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The effect of different mordants on natural dyeing of cotton, viscose and lyocell fabrics with pomegranate bark extracts

Abstract

Considering the growing environmental awareness, natural dyeing is considered as a sustainable approach in textile dyeing, which can minimize the environmental impacts of synthetic dyes. In this study, it was aimed to compare the dyeing properties of different cellulose-based fabrics (100% cotton, 100 % viscose, 100% Tencel) with pomegranate bark extract as a natural dye by using two different mordants (potassium aluminium sulfate and iron (II) sulfate). Results revealed that potassium aluminium sulfate (alum) and iron (II) sulfate gave different color shades from light to medium yellowish-brown shades with different color depth (K/S) and color vividness (chroma). Alum mordant resulted in lighter, more greenish and more yellowish shades with more vivid color than iron (II) sulfate. The maximum K/S of 3.5 was achieved with viscose fabric mordanted with iron (II) sulfate. All dyed samples had satisfied color fastness values and increased tensile and tear strength compared to raw fabrics.

Keywords: natural dyeing, cellulose-based fibers, pomegranate bark extract, potassium aluminium sulfate, iron (II) sulfate

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Introduction

Textile industry is one of the most polluting industries in the world and especially the effluents discharged from textile dyeing cause serious environmental impact on human health and other living organisms, soil, water and plants. Textile dyes are most dischargeable, highly toxic and potentially carcinogenic or mutagenic pollutants considering current environmental concerns. Synthetic dye effluents are major sources of pollution problems that pose serious threats to human health, soil fertility, and crop production. Due to increasing environmental consciousness, natural dyeing has gained considerable attention in order to minimize the environmental impacts of synthetic dyes in textile dyeing.[1–6] Natural dyes, which are biodegradable, non-toxic renewable source of colouring materials, are required for sustainable textile dyeing. However, natural dyes have a limited color range and poor color fastness to laundering and light compared to synthetic dyes, depending on the type of the mordant used. Mordants are used to form a chemical bridge between dye and fiber, helping the dyes to bind to the fabric, thus improving the color fastness properties in natural dyeing.[7–9]

There have been several studies about natural dyeing using extracted dyes from a variety of plants source. Çalışkan et al [2022] obtained natural dye extract from the dried and ground leaves of the Reseda Luteola plant. It was found that 100% cotton knitted samples mordanted with potassium aluminium sulfate had the highest K/S value of 3.6.[10] Özomay et al.[11] studied on dyeing of cotton fabric with the extract dye from olive leaves. It was reported that dyeings made with potassium aluminium sulphate mordant had higher K/S values compared to other mordants used in this study, citric acid and Quercus Aegilops natural mordant.[11] Kovacevic et al.[12] focused on dyeing of cotton and wool fabric with natural dye extracted from Spartium junceum L. (SJL) flowers, which is an acidic dye. Results indicate that wool samples treated with 3% alum had the lowest Delta E* value of 0.87 after 5 washing cycles, which was within the tolerance limits.[12] Haji[13] investigated the dyeability of plasma-treated and chitosan-coated cotton fibers with an extract of pomegranate rinds. Chitosan-coated and naturally dyed samples had a maximum K/S of 3.85 with plasma treatment time of 5 min and chitosan impregnation time of 35min.[13] Ayele et al.[14] reported that cotton fabrics can be dyed in different shades at various depths with the extracted dye from mango leave and mango peel using different mordants.[14]

Considering the growing environmental awareness, cellulose is one of the most abundant and available renewable resource for textiles. Natural cellulose fibers (cotton), and also regenerated cellulose fibers, which are solvent spun from cellulose pulp, are widely used in apparel industry as they have excellent of drapability, comfort and durability properties. All natural fibers have the same chemical structure of cellulose, but differ in the degree of polymerization, degree of crystallinity and orientation, which affect their mechanical properties.[15–17] Cotton fiber, which has the highest amount of cellulose (~90wt%), is the most used natural fiber for textile clothing and industrial products.[18–19] It contains non-cellulosic components such as waxes, pectins, inorganics and other substances, which are mostly found in the outer layers (cuticle and primary cell wall). Chemical composition of cotton fibers varies depending on environmental factors (water, soil, weathering etc.) and fiber maturity.[20] Cotton fibers exhibit excellent hydrophilic character due to the presence of large amount of OH groups. In the mid-19th century, regenerated fibers were developed as an alternative to natural fibers to efficiently use limited natural resources, promote a sustainable life cycle with a low environmental footprint, and meet people's changing clothing needs. Regenerated cellulose fibers are used in a wide variety of textile products due to their characteristic properties such as tensile strength, smooth and lustrous appearance, excellent water absorption ability and suitability to blend other natural or synthetic fibers.[21] Viscose rayon, the first regenerated cellulosic fiber, is the most commonly used conventional regenerated cellulose fiber. It has low wet strength due to lower degree of polymerization (200-400) than cotton cellulose (3000-4000) and has high elongation up to 25% under dry condition and 30% under wet condition.[22] Lyocell fiber (trade name Tencel), is a new generation of regenerated cellulose fiber produced by a more environmentally friendly process. Lyocell fiber has similar physical properties to viscose rayon fiber, but it has improved properties in terms of softness, drape, dimensional stability, dye retention and color fastness.[17,21] Lyocell fiber has higher tensile strength, higher modulus, lower elongation and slightly lower moisture regain than viscose fiber since the crystallinity of lyocell fiber (80%) is higher than that of viscose fiber (41%).[19,21]

In this study, the effects of two different mordants, potassium aluminium sulfate (alum) and iron (II) sulfate, on dyeing properties natural cellulosic fibers (100% cotton), and regenerated cellulose fibers (100% viscose and 100% Tencel) with pomegranate bark extract as a natural colorant were investigated. Apart from the literature, this study provides a comparison of the natural dyeing performance of three different cellulose-based fabrics with two different mordants. The dyeing performance of extracted pomegranate bark were evaluated in terms of color values (CIE L*, a*, b*, C*, h and K/S) and color fastness properties. Tensile and tear strength properties of the dyed samples were also investigated.

Materials and methods

Materials

In this study, 100% cotton (100Co), 100% viscose (100Cv) and 100% Tencel fabrics (100Tencel) were provided from İpekyol Tekstil ve Konfeksiyon ltd. co. The specifications of the fabrics are given in Table 1. Potassium aluminium sulfate (alum) and iron (II) sulfate were used as mordants to improve dyeability and color fastness properties.

Table 1 Technical properties of the fabrics used in this study

	Definition		
Sample codes	**100Co**	**100Cv**	**100Tencel**
Fiber content	100% Cotton	100% Viscose	100% Tencel
Fabric unit weight (g/m^2) (+/-5%)	108	93	180
Seam slippage (ISO 13936-2) - 6mm opening	Warp: 60 N	Warp: 60 N	Warp: not observed
	Weft: 60 N	Weft: 60 N	Weft: not observed
Tensile strength (ISO 13934-2)	Warp: 12 KG	Warp: 12 KG	Warp: 38.7 KG
	Weft: 12 KG	Weft: 12 KG	Weft: 20.1 KG
Tear strength (ISO 13937-2)	Warp: 4N	Warp: 7N	Warp: 11N
	Weft: 4N	Weft: 7N	Weft: 7N
Pilling martindale (ISO 12945-2)	at 2000 revs	at 2000 revs	at 2000 revs
	grade:3	grade:3	grade:4-5

Methods

Extraction

Pomegranate bark was washed with copious amounts of water and then dried under atmospheric conditions to remove its moisture content. The dried bark pieces were then ground into powder and sieved through a strainer. The dried powder was subjected to the extraction process at 100 ºC for 100 min with stirring. The obtained filtrate was used as stock dye solution for subsequent dyeing of fabric samples.

Mordanting and dyeing

Two different mordants, potassium aluminium sulfate (alum) and iron (II) sulfate, were used for pre-mordanting process of the fabric samples (100% cotton, 100% viscose and 100% Tencel). Pre-mordanting of each fabric samples were performed in a solution containing 1% (w:w) concentration of mordant at a liqour ratio of 1:40 at boiling point for 60 min.

Subsequently, fabric samples were dyed with natural dye extracted from pomegranate bark at 1:30 liquor ratio at 95°C for 35 min. Then, all dyed fabric samples were rinsed thoroughly with cold water and dried under ambient conditions.

Characterization and performance tests

FTIR analysis

Dyed samples were characterized by Fourier Transform Infrared (FTIR) Spectroscopy (Perkin Elmer) using ATR (attenuated total reflection) method at the frequency range of 4000 to 400cm^{-1} at a resolution of 4cm^{-1}.

Color measurements

Color measurements of the dyed samples (CIE L*, a*, b*, c*, h^0 and K/S values) were conducted with Datacolor Spectraflash Spectrophotometer SF 600 Plus using large aperture size (30 mm) under D65 standard illumination and 10° standard observer. In CIE L*a*b* and CIE L*C*h color systems, L* indicates lightness from 0 (black) to 100 (white), a* denotes redness (positive value) and greenness (negative value), "b*" represents yellowness (positive value) and blueness (negative value). The vividness or dullness of a color (saturation) is expressed by C*, called chroma. h° is the hue angle in the range of 0º to 360º.

The color strength expressed as K/S of the dyed samples was measured by the reflectance of light using Kubelka-Munk equation, where K is the absorption coefficient, S is the scattering coefficient, and R is the reflectance. Higher K/S value means higher dye uptake, hence better color yield.[23–25] The K/S values were assessed at λmax using Datacolor Spectraflash Spectrophotometer.

$$K/S = (1-R)^2/2R \quad (1)$$

Color fastness tests

The color fastness of the dyed samples against washing, rubbing and artificial day light was determined according to standard test methods ISO 105-C06:2012 (A2), ISO 105X12:2006 and ISO 105-B02:2014, respectively.

Results and discussion

FTIR analysis

FTIR spectra of dyed fabric samples mordanted with potassium aluminium sulfate and iron (II) sulfate are illustrated in Figure 1a & 1b, respectively.

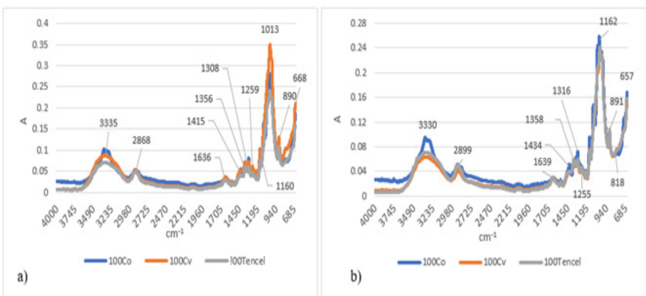

Figure 1 FTIR spectra of different fabric samples mordanted with a) alum b) iron (II) sulfate.

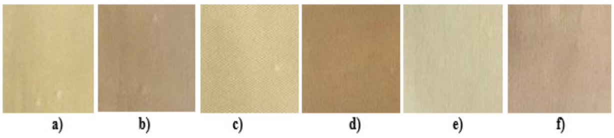

Figure 2 Dyed fabric samples with different mordants a) 100Co mordanted with alum b) 100Co mordanted with iron (II) sulfate c) 100Cv mordanted with alum d) 100Cv mordanted with iron (II) sulfate e) 100Tencel mordanted with alum f) 100Tencel mordanted with iron (II) sulfate.

In Figures 1a & 1b, broad peaks appeared at 3330, 3335cm^{-1} corresponds to characteristic O-H stretching in polysaccharides. The peaks found at 2868, 2899cm^{-1} are associated with the stretching vibration of C-H. The peaks centered at 1636, 1639cm^{-1} indicate O-H bending from absorbed water. The peaks observed at 1415, 1434cm^{-1} belong to the CH_2 symmetric bending of the cellulose, known as the "crystallinity band". The peaks at 1358, 1356, 1316, 1308, 1259cm^{-1} are attributed to the bending vibrations of the C-H and C-O groups. Sharp peaks located at 1013, 1162cm^{-1} is related with the CO and OH stretching vibrations in cellulose polysaccharides. The peaks around 890 cm^{-1} are assigned to C-O-C stretching at the β-glycosidic linkages in the amorphous region.[26–31]

However, characteristic O–H stretching band differs in natural and regenerated cellulosic fibers. Regenerated cellulosic fibers, viscose and Tencel, have rather wide band with narrower peaks around 3000-3500cm^{-1} while natural cellulosic fiber exhibits a distinct peak around 3330cm^{-1}.[32,33] The peaks belonging to the extracted natural dye and mordants on the fabric samples could not clearly detected by FTIR analysis. This may be due to the fact that the signals of cellulose dominate the ATR spectra, making it difficult to identify the dye and mordant components on the fabric samples.[34]

Color measurement results

All dyed fabric samples have different color shades from light to medium yellowish-brown shade with different mordants as shown in Figure 2. Figure 3 and Figure 4 show the color measurement results of the dyed samples with different mordants. In the presence of alum, Tencel fabric has the highest lightness (L*=85.62), and greener (a*= -1.58) and less yellowness (b*= 26.16) shades compared to cotton and viscose fabrics. Cotton and viscose fabrics have higher chroma values (c*=30.34 and 30.31, respectively) than Tencel fabric (c*=26.21), indicating more vivid color. All samples have similar hue angle in the range of 91-93, giving different shades due to varying saturation.

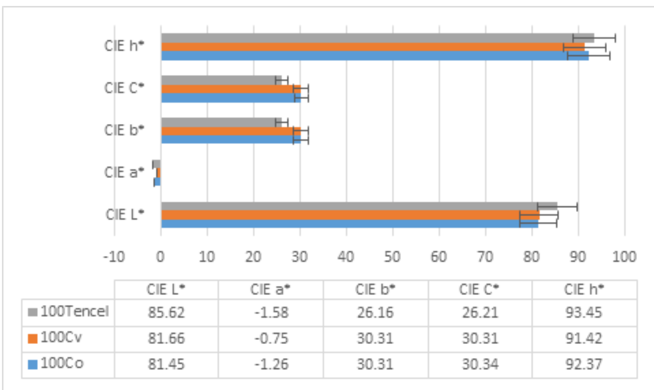

Figure 3 *CIE* L*a*b* and *CIE*. L*C*h* values of the dyed samples in the presence of alum.

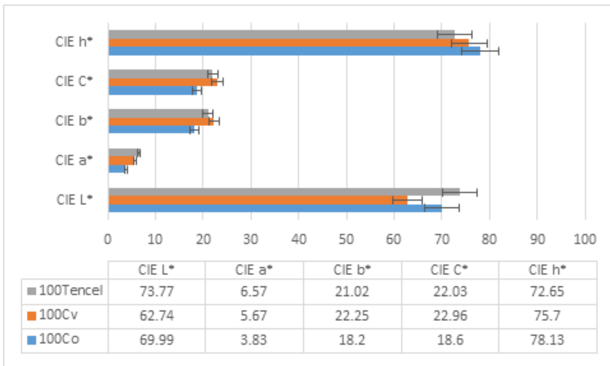

Figure 4 *CIE* L*a*b* and *CIE*. L*C*h* values of the dyed samples in the presence of iron (II) sulfate.

As a comparison of the two mordants, samples mordanted with alum had higher lightness (L*) and chroma (c*) value than those of iron (II) sulfate, indicating lighter and more vivid shades. Addition of iron sulfate results in reddish shades with hue angle ranging from 72-78 while alum gives more greenish and yellowish shades with hue angle ranging from 91-93. Viscose fabric mordanted with iron (II) sulfate has a maximum K/S of 3.5 and the lowest lightness (L*= 62.74) with a chroma value of 22.25.

Color strength values (K/S) of the samples are given in Figures 5. As can be seen from Figure 5a, among the samples mordanted with alum, cotton sample has the highest K/S value of 1.7 while Tencel fabric has lowest K/S value of 1.0. Figures 5a-b indicate that iron (II) sulfate and alum resulted in the same the K/S value for cotton sample but gave different color shades (Figure 2). In the presence of iron (II) sulfate, the K/S value of viscose and Tencel fabrics increased from 1.5 to 3.55 and from 1.0 to 1.4, respectively. The increase in K/S value of viscose fabric is found to be greater than that of Tencel fabric, which could be attributed to the higher crystallinity of lyocell fiber compared to viscose fiber, resulting in lower dye uptake.[19,21]

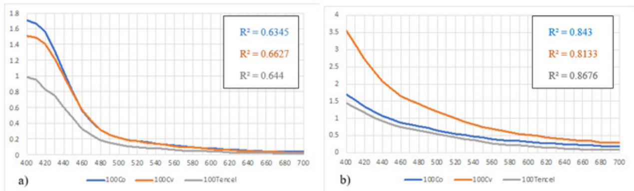

Figure 5 K/S graphs of the dyed samples in the

Tensile and tear strength results

Figure 6 illustrates the tensile and tear strength of raw and dyed samples. It can be clearly seen that dyed samples have higher tensile and tear strength compared to the raw ones. This could be due to the presence of metallic salts as mordant since the metallic salts can react with both the dye and the fiber, leading to the formation of coordination bonds with the dye molecules.[35,36]

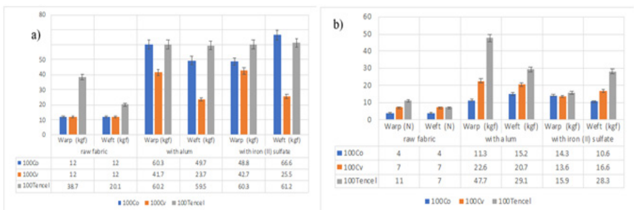

Figure 6 Dyed samples with different mordants a) Tensile strength results b) Tear strength results**.**

Color fastness results

Fabric samples dyed with different mordants were tested for color fastness properties, namely washing, rubbing, and light fastness. The results of color fastness to washing, rubbing and light fastness of all dyed samples are presented in Table 2. All samples have excellent (4/5) washing fastness, indicating that there is no perceived color change and staining on adjacent fabrics. However, the samples have excellent to good dry rubbing fastness (4/5-4) and medium to good (3/4-4) wet rubbing fastness, except from viscose mordanted with iron (II) sulfate, which exhibited slightly fair wet rubbing fastness (2/3). Light fastness of samples ranged from moderate to slightly fair (3-2/3) for the samples mordanted with alum whereas it was moderate for the samples mordanted with iron (II) sulfate (3/4).

Table 2 Color fastness test results

Material		Washing fastness							Rubbing fastness		Light fastness
		Color change	Staining								
			CA	CO	PA	PES	PAN	WO	Dry	Wet	
100Co	With alum	4	4/5	4/5	4/5	4/5	4/5	4/5	4/5	3/4	3
100Cv		4	4/5	4/5	4/5	4/5	4/5	4/5	4/5	3/4	2/3
100Tencel		4	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4	3
100Co	With iron (II) sulfate	4	4/5	4/5	4/5	4/5	4/5	4/5	4/5	3/4	3/4
100Cv		4	4/5	4/5	4/5	4/5	4/5	4/5	4	2/3	3/4
100Tencel		4	4/5	4/5	4/5	4/5	4/5	4/5	4	3/4	3/4

Conclusion

This study aims to investigate the natural dyeing performance of three different cellulose-based fabrics (100% cotton, 100% viscose and %100 Tencel) with pomegranate bark extracts in the presence of two different metallic mordants, potassium aluminium sulphate (alum) and iron (II) sulfate.

Experimental results show that two mordants gave different color shades from light to medium yellowish-brown shade with different vividness (C*= 18.6-30.34). The use of iron (II) sulfate as a mordant yielded darker, redder and less yellowish shades compared to the alum mordant. On the other hand, alum mordant gave lighter, more greenish shades with more vivid color. Based on the K/S value, cotton fabric has the same dye uptake (K/S= 1.7) capability in the presence of alum and iron (II) sulfate. Iron (II) sulfate provided an increase in K/S value from 1.5 to 3.55 and from 1.0 to 1.4 for viscose and Tencel fabrics, respectively. Maximum K/S of 3.5 and lowest lightness (L*= 62.74) were obtained for viscose fabric mordanted with iron sulfate. Both mordants resulted in increased tensile and tear strength compared to the raw fabrics. The dyed samples exhibited excellent washing fastness, excellent to good dry rubbing fastness (4/5-4) and medium to good (3/4) wet rubbing fastness. Iron (II) sulfate imparted better light fastness properties to all fabrics (gray scale rating of 3/4) compared to the alum mordant (gray scale rating in the range of 3-2/3).

This study presents the comparison of the dyeing performance of different cellulose-based fibers with pomegranate bark extract in the presence of different mordants. It can be concluded that cellulose-based fabrics dyed with pomegranate bark extracts by using different mordants have yellowish-brown color shades with different vividness and, may have the potential to be used in different areas depending on their color fastness values.

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Conflicts of interest

The authors have no conflicts of interest to declare that are relevant to the content of this article.

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