



Gaziosmanpaşa University

Graduate School of
Natural and Applied Sciences**Journal of New Results in Science**

Received: 27.11.2013

Accepted: 08.01.2014

Editors-in-Chief: Naim Çağman

Area Editor: Yakup Budak

Investigation of Dyeing Properties of Vine (*Vitis vinifera* L.) Leaves on Wool and Cotton FabricsAdem Önal^{1,a} (aonal@gop.edu.tr)Ferda Eser^a (ferda.eser@gop.edu.tr)Soner Kiraci^a (foreversonyy@hotmail.com)^aGaziosmanpaşa University, Natural Dyes Application and Research Center 60250 Tokat, Turkey

Abstract - In this work, *Vitis vinifera* L. leaf was used to dye wool and cotton fabrics at different dyeing conditions. The solutions of 0.1 M $K_2Cr_2O_7$ and $AlK(SO_4)_2 \cdot 12H_2O$ were used as mordant agents. Pre-mordanting, together mordanting and last mordanting methods were applied at a medium pH value in the dyeing of the fabrics. According to the fastness results, it can be concluded that cotton fabrics have better fastness values than wool fabrics. The best dyeing method was obtained using last mordanting method with $K_2Cr_2O_7$ as a mordant for cotton fabric. The results also reveal that the *V. vinifera* shall probably be an important raw material for dyeing process of cotton fibers.

Keywords -
Wool, cotton,
Vitis vinifera, dyeing,
fastness

1. Introduction

The use of eco-friendly and biodegradable materials is increasing day after day due to the global concern on the application of natural dyes in textile dyeing all over the world [1]. Although natural dyeing is practiced only as a handcraft and synthetic dyes are being used in all commercial dyeing processes in most of the countries, the use of natural dyes has again gained interest [2, 3] because of their non-allergic and non-toxic properties [4]. The incremental value of natural dyes is not only due to the fact that they are a rich and varied source, but also they have the possibility of an income [5]. The problems occur with the usage of natural dyes including reproducibility results, limited shades and blending problems [6, 7] can be overcome by using mordants which provide affinity between the dye and the fabric [8, 9]. Despite of some limitations of natural dyes they are renewable sources and have minimal health hazards, mild reaction conditions and no disposal problems [10].

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Vitis vinifera, which belongs to the *Rhamnales* family, is one of the most frequently consumed fruits in many regions throughout the World. It also contains sucrose, invert sugar and tannin compounds as well as quercetin [11-13]. Due to its high phenolic character, the leaves of the plant were investigated in terms of dyeing properties. The main flavonoids of *V. vinifera* were determined as catechin and quercetin [14]. The structures of the flavonoids are given below (Figure 1).

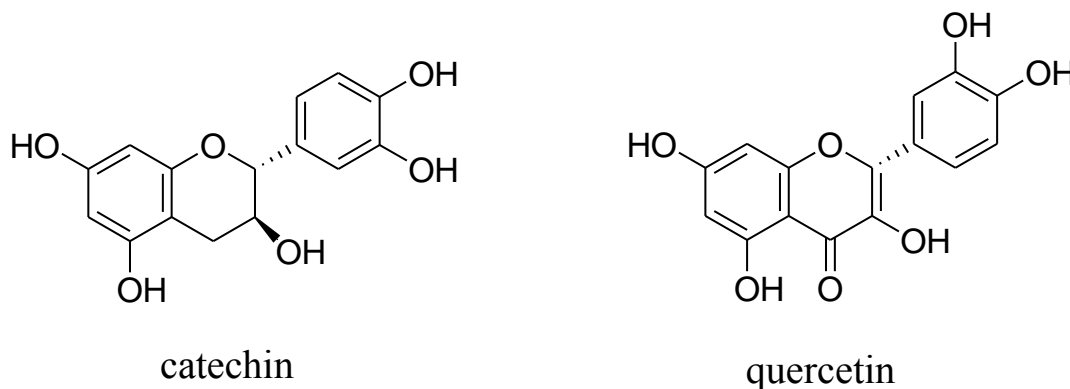


Figure 1. Chemical structures of *V. vinifera* flavonoids

The acquired dyeing and fastness properties of wool and cotton fabrics are very important characteristics in terms of users. The interaction of mordant compounds with wool and cotton fibers affects the affinity to fibers of dyestuffs. Thus, the present study evaluates the dyeing properties of wool and cotton fabrics via fastness values using *Vitis vinifera* L. leaves extract.

2. Experimental

2.1. Fabrics

Wool and cotton and fabrics were chosen as the fabric types to be studied. The characteristics of the fabrics are shown in Table 1.

Table 1. Characteristics of the used fabrics

Fibre type	Mass per unit area (g/m ²)	Surface type	Fabric density
Wool	180	weaved	Weft:28, warp: 30
Cotton	150	knitted	Course:18, Wale:13

2.2. Natural Dye Extraction and Mordanting

The leaves of *V. vinifera* were collected from the Tokat region (Turkey) and dried outdoors. The material was extracted with distilled water keeping the material to liquor ratio (M:L) at 1:20 by soxhlet apparatus until colorless. 1 L of distilled water was used (for 100 g plant

material) and then the dyestuff was transferred to the aqueous media. After the extraction, the solution was cooled to room temperature and used in the dyeing processes.

2.3. Reagents and Equipment

All chemicals used in this work, were purchased from Merck. Distilled water was used for all steps. $K_2Cr_2O_7$ and $AlK(SO_4)_2 \cdot 12H_2O$ (Merck) were supplied from Alternatif Medical, Tokat, Turkey. Extraction was performed with soxhlet apparatus. Colour codes were determined by Pantone Colour Guide. The wash-, crock- (wet, dry) and light fastnesses of all dyed samples were established according to ISO 105-C06 and to CIS, respectively [15].

2.4. Dyeing Procedures

Pre-mordanting, together-mordanting and last-mordanting methods were used in the dyeing of wool and cotton fabrics along with unmordanted processes. For pre-mordanting, the fabric was first dipped into 0.1 M mordant solution (100 ml) and the solution was heated for 30 min. at 90°C. Then it was cooled and rinsed with deionised water and then poured into the dye-bath solution (100 ml). Dyeing was carried out at 90°C for 1 h. Finally, the material was removed, washed with deionised water and dried at room temperature.

In together mordanting method, both mordant (in solid form that is equal to 0.1 M mordant solution) and the dye solution were transferred into same conical flask and the undyed material was placed into the mixture. Then the mixture was heated at 90°C until 1 h. Then it was cooled and washed with deionised water, squeezed and finally it was dried.

In last mordanting method, the non-colored material (1 g) was firstly treated with the dye solution for 1 h at 90°C. Then the material was cooled, washed twice with distilled water and poured into 0.1 M mordant solution (100 ml). It was heated for 30 min. at 90°C. After the end of the process, the dyed fabrics were rinsed with deionised water.

Finally, for unmordanted process, the fabric was placed into a flask containing 100 ml of dye solution and heated for 1 h at 90°C. At the end of the time, the material was removed and washed with deionised water.

3. Results and Discussion

Textile materials are subjected to various tests including rubbing, washing and light fastnesses in order to evaluate their usage. For this purpose, the durability of the dyeing applied on the textile material to these conditions is extremely important in terms of users, and fastness properties have to be assessed [16].

Caution should be exercised in drawing conclusions from comparison of the fastness ratings between dyeings owing to the significant differences in depth observed.

Table 2 show the fastness properties of dyed materials for wool and cotton fabrics, respectively. The rubbing fastnesses of the fabrics were higher in the dry form than in the

wet form. Generally the higher fastness values were obtained in the presence of $K_2Cr_2O_7$ mordant rather than $AlK(SO_4)_2 \cdot 12H_2O$ mordant for wool and cotton fabrics.

Table 2. Fastness properties of dyed wool and cotton fabrics

Dyeing method	Fiber	Mordant	Rubbing fastness		Washing fastness	Light fastness
			Dry	Wet		
Unmordanting	Wool	-	5	3	2	3
	Cotton	-	5	3	3	4
Pre-mordanting	Wool	$K_2Cr_2O_7$	5	3	3	3
		$AlK(SO_4)_2 \cdot 12H_2O$	5	2	3-4	3
	Cotton	$K_2Cr_2O_7$	5	3	4-5	6
		$AlK(SO_4)_2 \cdot 12H_2O$	5	2-3	2-3	5
Together mordanting	Wool	$K_2Cr_2O_7$	5	5	3-4	5
		$AlK(SO_4)_2 \cdot 12H_2O$	5	3	1-2	3
	Cotton	$K_2Cr_2O_7$	5	4	4	6
		$AlK(SO_4)_2 \cdot 12H_2O$	5	2-3	1-2	5
Last mordanting	Wool	$K_2Cr_2O_7$	5	4	5	5
		$AlK(SO_4)_2 \cdot 12H_2O$	5	2	2-3	3
	Cotton	$K_2Cr_2O_7$	5	4-5	4-5	6-7
		$AlK(SO_4)_2 \cdot 12H_2O$	5	2	2-3	3

The light fastness levels of the cotton fabrics are fairly higher than those of wool fabrics, depending on the types of mordant used. In addition, light fastness levels of fabrics Comparison of washing fastnesses of dyed samples showed that there is no considerable difference between the wool and cotton fibers. The lowest washing fastness was obtained with $K_2Cr_2O_7$ mordant using last mordanting method, in the dyeing of wool.

Table 3. Dyeing conditions and color codes of dyed samples

Dyeing method	Fiber	Mordant	Color codes	Color
Unmordanting	Wool	-	13-0333	limabean
	Cotton	-	14-1210	shifting sand
Pre-mordanting	Wool	$K_2Cr_2O_7$	14-0721	lemp
		$AlK(SO_4)_2 \cdot 12H_2O$	15-0710	silverfesh
	Cotton	$K_2Cr_2O_7$	14-1127	desertmist
		$AlK(SO_4)_2 \cdot 12H_2O$	15-1314	cuban sand
Together mordanting	Wool	$K_2Cr_2O_7$	12-0804	cloudcream
		$AlK(SO_4)_2 \cdot 12H_2O$	13-0624	goldenmist
	Cotton	$K_2Cr_2O_7$	14-1118	beige
		$AlK(SO_4)_2 \cdot 12H_2O$	13-0720	custard
Last mordanting	Wool	$K_2Cr_2O_7$	12-0824	palabanar
		$AlK(SO_4)_2 \cdot 12H_2O$	13-0932	corirsih
	Cotton	$K_2Cr_2O_7$	16-0713	slate green
		$AlK(SO_4)_2 \cdot 12H_2O$	13-1114	mellow buff

Color fastness to washing with soap, wet and dry rubbing rating of both fibers with three dyeing methods using $K_2Cr_2O_7$ mordant exceeded rating of 3 and could be considered as good. The rating ratio of 3 or 3-4 is the lower limit of light fastness value for application as

a textile dye [17]. From these results, it was concluded that $K_2Cr_2O_7$ was better mordant to improve the light fastness of wool and cotton fibers when compared with $AlK(SO_4)_2 \cdot 12H_2O$ mordant.

The color and color codes of dyed fibers are given in Table 3. Generally, beige and sand tones were obtained in the dyeing of cotton fibers, while yellow and cream tones were obtained in the dyeing of the wool fibers. According to the experimental results, it can be concluded that the brightness of the dyed cotton samples are higher than those of the wool fibers.

Dyeing Mechanism of the Fabrics

It is known that the technical performance, including fastness such as washing and light, of many natural dyes can be improved by treatment with metal ions [18].

First, material treats with mordant salt in pre-mordanting method and then dyeing is performed by addition of dyestuff solution. Color tones change by mordanting method. This is because of the light absorption wavelength variation of the complex which occur during bonding of the dyestuff and the fiber molecules. According to the mordanting method, the sequence of bonding of ligand (dyestuff)-central atom (mordant cation) can be considered as given below:

Pre-mordanting: [wool/cotton + mordant cation] (1).....Dyestuff (2)
 Together-mordanting : [wool/cotton + dyestuff + mordant] (all of them mix
 simultaneously)
 Last-mordanting : [wool/cotton + dyestuff] (1).....Mordant (2)
 (1) and (2) show the sequence of process

The detailed mechanism of the dyeing processes were discussed in our previous studies [19, 20].

In the present work, dyeing potential of vine (*V. vinifera* L.) leaves was investigated in terms of fastness values. Moderate values were obtained for washing fastness tests both for wool and cotton fibers. Dry rubbing fastness values were found very good while wet rubbing fastness values are moderate. Light fastnesses of dyed cotton samples were found higher than that of wool samples. The results of the study demonstrated that the fastness values of dyed samples were depended on the dyeing method and the mordant.

Best to our knowledge, there is no study on the dyeing performance of vine leaves extract in literature. However, the results showed that this plant may be used in textile industry. Further research can also be done for the improvement of the fastness values.

Finally, the outcome of this study will facilitate using vine leaves in the dyeing of natural fabrics.

4. Conclusion

Natural dyes were efficiently extracted from vine (*V. vinifera* L.) at $80^\circ C$, for 2 h. The extracted natural dyes were applied for the dyeing of wool and cotton fabrics using different dyeing methods. Mordants had a significant effect on the dyeing performance. Generally the

higher fastness values were obtained in the presence of $K_2Cr_2O_7$ mordant rather than $AlK(SO_4)_2 \cdot 12H_2O$ mordant for wool and cotton fabrics. As a result, the best fastness values were obtained using last-mordanting method for both wool and cotton fabrics.

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