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Applications of laser radiation on cotton fabrics Dyed gall oak (quercus infectoria olivier)

Mazı meşesi (quercus ınfectorıa olıvıer) ile boyanmış Pamuklu kumaşlarda lazer radyasyon uygulamaları

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APPLICATIONS OF LASER RADIATION ON COTTON FABRICS DYED GALL OAK (*QUERCUS INFECTORIA* OLIVIER)

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ABSTRACT: In this study, at different ratios mordanted with iron (II) sulfate ($FeSO_4.7H_2O$) and potassium aluminum sulfate [$KAl(SO_4)_2 12.H_2O$] cotton fabrics were dyed gall oak (*Quercus infectoria* Olivier). Laser radiation firstly was used on raw cotton fabrics and secondly, cotton fabrics with different ratios of metallic mordanted and dyed with gall oak were irradiated to examine the laser radiation effects on textile color using several combinations of the two main laser parameters namely the number of pulses and energy density at 1064 nm. Colorimetric analysis of the cotton fabrics was measured by CIEL*a*b* spectrophotometer and examined surface alterations. Morphological analysis was performed using optical microscopy. The color coordinates and fastness values of washing rubbing, and light were investigated by comparing them with each other.

Keywords: Cotton fabric, Laser radiation, natural dyes, metallic mordants, CIEL*a*b*.

MAZI MEŞESİ (*QUERCUS INFECTORIA* OLIVIER) İLE BOYANMIŞ PAMUKLU KUMAŞLARDA LAZER RADYASYON UYGULAMALARI

ÖZ: Bu çalışmada, farklı oranlarda potasyum alüminyum sülfat [$KAl(SO_4)_2 12.H_2O$] ve demir sülfat ($FeSO_4.7H_2O$) metal tuzlarıyla mordanlanan pamuklu kumaşlara mazı gomalağı bitkisiyle (*Quercus infectoria* Olivier) doğal boyama işlemi yapılmıştır. 1064 nm'de lazer radyasyonun farklı puls sayısı ve enerji yoğunluğu kombinasyonları kullanılarak ilk olarak ham pamuklu kumaşlarda ve ikinci olarak, farklı oranlarda metalik mordanlanmış ve mazı gomalağı ile boyanmış pamuklu kumaşlarda ışınlama yapılarak renk üzerindeki etkileri incelenmiştir. Pamuklu kumaşların kolorimetrik analizi CIEL*a*b* spektrofotometre ile ölçülmüş ve pamuklu kumaşların yüzey değişimleri incelenmiştir. Morfolojik incelemelerde optik mikroskop kullanılmıştır. Boyanmış örneklerin yıkama, sürtme, ışık haslık değerleri ve renk koordinatları karşılaştırılmıştır.

Anahtar Kelimeler: Pamuklu kumaş, Lazer radyasyonu, doğal boyalar, metalik mordanlar, CIEL*a*b*.

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1. INTRODUCTION

Natural fibers from plants and animals were used in the textile industry for about 5000 years, until the 19th century, when the first man-made (artificial) fiber was produced commercially. Cotton fabrics were used in different parts of the world such as ancient China, Egypt, and Peru. Even textile artifacts found in ancient Egypt indicate that cotton may have been used there as early as 12 000 BC, before flax fiber [1]. Cotton (*Gossypium hirsutum* L.) is a commercial fiber crop for thousands of consumer and industrial products that is being cultivated under diverse climatic conditions across the globe [2]. Today, with the awareness of a healthier and more natural lifestyle, it is an important source of income for millions of farmers and makes a significant contribution to the national economy of many developing countries [3]. The use of cotton in the food and textile industries is increasing because cotton, which supplies about half of global fiber requirements, is produced in more than 60 countries in addition to six major producers (USA, China, India, Pakistan, Uzbekistan, Turkey) [4].

The use of natural dyes in textiles is almost as old as the history of textiles. Archaeological excavations have revealed that natural dyes were the main coloring agents in textile dyeing from prehistoric times until the late 19th century when synthetic dyes were produced on a commercial scale [5]. After the discovery of synthetic dyes by William Henry Perkin in 1856, although natural dyes gradually lost their importance in many parts of the world, people recently have come to realize the toxic effects of synthetic dyes, and interest in natural dyes increased because they are harmless to health and environment [6]. Natural dyes are divided into two categories as herbal (dye plant) and animal (dye insect) based on their biological sources, and it has been explained that approximately 700 dye plants in the world can be used for natural dyeing [7, 8]. Pre-treatment for dyeing textile fibers with natural dyestuffs is called mordanting [9]. Mordants are metallic compounds that can form coordinated and covalent complexes between fiber and dyes in this way improving the staining ability of a dye along with increasing its fastness properties [10, 11]. In the historical process, Alum [$KAl(SO_4)_2 12.H_2O$], iron ($FeSO_4.7H_2O$), and copper ($CuSO_4.$ $5H_2O$) are the most commonly used mordant substances since ancient times, while tin ($SnCl_2 \cdot 2H_2O$) and chromium ($K_2Cr_2O_7$ or $Na_2Cr_2O_7$) were used in the 18th century [12]. *Quercus infectoria* belongs to the Fagaceae family and gall oak (*Quercus infectoria* Olivier) is a semi-deciduous little tree that grows in Asian, Mediterranean countries, and south-eastern Europe[13]. It has been used for dye and ink manufacture from Sumerian time to the present day. This plant contains diverse chemical compounds, such as alkaloids, flavonoids, saponins, and tannins [14, 15]. The natural dye plants are not toxic or carcinogenic and are not harmful to the environment [16]. These plants approximately live between 1 to 2 years and tannins (gallic acid, ellagic acid, tannic acid, and their derivatives) which are obtained from the gall oak shellac plants display different biological activities such as antioxidant, antimicrobial, antifungal, cytotoxic, larvicidal, antiviral, antimutagenic, anti-inflammatory and antiparasite. Gall oak is used both natural dye and also as a bio-mordant substance in the dyeing of natural fiber [17-18].

Textile conservation applications have been made by nations for almost as long as the history of textiles. Conservation science consists of various multidisciplinary branches such as chemistry, biology, physics, humanities, historians, and applied arts [19]. The use of lasers in conservation the of objects of cultural heritage in recent years has become a standard method in conservation since it provides a high degree of control that allows objects with a considerable amount of surface detail and different materials to be successfully and safely cleaned [20]. The unique properties of laser technologies such as density, monochromaticity, directionality, and consistency have supported their use in the conservation of cultural heritage and although laser radiation is not accepted today as one method for all conservation challenges, lasers are useful non-contact and environmentally-friendly vehicles offering great precision and control [21]. Laser technology has been used in different areas of the textile industry in recent years from cutting to the decoration of textile materials. Fading effect of laser engraving is popular and also garment decoration using the laser also gained much popularity [22].

The aim of this research was to investigate the potential use of the Nd: YAG laser as a textile conservation method and to evaluate whether laser radiation has an effect on color of the cotton fabric without causing fiber damage at the surface of a fabric as a direct result of irradiation. The cotton fabrics mordanted at the ratios of 1%, 5%, 10%, 20% of aluminum potassium sulfate [$KAl(SO_4)_2 \cdot 12H_2O$] and 0.1%, 0.5%,1%, 3%, iron (II) sulfate ($FeSO_4.7H_2O$) were separately dyed with 50% gall oak (*Quercus infectoria* Olivier) and a series of applications of laser radiation were carried out on cotton fabrics. It is very important to thoroughly research and analyze the materials before conservation using non-destructive and micro-analysis methods so that the artifact is not damaged.

2. MATERIAL and METHOD

2.1. Fabric and materials

In this study, 100 % cotton plain weave P 1/1 fabric, ready for dyeing, was used. The fabric area density is 90 g ⁄m². The warp and weft yarn count are Ne 35 and the twisted cotton yarn is 970 (Z)T/m. The warp density per cm of the fabric is 60, and the weft density per cm is 40. Gall oak (*Quercus infectoria* Olivier) were obtained from Natural dyes Company (Turkey). Potassium-aluminum sulfate [$KAl(SO_4)_2 \cdot 12H_2O$] and iron(II)sulfate ($FeSO_4.7H_2O$) were obtained from Merck (Darmstadt, Germany).

Figure 1. Gall oak (*Quercus infectoria* Olivier). ©Rkaradag

2.2. The procedure of the mordanting and dyeing:

In this work, seventeen 100 % natural cotton fabrics were dyed gall oak according to the conventional method. Initially, before mordanting cotton fabric was washed with 10 % non-ionic detergent in hot water (about 100 °C) for 60 min. and then dried. Iron (II) sulfate ($FeSO_4.7H_2O$) and potassium aluminum sulfate [$KAl(SO_4)_2 12.H_2O$] were used as pre-mordant in different ratios. Later, the cotton fabrics from the mordant baths were taken and separately dyed with 50% gall oak (*Quercus infectoria* Olivier). After this stage, the dyed-silk fabrics were taken from the beakers and rinsed with tap water. Then, they were dried in open air at room temperature. The procedure of dyeing and mordanting is shown in Table 1.

2.3. Color measurement

The reflectance values of all cotton samples were measured using Datacolor Spectraflash SF 600 + (Datacolor International, USA) instrument with specular-included mode and LAV 30 mm viewing aperture. The untreated fabric was taken as standard and the CIEL*a*b* values were calculated using illuminant D65 and 10° standard observer values [23].

The color differences, according to the CIEL*a*b* (1976) equation, were obtained from the color measuring software. The Kubelka–Munk equation relates the absorption function of the substrate (K), the scattering function of the substrate (S), and the reflectance (R) in the visible spectrum (400–700 nm), as shown below (Equation 1):

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

The color difference is expressed as ΔE* and is calculated by Equation 2:

$$\Delta E* = \sqrt{(\Delta L*)^2 + (\Delta a*)^2 + (\Delta b*)^2} \quad (2)$$

where ΔE* is the CIEL*a*b* color difference between batch and standard. The L* axis represents the lightness of the color (L* is 0 for black, and L* is 100 for white), a* represents the green-red axis (a* negative: green, a* positive: red), and b* represents blue and yellow axis (b* negative: blue, b* positive: yellow)[24, 25].

2.4. Laser radiation treatment:

Nd: YAG laser (Thunder Art Laser, an of the Quanta System) device was used with wavelengths of 1064 nm. The pulse duration is 8 ns, the repetition rate is 20 Hz and the unfocused laser beam diameter is 10 mm with the Gaussian distribution of energy. This is for 1064 nm maximum energy to 900 mJ [25]. The distance between the sample and the articulate arm was set to 10 cm. The diameter of the focused laser beam on the cotton sample surface was around 8.0 mm. Energy density was chosen between 200-250 mJ/cm² in order not to damage fabric because according to the literature energy fluences <1 J/cm² is less destructive for cellulose[26, 27]. For textiles, laser fluence should not be higher than 250 mJ/cm² for all wavelengths [28-29]. Each cotton sample (un-mordanted, mordanted, and dyed fabrics) was exposed to 100, 150, and 200 pulses at a repetition rate of 20 Hz at 1064 nm. The results of laser radiation on cotton fabrics are presented in Table 2.

Table 1. Mordanting and dyeing procedure of cotton fabrics.

Code	Mordant (%)		Gall oak (%)	Ph		Time (min)		Tampareture (^{0}C)		Rate of Mordant	Rate of Dyeing
	Alum (%)	Iron sufate (%)		Mordanting	Dying	Mordanting	Dyeing	Mordanting	Dyeing		
A-B	-	-	-	-		-	-	-	-	-	-
1		0.1	0	6-7	6-7		0		0		
2		0.5		6-7			0		0		0
3		1.0		6			0		0		
4		3.0		4-5			0		0		
5	0	0.1	50	6-7			60		100		
6		0.5		6-7			60		100		50:1
7		1.0		6			60		100		
8		3.0		4-5		60	60	100	100	50:1	
9	1.0		0	5-6	6-7		0		0		
10	5.0			4-5			0		0		0
11	10.0			4-5			0		0		
12	20.0	0		4			0		0		
13	1.0		50	5-6			60		100		
14	5.0			4-5			60		100		50:1
15	10.0			4-5			60		100		
16	20.0			4			60		100		

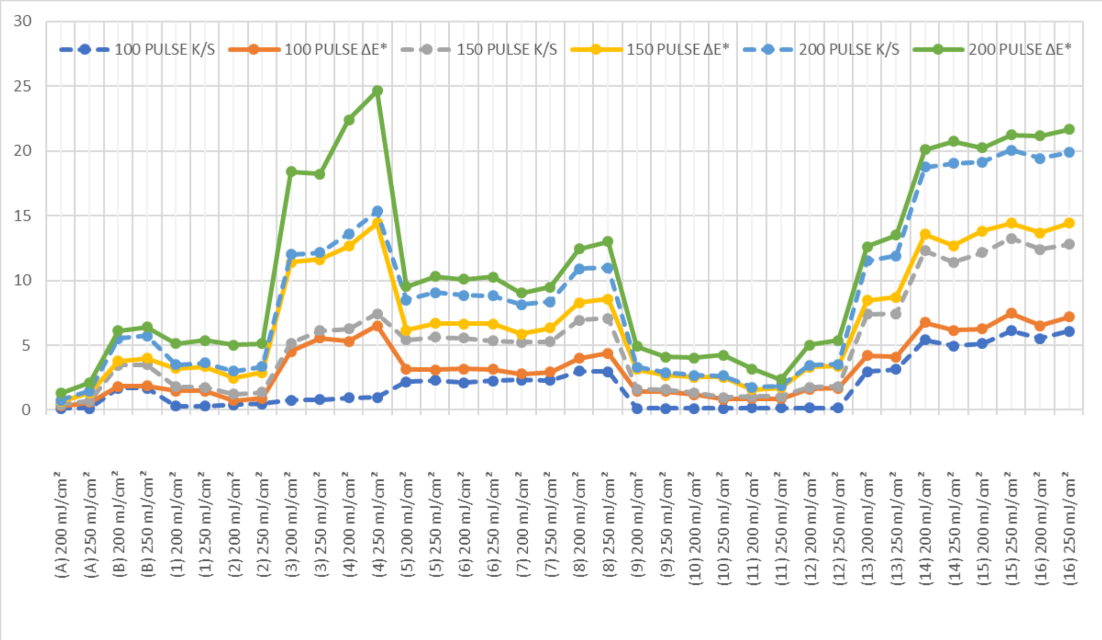
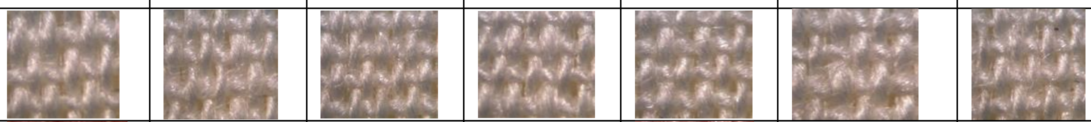
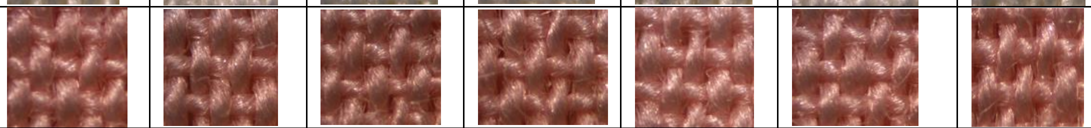
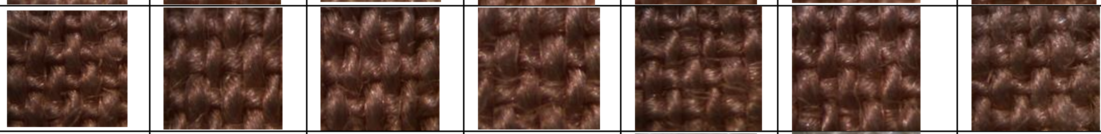
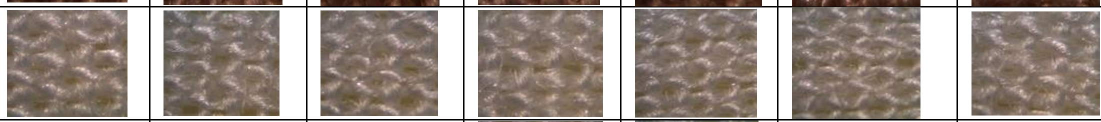
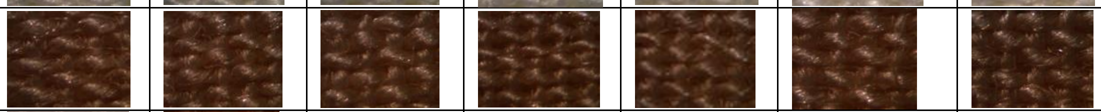
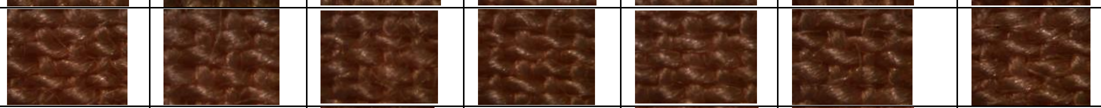


Figure 2. The effect of laser radiation on K/S and ΔE*.

Table 2. Microscopic examinations.

Code	Mordant (%)		Dye (%)	NON İRRİDATİON	Pulse Number					
					1064 nm – 200 mj/cm²			1064 nm - 250 mj/cm²		
	$FeSO_4$	(KAl $(SO_4)_2$.12 H_2O		0	100	150	200	100	150	200
A	0	0	0							
B	0	0	50							
1	0.1	0	0							
2	0.5	0	0							
3	1.0	0	0							
4	3.0	0	0							
5	0.1	0	50							
6	0.5	0	50							

Code	Mordant (%)		Dye (%)	NON İRRİDATİON	Pulse Number					
					1064 nm – 200 mj/cm²			1064 nm - 250 mj/cm²		
	$FeSO_4$	$(KAl(SO_4)_2.12H_2O$		0	100	150	200	100	150	200
7	1.0	0	50							
8	3.0	0	50							
9	0	1.0	0							
10	0	5.0	0							
11	0	10.0	0							
12	0	20.0	0							
13	0	1.0	50							
14	0	5.0	50							
15	0	10.0	50							
16	0	20.0	50							

2.5. Fastness properties

The washing, rubbing, and light fastnesses of eight dyed cotton samples were determined according to ISO105: C06 (A1S), ISO105-X12, and ISO105-B02 standards. The ISO 105:C06 A1S fastness test was carried out at 40°C for 30 min. The dyed cotton samples were exposed to light for 48 h from a xenon arc lamp (250W).

Table 3. The results of light, washing and rubbing fastness tests of dyed cotton fabrics.

Code	Mordant		Dye **(%)**	Light fastness	Rubbing fastness		Color fastness to Washing					
							Staining					
	Iron **(%)**	Alum **(%)**			Dry	Wet	Acetate	Cotton	Nylon 6.6	PES	Acyrlic	Wool
5	0.1	0	50	3	5	5	4-5	4-5	4-5	4-5	4	4-5
6	0.5			3	5	5	4-5	4-5	4-5	4-5	4	4-5
7	1.0			4	4-5	3-4	4-5	4-5	4-5	4-5	4	4-5
8	3.0			4	4	3-4	4-5	4-5	4-5	4-5	4	4-5
13	0	1		3	4-5	4-5	4-5	4-5	4-5	4-5	4	4-5
14		5		4	4-5	4-5	4-5	4-5	4-5	4-5	4	4-5
15		10		4-5	4-5	4-5	4-5	4-5	4-5	4-5	4	4-5
		20		4-5	4-5	4-5	4-5	4-5	4-5	4-5	4	4-5

3. RESULTS and DISCUSSION

The cotton fabrics mordanted at the ratios of 1%, 5%, 10%, 20% of aluminum potassium sulfate (KAl $(SO_4)_2 \cdot 12H_2O$) and 0.1%, 0.5%,1%, 3%, iron (II) sulfate ($FeSO_4.7H_2O$) were separately dyed with 50% gall oak (*Quercus infectoria* Olivier). After these dyeings, the aim was to study the potential use of the Nd:YAG laser and evaluate whether laser radiation has an effect on the color of cotton fabric without causing fiber damage on a fabric surface (Table 1). Different laser devices are used in many areas in the textile industry, but Nd:YAG laser is by far the most widely used laser technology in the conservation of historical textiles due to its local and controlled application.

In order to examine the color differences (ΔE*) and color yield (K/S) values, colorimetric analysis was performed with the Datacolor Spectraflash SF 600+ (Datacolor International, USA) device after fabric dyeing and after laser radiation application. The CIEL*a*b*, color strength (K/S), and color differences values obtained for the cotton dyed fabrics were displayed in Figure 2. Color characteristics of dyed fabrics improved with increasing mordant ratio. The highest K/S values for the dyed cotton fabric samples at the maximum absorption wavelength were 6.04 (20% alum mordanted and 2.63 (3% iron mordanted). When untreated laser radiation samples were taken as a standard, the color difference (ΔE*) values of the samples were found in the range of 0.54 to 9.28 after laser radiation. The color difference (ΔE*) was measured at 0.68 in the raw sample, 0.65 in the only dyed sample and 1,83 alum mordanted sample at 200 pulses. While there was no significant color difference in raw, only dyed and alum mordanted fabrics, the color difference value increased especially when the mordant ratio increased in iron mordant fabrics. The color difference values were measured as 1.72(0.1%), 1.79 (0.5%), 6.36(1%), and 9.28 (3%) in only iron sulfate mordanted cotton samples at 1064 nm and 250 mj/cm² (200 pulses). Color difference values were measured for dyed samples. The color difference values were measured as 1.25 (0.1%), 1.14 (0.5%), 1.45 (1%), and 2.05 (3%) in the dyed samples with iron sulfate mordant at 1064 nm and 250 mj/cm² (200 pulses). As the iron ratio increased, the color difference (ΔE*) value increased only in the fabrics only mordanted, while this difference was measured to a small extent in the dyed fabrics (Figure 2).

For the examination of structural damage in textile samples morphological analysis was performed on cotton fabric, both untreated and after laser irradiation by optic microscopy (SZ-PT Olympus, Tokyo-Japan optical microscope with 60 magnifica-tion). Laser radiation firstly was used on un-mordanted cotton fabrics and secondly, cotton fabrics with different ratios of metallic mordanted and dyed with gall oak were irradiated to examine the laser radiation effects on textile color using several combinations of the two main laser parameters namely the number of pulses and energy density at 1064 nm. The un-mordanted, mordanted, and dyed with gall oak fabric samples were irradiated in different pulses and energy densities.

Fiber morphology was not damaged and discoloration was not observed in un-mordanted raw cotton fabrics after laser radiation at 200 pulses. In the study, in order to better examine the effect of laser radiation on metal salts on cotton, laser radiation was applied to raw fabrics as well as fabrics that were only dyed without mordant. In optical microscope examinations, fiber morphology was not damaged and discoloration was not observed in un-mordanted only dyed cotton fabrics after laser radiation at 250 mJ/cm² energy density. To the naked eye, destruction was observed in cotton samples only mordanted with iron sulfate at 1064 nm laser radiation. It was observed that when the iron(II) sulfate mordant ratio increased, destruction occurred. In the samples with iron mordant, which was only mordanted, the fiber-metal complex was damaged in microscopy, while a trace amount of damage occurred in the fiber-metal-dye complex after dyeing. Such that, in microscopic examinations, in the samples where only iron-(II)-sulfate ($FeSO_4.7H_2O$) metal salt was used as mordant material, destruction occurred when the iron ratio was increased from 0.1% to 3% at 250 mj/cm² (Table 2).

The fastness results were performed for all cotton fabrics dyed and generally found at very good levels. All of the dyed cotton fabrics have excellent fastness levels to rubbing 4-5 in dry and the lowest 3-4 in wet. The results of washing fastness tests also were good and the same for all cotton. Lightfastness was not very good but it is normal for natural dyeing in the cotton fiber. Fabrics dyed with alum mordanted have better light fastness values (4 +) than fabrics dyed with iron mordanted (Table 3).

4. CONCLUSION

Cotton fiber has been produced and used in the textile industry as a textile raw material for many years until the emergence of synthetic fibers. Considered from a historical point of view, painting is as old as the textile industry itself, and many museums today contain artifacts colored with natural dyes. Today, the importance of natural colorants is increasing with various antioxidant, antibacterial and antimicrobial properties, environmental problems, and the negative effects of some synthetic colorants on human health. In this work, organic cotton fabrics were pre-mordanted with Iron-(II)-sulfate ($FeSO_4.7H_2O$) and potassium aluminum sulfate [$KAl(SO_4)_2 12.H_2O$] at different ratios and were separately dyed gall oak (*Quercus infectoria* Olivier). The application of laser radiation on dyed cotton textiles was made as an alternative to the traditional textile conservation of textiles. This study reveals that it is very important to collect data by working with non-destructive and micro-analysis methods and to develop methods in the light of these data, especially before the conservation of historical textiles. It is important to emphasize that laser radiation which has unique properties such as directionality, coherence, and monochromaticity controllably can use for color effect and textile conservation with the right parameters.

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