value of 0 indicates black and 100 indicates white. The values of a* and b* indicate red (+a*) and yellow (+b*) and indicates green (-a*) and blue (-b*).

Table 2 shows the colorimetric data and color strength value of the dyed yarns. All samples dyed with ternary dye solutions were found in the red-yellow quadrant of the CIELab color space.

Table 2. The colorimetric data and K/S of the dyed wool yarns based on CIELab coordinate

Dyeing no	L^*	a^*	b^*	ΔL^*	Δa^*	Δb^*	ΔE	K/S	
Control	70.090	-1.654	9.123						
2	30.22	3.14	10.32	-39.87	4.794	1.197	40.18	5.19	
3	25.12	8.76	7.12	-44.97	10.414	-2.003	45.98	4.52	
4	52.23	1.12	25.42	-17.86	2.774	16.297	24.336	4.11	

It was found that the redness (a^*), and yellowness (b^*) of shades have increased, and the brightness value L decreased compared to control samples. However, changes in dyed sample values have shown a different trend of variation. Color strength value (K/S) of dyed woolen on the wool yarn. This could be explained by the amount of ternary solution gradient arising between dye in the solution phase and wool fibers [19]. The high K/S was observed at pH 3.5 and the less K/S was observed at pH 4 and pH 5. Color strength (K/S) values have been shown to decrease with an increase in pH. The in alkaline solution, a reaction with hydroxide ions (OH^-) converts the ammonium ion (NH_3^+) to amino (NH_2) groups and the fiber contains more carboxylate ions (COO^-) [17]. Thus electrostatic repulsion between the anionic colorants and the protein fibers occurs, which leads to a decrease in the dye uptake [17,18]. There are several studies have shown that the color strength value decreases with the increase in with the increase in pH [19,20].

3.2. Fastness Properties

The fastness properties of wool yarns dyed without mordants are shown in Table 3. Samples (2-4) dyed without mordant with three different plant extracts had a fair to good light fastness rating of 3-4. The dyed wool yarns showed good washing

fastness for color change. This is attributed to the wool's ability to form strong coordination complexes with amine and carboxyl groups, which can ionize and serve as both anionic and cationic dye molecule binding sites; thereby resulting in a good fixation on the wool yarn [21-23].

Table 3. Fastness properties for the dyed samples without mordant

Samples	Washing fastness (Color change)	Light fastness
2	4	3-4
3	4	3-4
4	4-5	3-4

3.3. FTIR Analysis

The wool fiber is complex in structure, and it composed of 20 amino acids that can be divided into 4 different groups: cationic, anionic, polar, and non-polar. The main functional groups of amino acids are carboxylic ($-\text{COOH}$) and amino ($-\text{NH}_2$) groups [4-21]. In FTIR spectra (Figure 4) of un-dyed wool yarn, the main characteristic peaks are between 2160.45 and 1000 cm^{-1} which include peaks for amide I, amide II and amide III appearing at $1632,55 \text{ cm}^{-1}$, $1529,38 \text{ cm}^{-1}$, and $1455,37 \text{ cm}^{-1}$ respectively. Which indicates the asset of the amide group. This indicates that it is a

protein-based fiber. Measuring changes in the secondary structure of dyed yarns were evaluated in three ranges: 2500-2000 cm^{-1} , 2000-1500 cm^{-1} 1500-1000 cm^{-1} . It was found that the wavenumber shift of the amide groups (amide I, amide II, and AmidIII) were affected distinctly by the dye [24]. A comparison of the raw wool yarn and dyed wool yarns spectra (Figure 4) revealed that two and one new peaks 1227.62 cm^{-1} , and 1039,08 cm^{-1} ; and 1042.05 cm^{-1} ; and 1035 cm^{-1}) appeared after

dyeing No: 2 and dyeing No:3 and dyeing No: 4 two and one new peaks appeared after dyeing respectively. In dyed and undyed yarns, all peaks remained except 1000-1227 cm^{-1} peak, the peaks at 1000-1227 are an indication of the presence of a disulfide linkage [25,26]. The bands formed as a result of the ionic interaction between the yarn and the dye were not wide, they appeared below 600 cm^{-1} as shown in figure 4 dyeing No: 2,3 and 4 respectively.

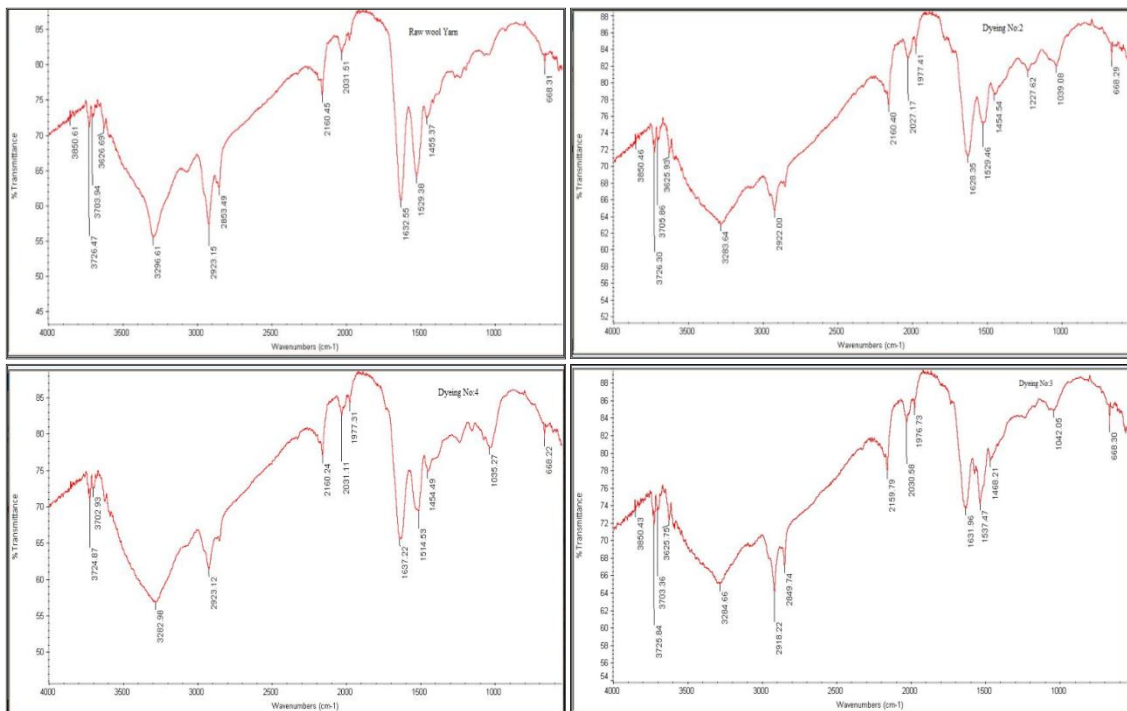


Figure 4. FTIR Spectra of raw wool yarn and dyed wool yarns

3.4. Scanning Electron Microscopy (SEM) Analysis

The morphological properties of raw wool yarn and dyed wool yarns are displayed in (Figure 5a and b, c, and d) Scanning electron microscopy micrographs of undyed wool yarn. The undyed wool yarn consists mainly of core cells surrounded by an outer layer containing cuticle cells. As presented in Figure 5a. It is observed from the undyed wool yarn that there is the cuticular cells of the surface can be clearly distinguished, overlap one another, and are much cleaner and smoother,

and cortical cells are arranged in an overlapping manner. Hassan and Car (2019) investigated the structure and morphologies of wool fiber similar results were found. As seen in SEM micrographs of dyed wool yarns, the visibility and sharpness of the scales decreased after dyeing in figure 5b,c,and d. However, It presented that the dyed yarns were seen without any physical changes such as surface roughness, cracks, etc. In addition, it was observed that the surfaces of the dyed yarns did not change due to the dyeing process. This consequences are in good agreement with a previous study [9,19,26].

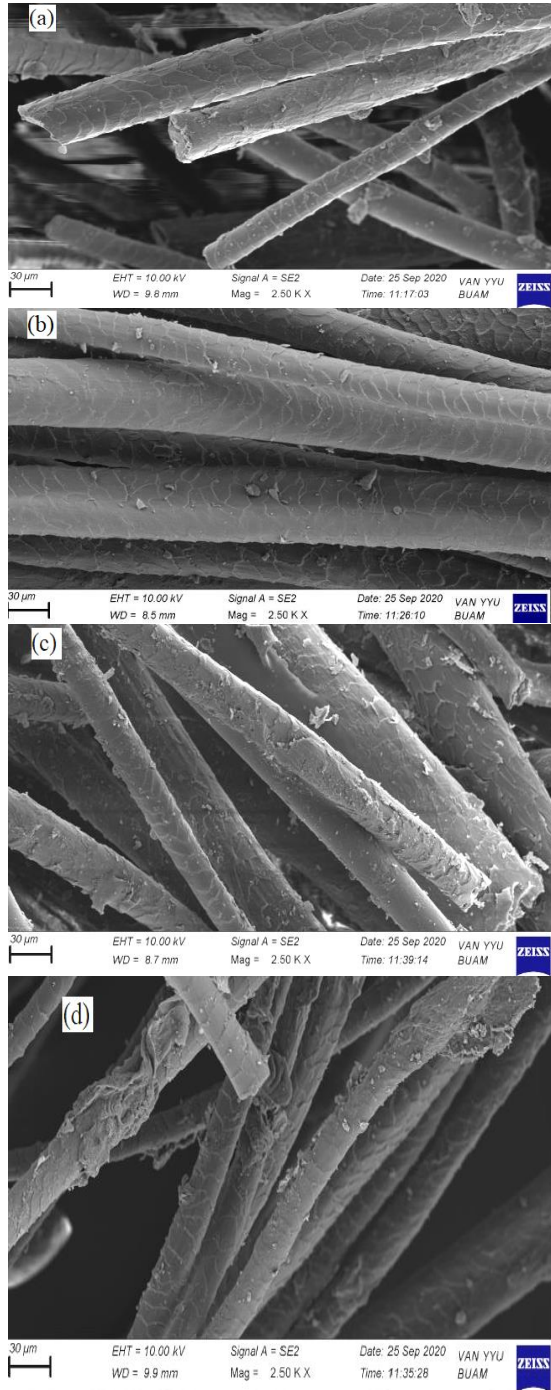


Figure 5. The SEM images of the raw wool yarn (a) and Dyeing No 2 (b) Dyeing No 3 (c) and Dyeing No 4 (d)

4. CONCLUSION

It was found in the study that a combination of ternary dye extracts can be successfully used for dyeing wool yarn to obtain to develop naturally beautiful shades of different colors. Color strength (K/S), and CIE L* a * b* values of dyed yarns were improved with triple dye solution combination, which may be profitably acceptable according to another dyeing process. Also dyeing with ternary dye solution combination method has more overdriven effects than the other conventional methods and this dyeing method will be more eco-friendly due to the use of fewer plants. Moreover, the dyeing process in the laboratory is simpler and more efficient and the dyeing performance is superior. As a result dyeing method with a triple dye solution combination could be a promising and fruitful dyeing method to produce beautiful shades of varying color depths with fair to good light and wash fastness properties.

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