



Natural dyeing of cotton fabrics with licorice extract: the role of mordants in enhancing color fastness and protection from ultraviolet rays

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Abstract

The usage of non-toxic, eco-friendly natural dyes on textiles has achieved notable attention due to increased environmental attention about avoiding hazardous synthetic dyes. This has prompted a return to natural dyes and the search for new sources, especially locally available ones like licorice. In this study, *Glycyrrhiza glabra* extract (70 g/l), prepared using ultrasound assistance, was used to dye cotton samples. The natural dyeing process employed a simultaneous mordanting method with zinc chloride and alum as mordants, in many concentrations (1, 3, 5, 7 and 10 g/l). The color fastness of the dyed samples was evaluated using a scanner and ImageJ. The fastness of the dyed fabrics was tested against washing and rubbing, and samples with licorice extract showed excellent stability. A tear strength test was also conducted to assess the impact of licorice extract dyeing on the mechanical properties of the samples. It was observed that dyeing with licorice reduced the tear strength, but increasing the concentration of mordants improved the resistance to tearing, making the mordanted samples stronger than the mordanted ones.

Keywords: Natural Dyes, *Glycyrrhiza Glabra* (Licorice), Mordants, Zinc Chloride, Alum, UV-Protection.

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1. Introduction

Natural dyes are coloring agents found in nature. They may come from plants, animals, or minerals [1]. The use of colorants dates back to the Ancient Age, and many old civilizations expanded their use of dyes for textiles as weaving techniques advanced [2]. Traditionally, textiles were colored with natural materials readily available in the environment. Nature has provided a wide variety of dye-producing plants, animals, and minerals that can be used for textile coloring [3]. Almost every part of a plant—including leaves, roots, fruit, flowers, bark, wood, and seeds—was used to produce different colors and combinations [2]. Interest in natural dyes has grown again due to strict environmental standards and increased health awareness. Since natural dyes are generally less allergenic and toxic than synthetic dyes and produce wastewater that can be treated biologically, they are increasingly seen as an environmentally friendlier alternative to certain synthetic dyes. Besides textile coloring, natural dyes have notable applications in other fields, such as antimicrobial uses [4] and UV protection [5]. Because most natural dyes lack a strong affinity for

textile fibers—especially cellulosic ones—mordanting is a necessary step in the dyeing process [6]. Mordants are substances that bond with both the dye and the fiber, creating a link that secures the color. There are three main classes of mordants: metallic, oil, and tannins [1]. Metal salts, known as mordants, are added to textile dyeing to solve these problems. Mordants bond with both the dye and the fiber, improving color fastness and depth [6]. Mordanting can be achieved by pre-mordanting (before dyeing), simultaneously mordanting (called meta-mordanting), or it may be a post-mordanting system (after dyeing) [1].

There has been a significant focus on developing efficient and environmentally friendly methods for extracting colorants from plants [3]. Extraction techniques such as maceration, steam distillation, ultrasound-assisted extraction, and supercritical fluid extraction are commonly used. Recently, extraction methods aspire to maximize dye yield and minimize the environmental effect while saving the integrity and quality of the extracted natural dyes [7]. Ultrasonic extraction is more efficient than traditional methods. It is considered environmentally friendly because it reduces energy

and solvent use, improves extraction quality and yield, and takes less time [3].

Glycyrrhiza glabra, known as licorice, belongs to the Fabaceae family and is used in the pharmaceutical, cosmetic, and food industries for its therapeutic benefits as well as its sweetening properties [8]. Licorice extract consists of a wide range of ingredients and nutrients. It has been shown to contain sugars and mineral salts, with key minerals including potassium, calcium, magnesium, and phosphorus, along with saponins. It also contains flavones, tannins, and glycosides [9]. About 400 compounds have been isolated from licorice, including approximately 300 flavonoids, as well as phenolic compounds. The primary active ingredient is glycyrrhizin. It also contains quercetin, one of the most important plant flavonoids, responsible for the yellow coloration [10].

Licorice extract has been used in many studies to color and functionalize fabrics. One study examined the color characteristics and dyeing properties of cotton fabrics dyed with licorice root extract. Mixtures with varying water and ethanol concentrations were used during extraction. Cotton fabrics dyed with licorice extract showed improved ability to remove unpleasant odors and offered UV-blocking properties [11]. Methanolic extract of licorice after ultrasonication treatment showed excellent color depth (K/S) on ultrasonically treated cotton fabric at 65°C for 45 min. It was found that Fe salt (3%) as a pre-chemical mordant and turmeric extract (7%) as a post-bio-mordant enhanced color strength [12]. Licorice was prepared and characterized as a medicinal plant, and the thermal comfort (coolant) and microbial resistance of cotton fabrics treated with licorice were evaluated. Additionally, the study explored the potential use of licorice in treating eye diseases [13]. Licorice roots are used to dye wool fabrics and impart natural antibacterial properties to the dyed textiles. Copper sulfate, tin chloride, iron sulfate, zinc chloride, and potassium aluminum sulfate served as mordants [14]. Licorice root was also used as a flame retardant. The flame-retardant properties were tested on cotton and blended fabrics (cotton/polyester) after treatment with licorice roots and commercial flame-retardant agents. Results indicated that licorice root extracts improved the thermal behavior of fabrics following treatment and dyeing processes [15]. This study aims to dye cotton samples with a natural substance, namely licorice root, due to its environmentally friendly properties, and to impart UV protection to the dyed samples. It also investigates the effect of additives used during the dyeing process on the physical properties of the fabric. So, cotton samples were dyed with ultrasonic-assisted licorice root extract. Both zinc chloride and alum were used as mordants via the simultaneous mordanting method. The impact of zinc chloride on the dyed cotton samples was then compared to that of alum regarding color characteristics and dye fastness to washing and rubbing. The influence of each mordant's concentration on the fabric's mechanical properties was also assessed, along with its effects on functional properties such as UV protection. The findings were compared to those of unmordanted samples dyed with licorice extract.

2. Research materials and methods:

Plain-woven 100% cotton fabric with weights of (185 g/m²) and licorice root powder were obtained from the local market. Zinc chloride (ZnCl₂), aluminum and potassium (II) sulfate (alum) (AlK(SO₄)₂.12H₂O) were purchased from Riedel-deHaën, and used as mordants.

The research process includes the following basic steps: cotton samples pretreatment, extracting licorice dye, and dyeing samples with licorice extract using both zinc chloride and alum as mordants.

The cotton pretreatment process was done using NaOH (4%) and H₂O₂ (8%). The process involves the removal of starch and increasing the hydration of the fabric, thus accepting the fabric for subsequent chemical treatment and dyeing.

The licorice extraction is performed using distilled water with (70 g/l) licorice root powder. Extraction occurred at room temperature using the ultrasound-assisted method (frequency 40 kHz, power 240 W). The mixture is then filtered and placed to remove solid parts.

The pre-treatment cotton samples were dyed using the simultaneous mordanting method at 90°C for 30 min, after dissolving the mordants in the licorice extract, according to the concentrations listed in Table 1. The samples are then rinsed and dried at room temperature.

3. Characterization Techniques:

The rubbing fastness test evaluates the resistance of surface dye molecules to transfer from the tested fabric to a white cloth placed against the tested fabric and in contact with it. The samples were tested using an electronic crockmeter produced by SDL, where the fastness to rubbing is tested according to ISO 105-X12.

The washing fastness test measures the ability of a dye to resist fading when exposed to a washing solution containing soap at a specific temperature. The test is carried out according to the ISO 105-C06:2010.

To examine the strength of dyed samples and the effect of licorice extract and mordants on the cotton samples. The Elmendorf tearing strength tester was used, which operates on the principle of the pendulum motion. The test is performed by releasing a pendulum arm, which strikes and tears a fabric specimen clamped in the instrument. The energy required to tear the specimen is measured and used to calculate the tear strength. The test method is based on the standard ASTM D1424-2115.

The UV protection ability of the dyed samples was also evaluated using a UV spectrophotometer. The eq. (1-2) were used to calculate the UVA average and UVB average values.

$$UVA_{average} = \frac{1}{n} \sum_{320}^{400} T(\lambda) \dots \dots (1)$$

$$UVB_{average} = \frac{1}{n} \sum_{290}^{320} T(\lambda) \dots \dots (2)$$

Where:

T(%) transmission values
λ (nm) wave length

4. Results and discussion:

4.1. Colorimetric properties

Cotton fabric samples were dyed with licorice root extract using two different mordants, zinc chloride and alum, resulting in different color shades. Light yellow color shades resulted when mordanting using zinc chloride, and bright yellow using alum, although the sample dyed with licorice extract without mordant tends to be pale brown, as shown in Table 1.

ImageJ can be used to study fabric color by analyzing images of the fabric and extracting color information. This involves using ImageJ's tools to measure color intensity, identify specific color channels (like red, green, and blue in RGB).

Dyed samples were scanned using an HP Scanjet 3970 scanner, and the Image-J program was used to analyze the colors of the licorice-dyed fabrics, using the image type (RGB color) and the Histogram instruction. The three color constants (R, G, B) were obtained, and eq. (3) was used to convert to grayscale, which depends on the difference in the eye's sensitivity to each color. One of the three colors, representing a large group of shades that fall within the range from (0-black) to (255-white). Also, RGB colors are converted to Lab colors. Because Lab colors are designed to be perceptually uniform, meaning that equal changes in L*, a*, and b* values correspond to roughly equal perceived changes in color by the human eye. This contrasts with RGB, where equal changes in RGB values don't necessarily translate to equal perceived color differences [16].

$$Gray\ scale = 0.299R + 0.587G + 0.114B.. (3)$$

Where:

Table 1. shows the values of the color constants for all samples. It is clear from observing the values that:

1- Samples mordants with zinc chloride, it is noted that the values of the color constants are very close and increase constantly until sample 4 and then decrease in sample 5. Although samples 1-5 are very similar in that they appear to have the same color shade as the stripped eye.

2- Samples mordants with alum are similar in color constants to those mordants with zinc chloride, although they appear more in-depth to the stripped eye, and this is what makes samples (6-10) appear less blue in the color constants table.

3- By observing the grayscale values, it was confirmed that 11 new color shades were obtained differently from the undyed sample. The closest to black was the undyed sample, which took a value of 221, while all other samples were closer to white, that is, using Mordants led to lightening the colors of samples dyed with licorice. This provides different color shades to satisfy consumer preferences.

4- Samples (1-5) have a color closer to light yellow, while samples (9-10) are darker. The reason for this is the formation of zinc oxide, which is white, in the dye bath as a result of the presence of zinc chloride in a alkaline medium, which is licorice extract. This was confirmed by the infrared spectroscopy (FTIR) that tested the resulting residue from the dye bath in Fig.1

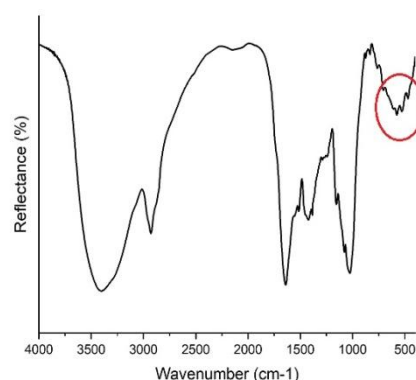


Figure 1. Infrared spectrum of the resulting residue from dyeing bath

4.2. Color fastness to rubbing:

Table 1 shows the results of the wet and dry rubbing test for the dyed samples, where it is initially noted that the sample dyed with licorice extract has excellent fastness to both dry and wet rubbing. Therefore, the role of the mordants in this case is limited to changing the color shades of the dyed samples and not improving the fastness. On the contrary, it's noticed a decline in the fastness of samples dyed using zinc chloride as a mordant has been noticed, especially at high concentrations. The fastness of wet and dry rubbing also decreased when alum was used as a mordant, becoming good after it was excellent without using a mordant.

Table 1. Samples dyed with licorice root extract and their color constants and color fastness

Sample No.	Mordant	Mordant concentration	Sample	R	G	B	Gray Scale	Color Lab	Color Fastness to Rubbing		Color Fastness to Washing	
									dry	wet	dry	wet
undyed	-	-		247,7 8	200, 0	248,9 7	248.95	Lab (98.03, -1.27, 0.14)	-	-	-	-
unmordant	-	-		233.60	223, 1	181,3 0	221.43	Lab (88.71, -2.91, 21.7)	0	ε/0	ε/0	ε
1	Zinc chloride	1		244,0 7	238,1 2	190,1 8	235.15	Lab (93.7, -4.62, 21.62)	0	ε	ε	ε
2	Zinc chloride	3		240,0 0	230, 0	193,3 9	231.93	Lab (92.55, -4.8, 20.93)	0	ε/0	ε/0	ε/0

3	Zinc chloride	5		٢٤١,٦ ٩	٢٣٦,١ ٣	١٩٣,٥ ٤	232.97	Lab (92.93, -4.92, 21.4)	٥	٤/٥	٤/٥	٤/٥
4	Zinc chloride	7		٢٤٣,٦ ٩	٢٣٨,٣ ٦	١٩٧,٤ ١	235.29	Lab (93.55, -5.46, 20.3)	٤/٥	٤	٤/٥	٤
5	Zinc chloride	10		٢٣٩,٢ ٢	٢٣٣,٩ ٢	١٩٧,٥ ٤	231.36	Lab (92.24, -4.09, 18.3)	٤/٥	٤	٤/٥	٤
6	Alum	1		٢٣٩,٤ ٨	٢٢٩,٣ ٦	١٨٠,٨ ٩	226.86	Lab (90.8, -4.04, 24.92)	٤/٥	٤	٤	٤
7	Alum	3		٢٤٠,١ ٦	٢٢٨,٨ ٧	١٧٥,٦ ٣	226.17	Lab (90.62, -4.23, 27.4)	٤/٥	٣/٤	٤	٤
8	Alum	5		٢٣٨,٩ ٢	٢٢٨,٨ ٩	١٦٩,٧ ٢	225.14	Lab (90.4, -5.47, 30.15)	٤	4	٤/٥	٤
9	Alum	7		٢٤٨,١ ١	٢٤٢,٤ ٩	١٩١,٢ ٢	238.32	Lab (94.9, -6.08, 25.48)	٤/٥	٤	٤/٥	٤
10	Alum	10		٢٤٦,٧ ٥	٢٤١,٤ ٢	١٩١,٣ ٣	237.30	Lab (94.6, -6.03, 24.9)	٤/٥	٤/٥	٤	٤/٥

The reason for the low fastness of the dyed samples is that the mineral salts bind the dye to the fabric instead of the natural mordant contained in the licorice extract.

4.3. Color fastness to washing:

The sample dyed with licorice extract has good fastness to washing in terms of color change and staining, as a slight change in color of both the original and accompanying samples is observed after washing. If zinc chloride is used as a mordant, the staining of the accompanying sample is reduced for both concentrations (5, 7 g/l) while the rest of the samples maintain the same stability compared to the reference sample. When using alum as a mordant, a slight color change occurs for two concentrations (1, 3 g/l).

4.4. Tearing Strength of dyed samples:

There were some changes observed in the texture of the dyed samples applied with zinc chloride, as their surfaces had some roughness. Some mineral mordants affect cotton fabrics when applied with natural dyes. This is due to the electrolytes released from these salts and their ability to interact with the cellulose chains that make up the cotton fibers. Fig.2 shows a chart of comparison between samples dyed with licorice extract and mordants using zinc chloride and alum, compared to the unmordant sample as well as an undyed sample. It is clear from the diagram that dyeing cotton samples with licorice extract led to a decrease in the resistance of some samples to tearing compared to the prepared, undyed sample, especially the undyed sample. However, as the concentration of the mordant increased, the resistance to tearing of the samples improved and they became stronger than the reference sample for the concentration (7, 10 g/l) of both zinc chloride and alum. Note that samples fixed with alum have a higher tear resistance than those fixed with zinc chloride. The reason for this is the possibility of zinc oxide being deposited on the surface of cotton samples, which weakens the mechanical properties of these samples.

The mordants and the cotton samples work as a composite material where the mordants enhance reinforcement, which makes the dyed samples stronger.

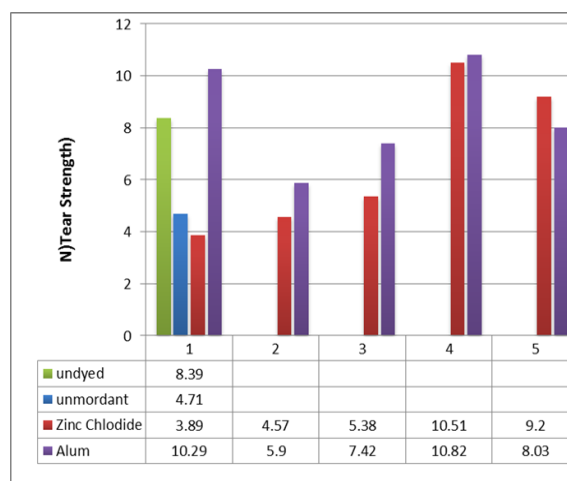


Figure 2. Tearing Strength of dyed samples

4.5. UV Protection of treatment fabrics:

To determine the UV protection of samples, it was calculated UVA average and UVB average by using a spectrophotometer device type (Jasco 530) where weave length range (290-400) nm. The device gives transmission values T(%), and to obtain on UVA average and UVB average, the applied equ (1-2). The fig. 3. shows the values that were obtained.

As it is clear all treatment samples have UV transmission values less than control fabrics, the dyeing with natural dyes improves the protection. Adding mordants change that protection, where: Zinc chloride improved UV protection but with increased concentration above (5g/L) the UV protection starts to decrease. For that sample (4) is the best UV protection in group zinc chloride for both the UVA average and UVB average. At high concentrations, the particles tend to clump together, forming larger aggregates. These larger particles are less effective at scattering and absorbing UV radiation because they have a lower total surface area for a given weight, reducing their ability to block a broad spectrum of UV light effectively.

On the other hand, alum improves UV protection until concentration (3g/L) after that the UV protection decreases. In this group, the best protection for UVA was a sample (6), and the best for UVB was a sample (7). but for all samples, fabric No.3 has the lowest UVA average transmission what is means the best protection

from UVA, and fabric No.7 has less UVB average transmission what is means the best protection from UVB.

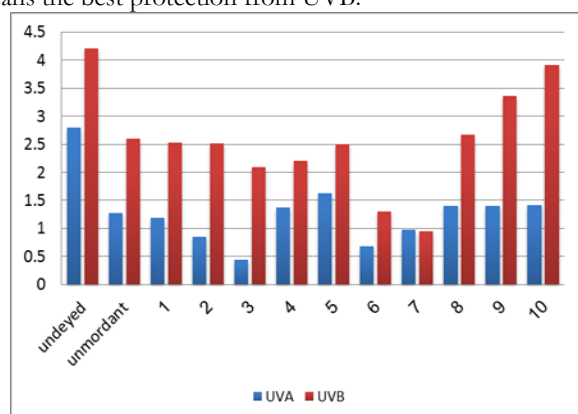


Figure 3. UVA average and UVB average values

In general, changing the concentration of chloride zinc has no significant effect on UVB transmission, also increasing the concentration of alum does not affect UVA transmission even if it increases UVB transmission, which could be explained because of the structure of complex mordants, ZnO, and glycyrrhiza glabra dye.

For more specification, it was calculated ratio UVB/UVA, if the ratio is greater than one the fabric is recommended for use as UVA protection, and if it is less than one, it is used as UVB protection. Fig.4. shows ratio values.

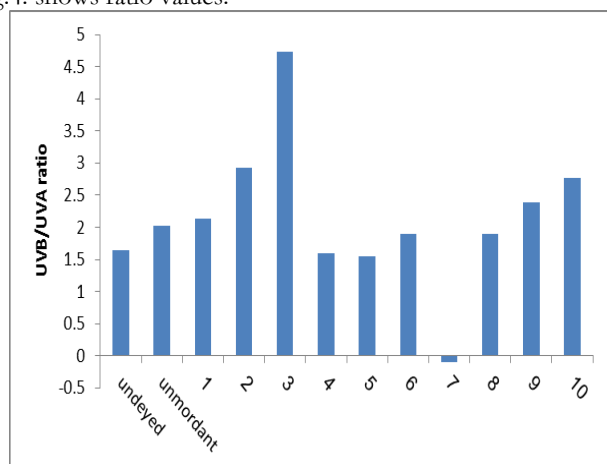


Figure 4. UVB/UVA ratio

It is noticed from fig. 4. All samples recommended for use as UVA protection except sample (7) that recommended for use as UVB protection. And that depend on the skin's situation if it sensitive to UVA rays or UVB rays.

5. Conclusions:

Licorice extract, prepared with ultrasound assistance, was used for dyeing cotton samples. The natural dyeing process was carried out using the simultaneous mordant method, using zinc chloride and alum as mordants. Different color shades were obtained. The color constants of the dyed samples were determined using a scanner and ImageJ software. The fastness of the dyed fabrics was tested with both washing and rubbing. Tearing strength test was conducted to determine the effect of dyeing with licorice extract and the mordants used on the mechanical properties of the studied samples. As it is clear all treatment samples have UV transmission values less than undyed fabric, the dyeing with natural dyes improves the protection. Adding mordants changes that protection. Changing the concentration of chloride zinc has no significant effect on UVB transmission, also increasing the concentration of alum does not

affect UVA transmission even if it increases UVB transmission, which could be explained because of the structure of complex mordants, ZnO, and glycyrrhiza glabra dye. In general, all samples are recommended for use as UVA protection.

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