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Dyeing of Cotton Fabric with Natural Dyes Improved by Mordants and Plasma Treatment

A. Haji

Department of Textile Engineering, Yazd University, P. O. Box: 98195-741, Yazd, Iran

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ABSTRACT

Nowadays, there is a great tendency for eco-dyeing and finishing of textiles with natural products. several natural dyes have been used for dyeing of protein fibers but a low number have the ability to dye cotton fiber because of low affinity of cellulose to the natural dyes. In this study madder and weld natural colorants were used to dye cotton fabric premordanted with three metallic salts including alum, ferrous sulfate, and copper sulfate. The effects of the concentration of mordant, dyeing temperature, dyeing time and amount of electrolyte on the color strength of dyed samples were investigated. Also, an eco-friendly process, low temperature plasma treatment was employed as a pretreatment to improve the dye exhaustion into the cotton fibers. The fastness properties of cotton samples dyed under different conditions were evaluated and the optimum amount of each variable for obtaining the highest values of color strength and fastness properties in the dyeing of cotton with madder and weld natural dyes was specified. Prog. Color Colorants Coat. 12 (2019), 191-201© Institute for Color Science and Technology.

1. Introduction

Textiles made of pure cotton or its blends with synthetic fibers are very popular due to desirable characteristics such as water absorbency, easy dyeing, comfort and stability [1]. Several classes of synthetic dyes including vat, sulphur, azoic, direct and reactive dyes can be applied on cotton fibers [1-4]. The process of cotton dyeing with these dyes usually consumes high amounts of dyes and auxiliaries and produces large quantities of potentially toxic effluent. Furthermore, some synthetic dyes decompose in contact with human sweat and produce banned amine compounds which may cause several diseases. Consequently, the tendency to natural dyes is renewed nowadays [3, 5-12]. Natural dyes are able to dye wool and silk fibers by a simple procedure but the process of cotton natural dyeing is problematic in most cases due to the low affinity and weak fastness properties of natural dyes on cotton fiber [4, 11, 13-18]. Researchers are examining different methods and technologies for the preparation of cotton fibers with the aim of better dyeability with natural dyes.

Ultrasound energy, enzyme, mineral mordants, and biomordants have been used in several studies to improve the dyeability of cotton fiber with different natural colorants [8,19-23]. Pretreatment of cotton with chitosan has been used to increase the affinity of cotton fiber for natural dyes [13, 24-26]. In other studies, anionic and cationic active compounds, cross linking agents and ozone treatment have been employed as pretreatments to improve the dyeability of cotton with natural dyes [3, 8, 11, 20, 21, 27-33]. Application of radiation technologies like plasma treatment, microwave, gamma, and UV radiations has shown the

*Corresponding author: ahaji@yazd.ac.ir

potential to increase the exhaustion and fixation of natural dyes on cotton [13, 19, 34-40].

Madder roots contain different anthraquinones, of which the most prominent structures are alizarin and purpurin that are believed to account for the red color [41, 42]. Weld (*Reseda luteola*) is a perennial plant that produces a yellow dye (luteolin) from its foliage and flowers. It produces the most stable yellow shades and thus have been widely used for dyeing [43].

In this study, the effects of pre-mordanting with three metallic mordants and dyeing conditions on dyeability of cotton fibers with madder and weld have been studied. Also, cotton fabric samples were pretreated with oxygen plasma and the possibility of reducing or elimination of mordanting process before the natural dyeing was investigated.

2. Experimental

2.1. Materials

Scoured and bleached cotton fabric with plain weave (142 g/m^2) was purchased from Mazandaran textile Company, Iran. Madder roots and weld flowers which were obtained from Yazd, Iran and Fariab, Afghanistan, respectively, were finely powdered after being thoroughly washed and dried in the shade. The original solution each plant dye was prepared by boiling 100 gram of powder in 1 liter of distilled water for 2 hours. The resultant solution (10% W/V) was filtered and used for cotton dyeing. All chemicals used in this study were of analytical grade and obtained from Merck, Germany.

2.2. Methods

1- Plasma Treatment: A low pressure, radio frequency (13.56 MHz) plasma treatment system (model: Junior advanced, Europlasma, Belgium) was used. Oxygen with a flow rate of 20 sccm was used as working gas. The chamber base pressure was 100 mTor and plasma treatment was performed at a power of 100 W for 3 min.

2-mordanting: Alum, copper sulfate and ferrous sulfate were used for mordanting of cotton samples. The mordanting process was done at 80 °C for 45 min with a liquor to goods ratio equal to 30:1.

3- Dyeing: The weight of each sample was 5 g For dyeing of each sample, 100 mL of the original dye solution and 100 mL of distilled water were mixed. The dyebath initial temperature was 40 °C and the rate of temperature increment to the final temperature (60, 70, 80, 90 °C) was 2 °C/min. The dyeing was continued for a predefined time (45, 60 and 90 minute) and the samples were rinsed and air dried finally.

4- Color measurements: A reflectance spectrophotometer (Color eye 7000A, X-rite, USA) with illuminant D_{65} and 10° standard observer was employed to obtain the reflectance data and color coordinates (CIE L*, a*, b*) of the dyed samples. Color strength (K/S) of dyed samples were calculated using Kubelka-Munk equation in which R is the observed reflectance, K is the absorption coefficient and S is the light scattering coefficient (Eq.1):

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

5- Color fastness tests: Color fastness of the dye samples against washing, artificial light and rubbing were measured according to ISO 105-C01:2006, ISO 105-B02:2014, and ISO 105-X12:2016 standards, respectively.

Scanning electron microscopy (SEM): An AIS2100 scanning electron microscope (Seron Technology, South Korea) was used to take SEM images and compare the surface morphology of raw and plasma treated cotton fibers.

FT-IR analysis: Fourier transform infrared spectra of raw and plasma treated samples were recorded using a Nicolet 670 equipment in the ATR Mode. 40 scans were done with a resolution of 4 cm^{-1} and their average was used for drawing the spectra.

3. Results and Discussion

3.1. Effect of mordants

The CIELAB color space position of the dyed samples with weld in presence of different metal mordant is presented in Figure 1. The results and observations indicate that they have a yellow color shade due to the higher value of b^* compared to the a^*. However, the pretreatment with different metal salts as a mordant resulted in a little change in their chroma and position. The cotton sample which was pretreated with ferrous sulfate mordant presented the lowest chroma.

The CIELAB color space position of the dyed samples with madder in presence of different metal mordant is presented in Figure 2. The results and observations indicate that they have a red and yellowish-red color shade. However, the pretreatment with different metal salts as a mordant resulted in a change in their chroma and position. Pretreatment with copper sulfate and ferrous sulfate resulted to appear a reddish color compared to the alum mordant. However, the cotton sample which was pretreated with ferrous sulfate mordant presented the lowest chroma.

The effect of three mordants on the L* of cotton samples dyed with weld and madder is presented in Table 1. The L* corresponds to the lightness of them and ranged from 0 (black) up to 100 (white). It is obvious that the L* of the samples dyed with weld decreased as the amount of mordants increased which means that the increase of the amount of mordants in the dyebath has increased the amount of exhaustion of metal ion on the cotton which caused the increase in the color strength of the dyed samples.

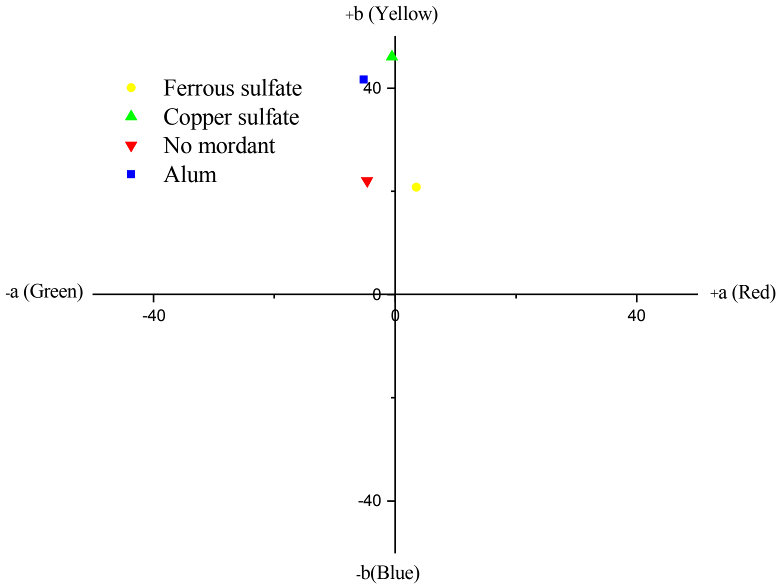

Figure 1: The CIELAB color space position of dyed cotton samples with weld which pretreated with 5 % owf of different metal mordants (dyeing at boil for 60 min).

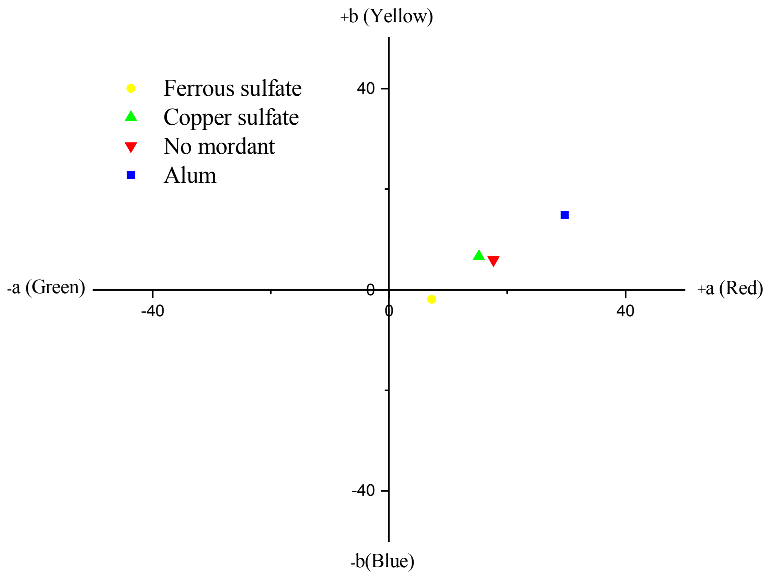

Figure 2: The CIELAB color space position of dyed cotton samples with madder which pretreated with 5 % owf of different metal mordant (dyeing at boil for 60 min).

Table 1: Effect of mordants on color coordinates of samples dyed with weld and madder.

Mordant	Amount of mordant (% owf)	L*	
		Weld	Madder
Alum	1	79.94	49.63
Alum	3	79.02	50.76
Alum	5	78.34	53.11
Ferrous sulfate	1	50.02	31.18
Ferrous sulfate	3	44.36	32.25
Ferrous sulfate	5	41.41	35.86
Copper sulfate	1	70.82	58.31
Copper sulfate	3	68.23	61.03
Copper sulfate	5	66.98	65.57
No mordant	-	81.75	59.32

The data presented in this table show that the use of 1% owf of all mordants decreased the L* of madder-dyed samples compared with the non-mordanted sample, but increasing the mordant amount from 1 %owf to 5 %owf increased the L* that means a decrease in dye absorption in case of madder. This suggests dye desorption from the fabric occurred, in favor of a dye-mordant interaction in the bath rather than on the fiber. Metal mordants can form an insoluble complex with dye molecules. So, this may be due to the formation of a complex between metal ions and the alizarin dye present in the extract of madder in the dyebath, without any attachment to the fiber [44]. It can be concluded that in this case, 1% owf of mordants is the optimum amount for obtaining the highest color strength.

3.2. Effect of Sodium Sulfate

Sodium sulfate (Glauber's salt) is usually used in cotton dyeing with synthetic dyes to accelerate the dyeing process. As shown in Figure 3 and 4, increase in the amount of sodium sulfate decreased the L* of samples dyed with both dyes and all mordants. Cellulosic fabrics gain negative charge in water due to their lower dielectric constant compared with water which cause it to repel the anionic dyestuffs. The electrolytes reduce or distinguish the negative charge on the fiber and facilitates the transfer of the dye molecules from water to the fibers [45, 46].

3.3. Effect of time

Figure 5 shows that the color strength of all samples dyed with weld and madder and different mordants increased as the dyeing time increased. This is simply due to more time for the dye molecule to penetrate to the fiber.

3.4. Effect of temperature

Figure 6 shows that the color strength of all samples dyed with weld and madder and different mordants increased as the dyeing temperature increased. When the temperature increases the aggregates of dye molecules break down, the dye molecules gain more kinetic energy and the fiber swells more and therefore the dye absorption is increased. The lower color strength of the iron mordanted samples may be due to the formation of iron-dye insoluble complex in the dyebath and avoiding the sorption of the dye to the fiber [46]. Thus, the K/S value of the dyed fabric decreases.

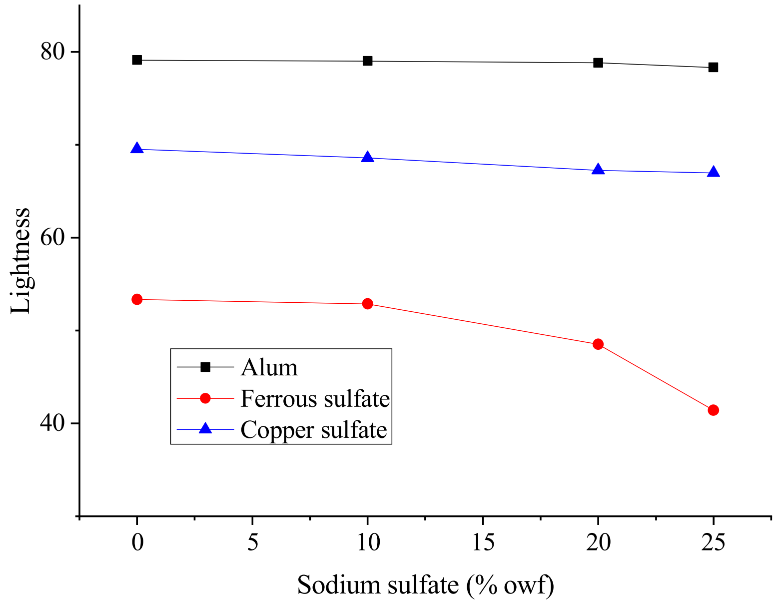

Figure 3: Effect of sodium sulfate concentration on the brightness of samples dyed with weld (mordant concentration = 5 %owf, dyeing at boil for 60 min).

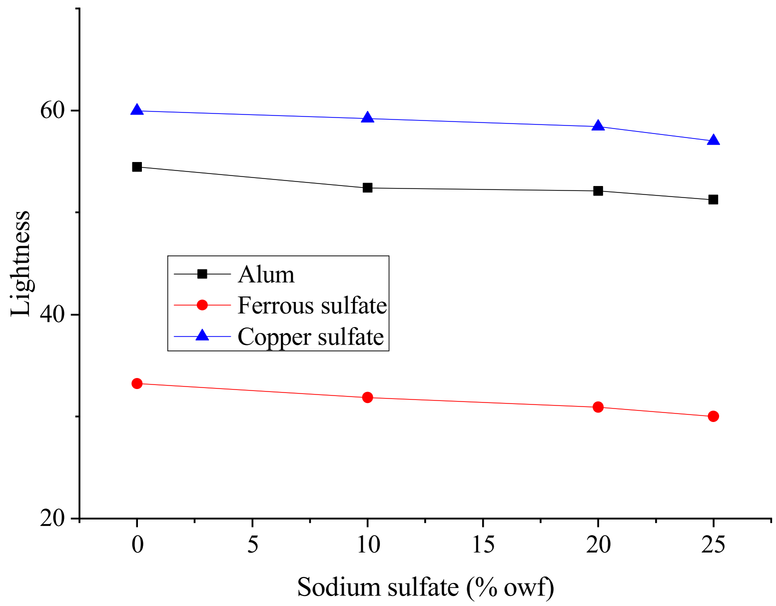

Figure 4: Effect of sodium sulfate concentration on the brightness of samples dyed with madder (mordant concentration = 5 %owf, dyeing at boil for 60 min).

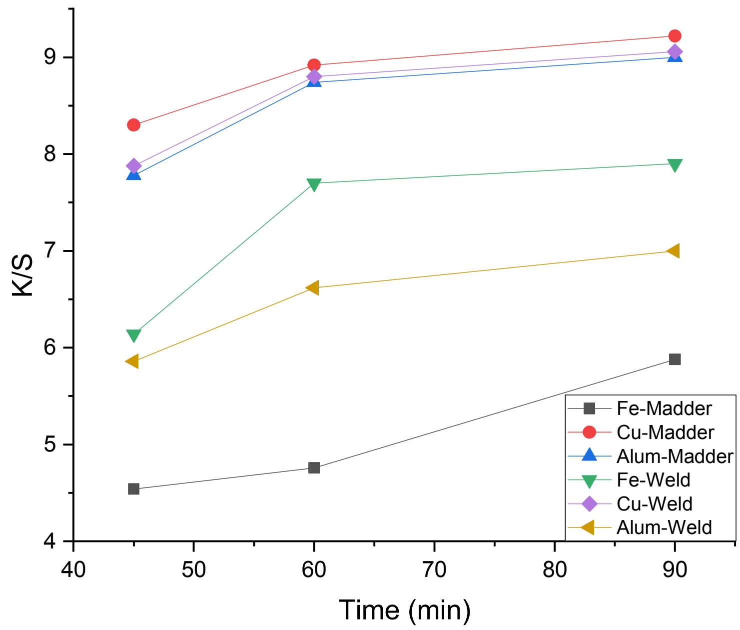

Figure 5: Effect of dyeing time on color strength of dyed samples with weld and madder and different mordants (mordant concentration = 5 %owf, dyeing at boil).

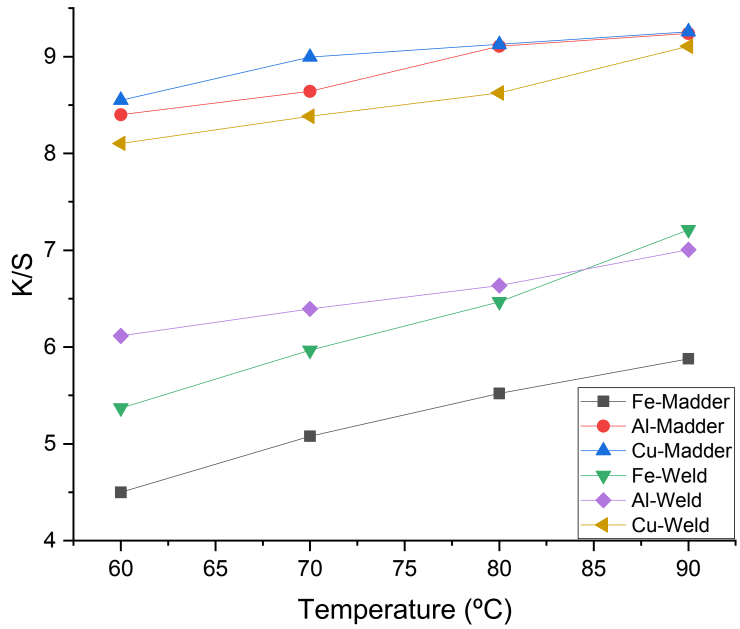

Figure 6: Effect of dyeing temperature on color strength of dyed samples with weld and madder and different mordants (mordant concentration = 5 %owf, dyeing time = 60 min).

3.5. Effect of plasma treatment

Untreated and plasma treated cotton samples were dyed with madder and weld at 90 ºC for 90 min with and without pre-mordanting with alum, ferrous sulfate, and copper sulfate. As can be seen from Figure 7, plasma treatment showed a great effect on dyeability of cotton with both natural dyes with and without mordant. This is due to the etching effect besides the introduction of functional groups to the surface of cotton fibers which caused more absorption of mordant and dye molecules into the plasma treated fibers [13]. The possible interaction between the functional groups of plasma-treated cotton fibers and natural dye molecules (alizarin and luteolin) is presented in Figure 8. It shows hydrogen bonding between the dye molecules and different functional groups of cotton surface, which caused better adsorption and attachment of the dyes on plasma-treated cotton fibers.

The surface morphology of raw cotton fiber and plasma treated sample is presented in Figure 9. The SEM image confirms that cotton fibers sample presented a visible fibril outline and collapsed inward structure. In addition, the plasma treatment resulted in an etching effect on the surface layer of cotton fibers and removed some contaminants. Plasma treatment of cotton fibers resulted in an increase in the diffusion rate of cotton fibers and increased the dye uptake due to their higher color value. As shown in Figure 10, the appearance of small peaks at 1645 cm^{-1} and 1724 cm^{-1} in the ATR-FTIR spectrum of the oxygen plasma-treated sample means that oxygen containing (carbonyl) groups have been created on the surface of cotton fibers after oxygen plasma treatment. The results indicate that plasma treatment can be used as a pretreatment to improve the dyeability of cotton and reduce the need for toxic metal mordants [4].

3.6. Fastness properties

Tables 2 shows that all mordants have a positive effect on fastness properties of samples dyed with weld and madder. This may be due to complex formation between the dye molecules and metal ions in the cotton fiber. Also, plasma treatment improved the fastness properties of dyed samples which may be due to better penetration of dye molecules and more chemical bonding between the newly introduced functional sites of cotton fibers and dye molecules [13].

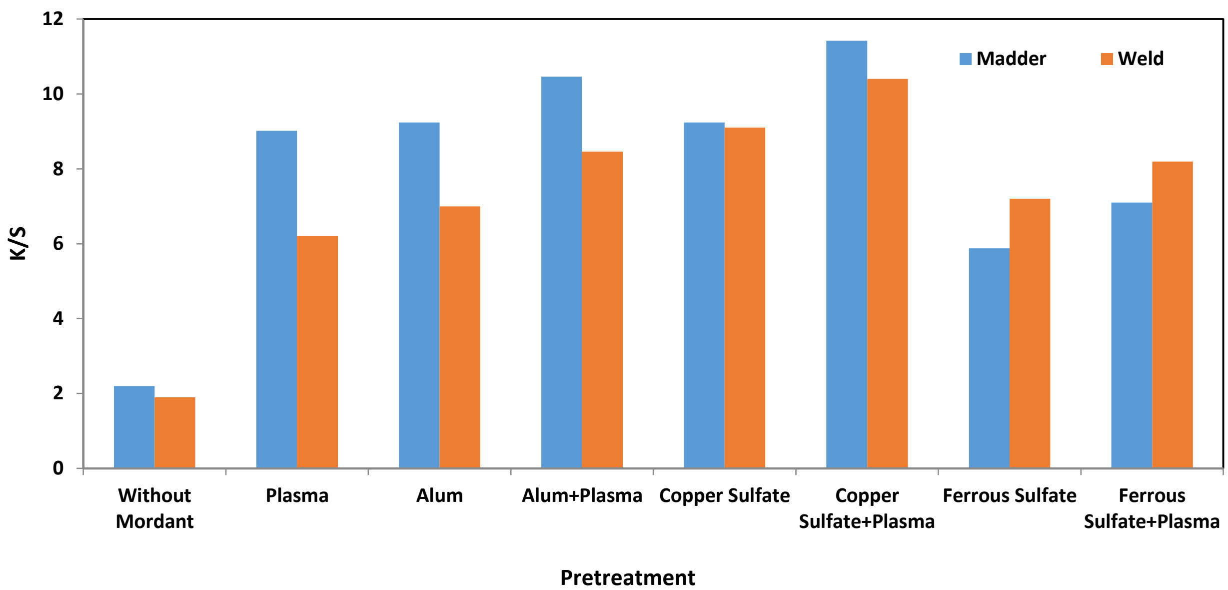

Figure 7: The effect of plasma treatment on color strength of cotton samples (mordant concentration = 5 %owf, dyeing at boil for 60 min).

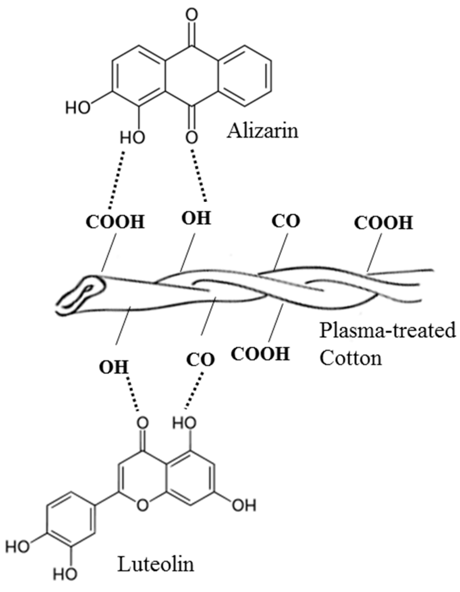

Figure 8: Suggested interaction between plasma-treated cotton fibers and dye molecules.

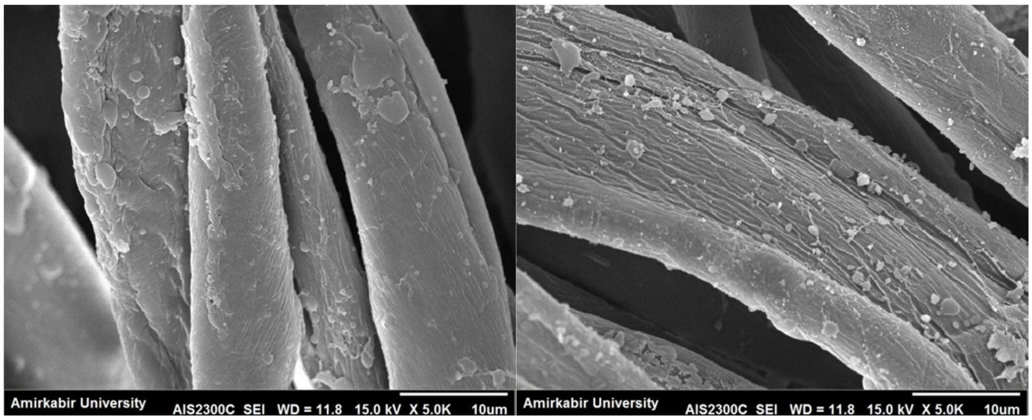

Figure 9: SEM images of raw (left) and plasma treated (right) cotton fibers.

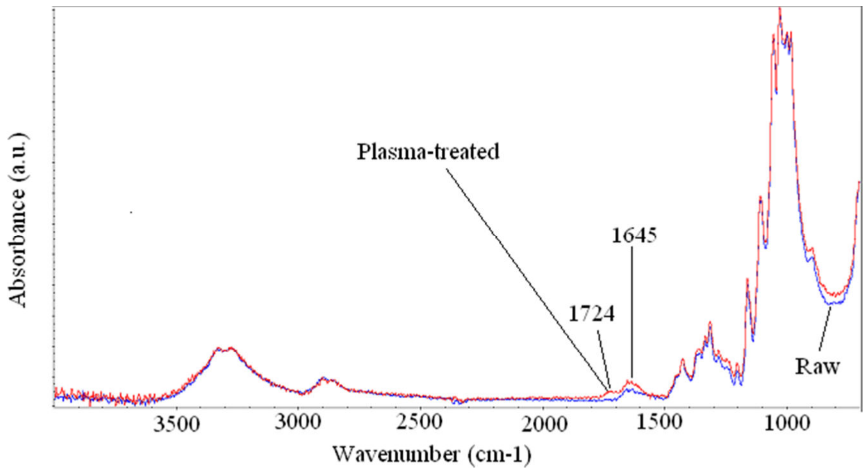

Figure 10: ATR-FTIR spectra of raw and plasma treated cotton samples.

Table 2: Effect of mordanting on fastness properties of samples dyed with weld and madder.

Mordant	Amount of mordant (% owf)	Weld			Madder		
		Rub fastness	Wash fastness	Light fastness	Rub fastness	Wash fastness	Light fastness
Alum	1	3	2	4	3-4	2-3	4-5
Alum	3	3-4	2-3	4	3-4	2-3	4-5
Alum	5	4	3-4	4-5	4	3-4	5-6
Ferrous sulfate	1	2-3	2-3	4-5	3	3	5
Ferrous sulfate	3	3	2-3	5	3	3-4	5-6
Ferrous sulfate	5	4	3-4	5-6	4	4	6
Copper sulfate	1	2-3	2-3	4	2-3	2-3	4-5
Copper sulfate	3	3-4	3	4-5	3-4	3	4-5
Copper sulfate	5	4	4	5-6	4	4	5-6
Without mordant	-	2	2	3-4	2-3	2-3	4
Without mordant, plasma treated	-	3-4	4	6	3-4	4	6

4. Conclusions

Two natural dyes were used in this study. The results showed that these dyes can be used for coloration of cotton fiber with the help of metal mordants. If not mordanted, the dye absorption is not good but mordanting with alum, ferrous sulfate and copper sulfate increased the dye absorption and fastness properties of dyed samples. The use of sodium sulfate in dyeing bath can increase the dye absorption. Increasing dyeing time and temperature has an increasing effect on dye absorption. The plasma treatment was used as a pretreatment to improve the dyeability of cotton with natural dyes and improved the fastness properties of the dyed samples.

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