

Green Dyeing of Woolen Yarns with weld and Madder natural Dyes in the Presences of Biomordant

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ABSTRACT

Nowadays, using sustainable dyeing process has become a necessity, but does not meet performance requirements. Application of biomordants was recommended to enhance the quality of dyeing. In order to design a green dyeing process, oak extract as bio-mordants and madder and weld luteola as natural dyes were used. The FTIR-ATR spectra of the washed wool yarn, mordanted wool yarn and mordanted dyed wool yarns confirmed bonding of wool yarns to the green mordant and dyes through a comparing chemical structure. The effective linkage between yarn, mordant and dye molecules removes the C-N peaks in FTIR-ATR spectra. After dyeing process, fastness and colorimetric attributes of dyed wool yarns were investigated. The K/S values of dyed wool yarns with extracted madder and weld luteola was quantified; properties including light, wash, and rubbing fastnesses were defined as per ISO standard methods. Prog. Color Colorants Coat. 14 (2021), 35-45© Institute for Color Science and Technology.

1. Introduction

The art of using dyes for coloration of textile materials is one of the oldest techniques ever known to man and dates back to the dawn of the civilizations [1]. This is a result of ecological concerns related with the use of azo dyes due to release banned carcinogenic amines as their primary photolytic degradation products, which have motivated researchers all over the globe to explore new eco-friendly substitutes for minimizing their negative environmental impacts [2, 3]. Iran has a rich wealth of dye yielding plants which can be used for coloration of textiles, food, leather and hair, in addition to their advantageous nature in terms of perfumes, leather finishing, and cosmetic [4, 5]. In the application of

natural dyes, different dyeing and mordanting techniques and post-treatment were used to improve color fastness properties. As a result, optimization of the dyeing conditions with regard to the type of natural dye is quite common and a broad set of variations in the dyeing recipes is given in the literature [2, 6].

Chakraborty et al. used *Acacia auriculiformis* for simultaneous dyeing and finishing of protein and cellulosic textile substrates. Wool and silk fabrics dyed with *Acacia auriculiformis* exhibit excellent color fastness to washing and good light fastness properties. The phytochemical studies indicate the presence of saponin, phenols, tannin and glycosides in the aqueous extract of the colorant. Cotton and wool fabrics dyed

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with aqueous extract of *Acacia auriculiformis* (200 g/L) results in maximum UPF value [7]. Yin et al. extracted natural colorant anthocyanin from purple sweet potato powder via ultrasound-assisted ammonium sulfate/ethanol aqueous two-phase system. The extracted natural colorant anthocyanin was used to dye silk fabric in a relatively ecological approach. Based on the single-factor extracting experiment, central composite design-response surface methodology was employed to optimize the ultrasound-assisted aqueous two-phase system extracting conditions including ultrasound temperature, ultrasound time and dosage of purple sweet potato powder. The color strength value of the silk fabric treated with mordant dyeing method was 4.5, and increased by 40.6% compared to that of the direct dyeing method [8]. Ali et al. extracted red prickly pear was used for dyeing wool with different types of mordants. The effect of mordant concentration on the color strength was discussed; the results obtained indicated that the color strength decreases with the increase of mordant concentration. Good fastness properties were achieved from the dyed fabric [9]. Prabhu et al. extracted fruit tannin from *Emblia officinalis* G. and used as a natural mordant alone and in combination with metal mordant namely copper sulfate for dyeing on cotton and silk fabrics using natural dyes. The color strength, color-coordinates, wash and light fastnesses were also evaluated for cotton and silk fabrics with and without mordanting. The pre-mordanted cotton and silk fabrics on dyeing gave better color strength, wash and light fastnesses than those dyeing obtained without mordanting [10]. Adeel et al. studied the utilization of food products (Saffron and madder) as a source of dye for woolen yarn. It is found that, in the case of Saffron, irradiation of yarn for 6 min gives high color strength by dyeing for 45 min in the presence of 2% of salts using un-irradiated dye bath of pH 1, whereas for madder, 4 min is optimal irradiation time for extract to dye irradiated yarn for 25 min in the presence of 6% of salts using the irradiated dye bath of pH 1 [11]. Zia et al. investigated the exploration of coloring potential of neem bark under the influence of ultrasonic treatment. It is found that good color strength is obtained when tannin from bark is extracted in acidified methanol medium and is used to dye irradiated cotton fabric [12]. Haji et al. used *Berberis vulgaris* L. wood as an agricultural waste for dyeing and functional finishing of cotton. The dyed sample prepared under the optimum conditions of crosslinking showed good wash and light fastness

properties besides very good antibacterial activity against gram-negative and gram-positive bacteria [13]. Zuber et al. studied the utilization of bio-mordants to make natural coloring process more eco-friendly is gaining worldwide fame, whereas the addition of microwave treatment has added value to the extraction of colorant under mild conditions. It is found that microwave radiation for 2 min. to acidified methanolic extract of 2 pH obtained from 6 g powder has given good color strength in irradiated silk when it is used to dye at 75°C for 65 min [14]. There is a great deal of research on the natural dyeing of yarns. Various natural components such as neem bark (*Azadirachta indica*) [15], Arjun bark (*Terminalia arjuna*) [16], and cochineal-based anthraquinone dyes [17] were used for green dyeing of fibers and fabrics. The results show that bio-components (dye and mordant) could be optimized conditions to make the process greener and sustainable. Singh et al. investigated Sustainable method of functional dyeing of wool. Simultaneous dyeing and functionalization of wool applied by kapok flower extract and bio-mordant. The pre-mordanted wool displayed enhanced color values and improved fastness ratings [18]. Kadam et al. used Rice straw as source of biomolecules which can be utilized in functionalization of wool yarns. The results showed that dyeing with rice straw extract in the presence of inorganic metal salts produces different elegant hues with acceptable fastness properties [19]. Sheikh et al. used pineapple peel extract (PPE) for simultaneous dyeing and multifunctional finishing of wool fabric. It was confirmed that wool fabric can be dyed using pineapple peel extract without mordants; however, additional color strength can be gained using pre-mordanting. The dyed fabrics showed light shades with good fastness properties along with excellent functional properties [20].

In this work, we investigated the effect of presence biomordant as the green method for dyeing wool carpet yarns as model yarns. We assessed the structure of various sorts of natural sources and recognized that oak is fertile in tannin, hence we applied it in dyeing wool by the aid of Iranian madder and weld natural dyes. Through a chronological procedure, we first extracted oak as biomordant and applied in dyeing of wool yarns with selected natural dyes. The color strength, color coordinates, wash, light and rubbing fastnesses of wool yarns dyed in the presence of oak extracts are evaluated with and without mordanting.

2. Experimental

2.1. Materials and instrumentation

100% wool carpet yarns (215 tex/2 fold) are natural protein-based yarn, which have been used in Persian carpets or rugs for thousands of years. Madder is the main source of natural dyes obtained from vegetable for wool dyeing. Natural dyes (Madder and weld) and oak fruit as natural mordant were obtained from underbrush grown in the north and center of Iran. The madder and weld extracted and fully characterized in our pervious paper [5].

For the analysis of chemical bonding between ingredients, FTIR-ATR spectra were collected on a spectrometer (Perkin Elmer, USA) equipped with ZnSe crystal to help qualitative evaluation of changes in the main characteristic group absorption bands in the mordanting wool as well as dyed wool yarns. The FTIR-ATR spectra were recorded using a single reflection horizontal ATR accessory with a ZnSe crystal fixed at an incident angle of 45°.

In order to measure the reflectance spectra of the dyed yarns within 380 to 750 nm at 10 nm intervals, the Color Eye 7000A spectrophotometer from Gretag Macbeth was used. The measurement geometry was d/8 while the specular component and the UV content of the standard illuminant were included. Then, the colorimetric coordinates of samples over the CIEXYZ, CIELAB and CIELCH color spaces were computed under D65 standard illuminant and CIE1964 standard colorimetric observer.

Heidolph rotary evaporator (Hei-VAPValue digital, Germany) was used to get the supersaturated solution extraction. Light fastness was performed on a Xeno test 150s Hanau according to ISO 105-B02:2014(en). Wash and rub fastness properties of the dyed yarns were determined according to ISO 105-C10 2006(en) and ISO 105-X12 2016 standards.

2.2. Extraction procedure of oak

The oak fruit was dried and grounded in mixer and stored in glass bottle at room temperature (25-27 °C). Fine powder (50 g) was extracted with 200 mL of ethanol on shaker for 20 h. The total extract was heated to boil and allowed to stand overnight and filtered again. The clear filtrate was concentrated under vacuum using rotary evaporator instrument and the semi-solid mass obtained was diluted with distilled water. The precipitate was subsequently dried in an

oven and collected in powder form.

2.3. Mordanting procedure

Before mordanting, wool yarns were washed by non-ionic soap solution containing 2 g/L of soap at 60 °C for 20 min to enhance surface wettability. The wool yarns were mordanted by meta-mordanting method in water keeping, L:R ratio 1:40. Wool yarns were treated with a mordant solution of oak extraction with 5, 10 and 20% of weight of yarns (owf).

2.3. Dyeing of wool

Dyeing of wool yarns was carried out in a solution media using L:R ratio of 40:1 at 5, 10, 20 and 40 % (o.w.f %) of the weld and madder as natural dyes. Figure 1 illustrated the dyeing diagram of wool yarns. The wash, light and rubbing fastness properties of the dyed yarns were measured according to ISO105-C06:2010, ISO105-B02:2014 and ISO105-X12:2016 protocols, respectively.

3. Results and Discussion

Reseda with the common name dye's weld is a member of the flavonoid group, while madder having the common name of *Rubia argyi*, belongs to the Rubiaceae family. The common name "oak" also appears in the names of species in related genera, notably *Lithocarpus* (stone oaks), as well as in those of unrelated species such as *Grevillea robusta* (silky oaks) and the *Casuarinaceae* (she-oaks). Oaks have spirally arranged leaves with lobate margins in many species; some have serrated leaves or entire leaves with smooth margins. In spring, a single oak tree produces both male flowers (in the form of catkins) and small female flowers. Oak wood has a density of about 0.75 g/cm³ (0.43 oz/cuin) creating great strength and hardness.

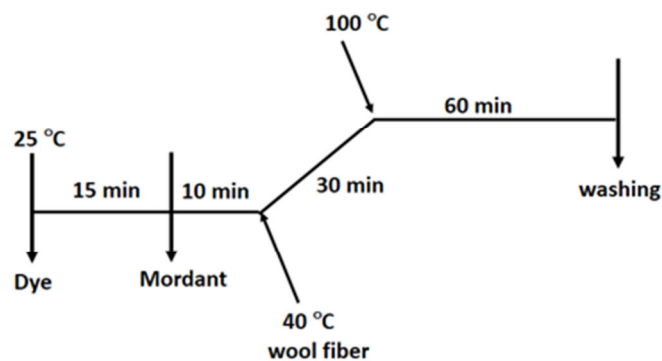


Figure 1: Dyeing diagram of wool yarns.

The wood is very resistant to insect and fungal attack because of its high tannin content. It also has very appealing grain markings, particularly when quarter sawn. Oak planking was common on high status Viking long ships in the 9th and 10th centuries [21, 22].

The FT-IR ATR spectra of washed wool yarn and mordanted dyed wool yarns are represented in Figure 2. Infrared spectra of the wool yarn indicated absorption peaks assigned to peptide bonding, as reference structure one may expect from wool [23]. Wool yarns are hygroscopic and the most hydrophilic

textile yarn. The standard regain is around 16-18% water. The actual regain of purified wool is quite sensitive to traces of residual impurities and any chemical modification of the protein yarn [24]. The N-H and C=O bonding stretching appeared at 3273 and 1630 cm^{-1} , respectively. All characteristics peaks of the mordanted wool yarns are similar to those of wool yarns with low intensities confirming the formation of a complex between mordant molecules and wool yarns [25]. The FT-IR spectra of dyed wool yarns show that all C-N peaks are disappeared due to an interaction between yarn, mordant and dye molecules.

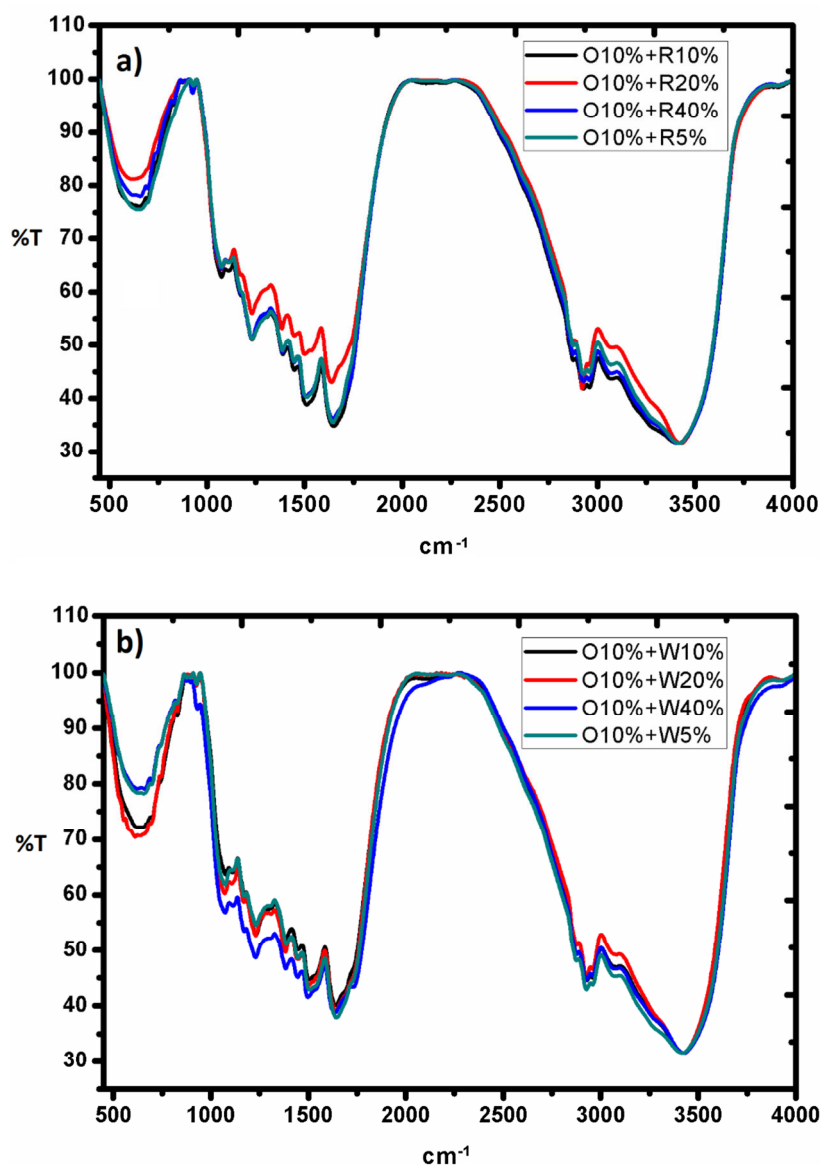


Figure 2: FT-IR of the dyed yarns with a) madder and b) weld.

Mordanting has an important effect on the color strength values and fastness properties of the dyed yarns. It is believed that mordanting process increases the interaction between dye molecules and yarn through the formation of chemical complexes [26]. Mordants not only give the dye an affinity, but in many cases, they produce different colors and improve the fastness of a dye. There are many plants which may yield a brilliant and pleasing color but fades easily, unless fixed by using a mordant. There are different fading such as light, washing, and perspiration. Three mordanting methods can be used for dyeing for the sake of effectiveness of process, including pre-mordanting (on-chrome), meta-mordanting (meta-chrome) and post-mordanting (after-chrome) [27, 28]. In this study, meta-mordanting method

was considered. Figure 3 shows K/S values at the wavelength of 400 nm for dyed wool in meta-mordanting. The results show that the K/S value is remained almost unaffected by 20 % o.w.f. Hence, this concentration was considered for further evaluations.

The reflectance spectra of dyed wool yarns with 20% concentration of Madder in the presence of different concentrations of natural mordant over the visible wavelengths from 360 to 750 nm are shown in Figure 4. According to Figure 4, the dyed wool yarns show various behaviors over the short and long wavelengths within the visible area. To more clarify the colorimetric behavior of wool yarns dyed with madder in the presence of natural mordant, their colorimetric characteristics will be presented and described.

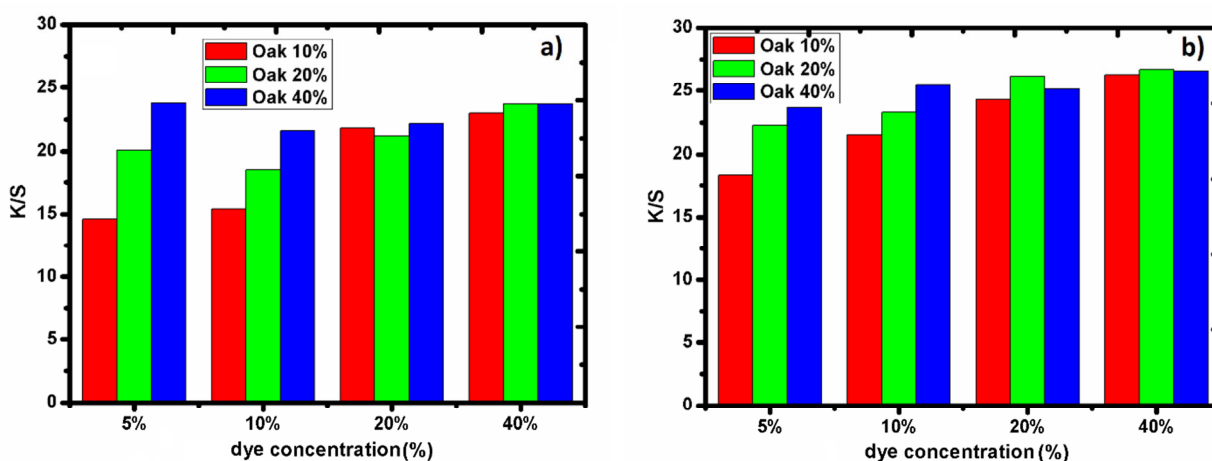


Figure 3: K/S value of dyed yarns by a) madder and b) weld.

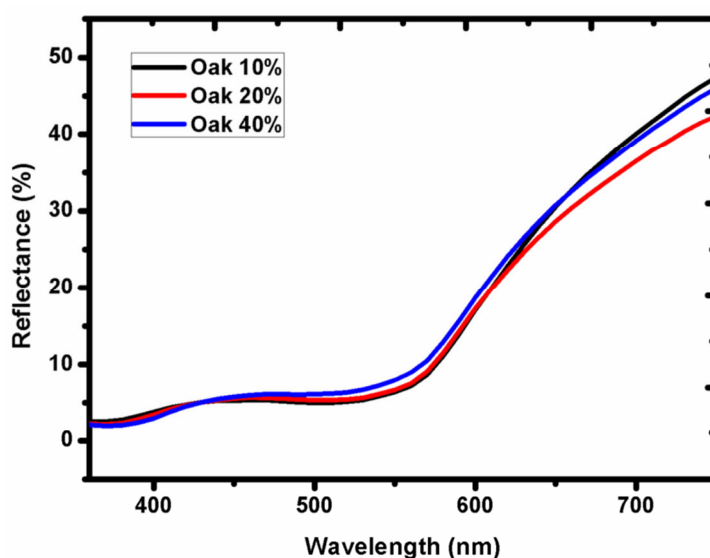


Figure 4: The reflectance spectra of wool yarns dyed with madder in the presence of natural mordant.

Figure 5 indicates the reflectance spectra of wool yarns dyed with 20% o.w.f weld concentration in the presence of different concentrations of natural mordant. The x axis shows the visible wavelengths from 360 to 750 nm. As Figure 5 shows, there is no significant difference between the reflectance spectra of dyed wool yarns treated with 20 and 40% o.w.f Oakes natural mordant. In fact, it seems that the application of 20% o.w.f natural mordant results in optimum spectral behavior of dyed wool yarns and no excess Oak is needed to be introduced.

Results of color depth, i.e. K/S values of dyed wool yarns with natural dyes, namely madder and reseda, obtained using natural mordants are given in Tables 1 and 2. It can be seen that K/S values of the wool yarns meta-mordanted with natural mordant and the ones dyed with natural dyes are higher than those of natural dyed wool yarns. It can be concluded that the improvement of the dyeing depth in the presence of oak contributed to the natural mordant with phenolic hydroxyl group to form a complex in assist with dye molecules leading to goodness of fixation of dye onto the yarns [29, 30].

On the other hand, Table 1 indicates the colorimetric specifications of wool yarns dyed with madder in the presence of natural mordant (Oak). According to Table

1, by increasing the Oak concentration, the redness attribute (a^*) [31, 32] of dyed yarns decreases in contrast to their yellowness tint factor (b^*) [31, 32]. Considering the hue angles shift toward the yellow axis (b^*) in the Hunter color space, the fact of changing the tint attribute of dyed yarns by increasing the concentration of applied natural mordant is proved. Regarding the reverse changes observed for both a^* and b^* parameters, no significant variations in chroma attribute (C^*) of dyed wool yarns is expected, reasonably. Besides, the lightness attribute of dyed yarns decreased expectedly while the concentration of natural dye (madder) increased.

The colorimetric specification of dyed wool yarns with natural dye (Weld) in the presence of natural mordant (Oak) is shown in Table 2. As Table 2 shows, the lightness attribute (L^*) of dyed wool yarns decreases by increasing the concentration of Oak while the outcome was not observed in the case of dye concentration enhancement. In other words, by increasing the concentration of the applied natural dye, the lightness parameter does not show any evident ascending or descending trend. On the other hand, the yellowness attribute (b^*) of dyed wool yarns shows the strange variations while the concentration of applied natural mordant increases.

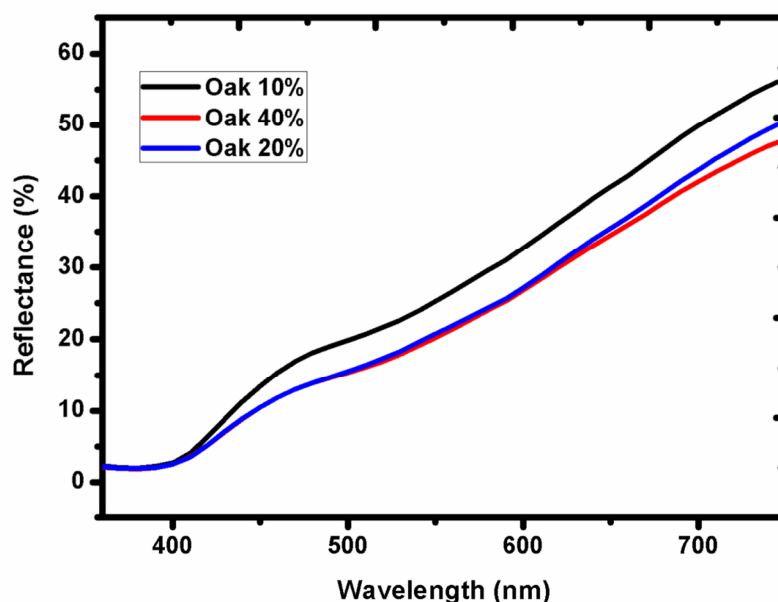


Figure 5: The reflectance spectra of wool yarns dyed with weld in the presence of natural mordant.

Table 1: The K/S values and the colorimetric characteristics of dyed wool yarns with madder in the presence of natural mordant (Oak).

Madder (%)	Oak (%)	K/S (400 nm)	Colorimetric characteristics				
			L*	a*	b*	C*	h°
5	10	14.59	48.48	17.49	17.36	24.64	44.80
10	10	15.42	43.88	22.51	15.84	27.52	35.13
20	10	21.80	37.20	26.84	17.07	31.81	32.45
40	10	22.99	31.61	28.07	17.51	33.08	31.96
5	20	20.11	46.77	15.89	18.64	24.50	49.56
10	20	18.50	43.25	20.99	18.60	28.04	41.55
20	20	21.18	37.57	25.12	16.76	30.20	33.71
40	20	23.71	32.31	27.68	16.77	32.36	31.21
5	40	23.79	47.92	15.07	20.95	25.80	54.28
10	40	21.60	44.61	18.47	20.46	27.56	47.92
20	40	22.18	39.62	23.96	19.24	30.73	38.77
40	40	23.71	34.10	27.28	18.55	32.98	34.22

Table 2: The K/S values and the colorimetric characteristics of dyed wool yarns with weld in the presence of natural mordant (Oak).

Weld (%)	Oak (%)	K/S	Colorimetric characteristics				
			L*	a*	b*	C*	h°
5	10	18.31	57.48	7.95	20.90	22.36	69.18
10	10	21.55	56.12	8.42	22.29	23.82	69.30
20	10	24.32	58.08	7.27	25.67	26.68	74.20
40	10	26.28	57.70	6.53	29.26	29.98	77.43
5	20	22.29	53.19	8.26	21.78	23.29	69.24
10	20	23.29	54.33	7.94	23.32	24.63	71.19
20	20	26.15	52.88	8.16	24.92	26.22	71.87
40	20	26.69	54.10	7.59	27.67	28.69	74.66
5	40	23.67	53.20	8.29	23.45	24.87	70.53
10	40	25.42	51.23	8.62	24.56	26.03	70.65
20	40	25.10	53.30	8.14	25.54	26.80	72.32
40	40	26.57	52.81	8.03	27.49	28.64	73.72

As Table 2 shows in the application of 10 to 20% concentration (o.w.f) of Oak, the yellowness attribute of dyed wool yarns increases while by 40% concentration (o.w.f) of natural mordant, the yellow

tint effect decrease. Besides, as it is expected, the yellowness property of dyed wool yarns increases with the increase of the dye concentration. Meanwhile, the redness factor (a^*) of the wool yarns dyed with weld in

the presence of Oak does not show a clear trend by increasing the concentration of the applied natural dye/mordant. Oak is a famous plant fertile in tannin and suitable biomordant for dyeing. Polypeptide structures present in wool yarns provide the matter with so many active sites containing NH_2 and COOH functional groups that bridge the dye molecules with the yarn [33]. Correspondingly, tannin-based biomordants with 5% Babul, which were applied in wool dyeing, resulted in K/S value of 4.14 [34]. The use of Gallnut as tannin-based in pre- and post-mordanting method resulted in K/S values of 16.09 and 14.66, respectively [35].

The wash, light and rubbing fastness properties of the dyed wool yarns were measured according to ISO105-C02:1998(E), ISO105-B02:1994(E) and ISO105-X12, respectively. All data of fastness properties are summarized in Tables 3 and 4. The data suggest that washing fastness of samples was very good (4-5 to 5). The staining of three green dyed samples revealed good fastness with grade 5. The dyed

yarns showed good light fastness in the presence of mordants. However, the light fastness increased one rating for all dyed yarns experienced meta-mordanting with a mixture of natural and mineral mordant. Similar results have been reported by Prabhu et al. [29] for light fastness of dyed wool, cotton and silk. All dyed wool yarns showed good rubbing fastness due to the ability of the dye molecules to self-associate through intermolecular hydrogen bonding because of the presence of primary amino grouping dye molecules [36]. Such a green process based on green ingredients enables clean productions of Iranian carpet using locally available green compounds. Yusuf et al. evaluated eco-dyeing of wool with madder extract in the presence of Acacia catechu as biomordant. It was also claimed that Shades on pre-mordanting wool of red tones with good to excellent color fastness properties were achievable [37, 38]. In a similar manner, the results of this study indicate that such a clean dyeing process that contains green ingredients allows for clean production of Iranian carpet.

Table 3: Fastness properties of the dyed wool yarns with madder.

Madder (%)	Mordant (%)	Light fastness	Wash fastness		Rubbing fastness	
			Change	Staining	Change	Staining
5	-	2	2	4	4-5	4-5
10	-	2	2	4	4-5	4-5
20	-	2	2	4	4-5	4-5
40	-	2	2	4	4-5	4-5
5	10	4	4	4-5	4-5	5
10	10	4	3-4	4	4-5	5
20	10	4	3	3-4	4-5	5
40	10	5	3	3-4	4-5	5
5	20	4	4-5	4-5	4-5	5
10	20	4	4	4	4-5	5
20	20	4	4	4	4-5	5
40	20	5	3-4	3-4	4-5	5
5	40	4	4-5	5	4-5	5
10	40	4	4	5	4-5	5
20	40	4	4	4-5	4-5	5
40	40	5	4	4-5	4-5	5
5	1% Alum	5	5	5	4-5	4-5

Table 4: Fastness properties of the dyed wool yarns with weld.

Weld (%)	Mordant (%)	Light fastness	Wash fastness		Rubbing fastness	
			Change	Staining	Change	Staining
5	-	2	2	4	4-5	4-5
10	-	2	2	4	4-5	4-5
20	-	2	2	4	4-5	4-5
40	-	2	2	4	4-5	4-5
5	10	4	4	4-5	4-5	5
10	10	4	3-4	4	4-5	5
20	10	4	3	3-4	4-5	5
40	10	5	3	3-4	4-5	5
5	20	4	4-5	4-5	4-5	5
10	20	4	4	4	4-5	5
20	20	4	4	4	4-5	5
40	20	5	3-4	3-4	4-5	5
5	40	4	4-5	5	4-5	5
10	40	4	4	5	4-5	5
20	40	4	4	4-5	4-5	5
40	40	5	4	4-5	4-5	5
5	1% Alum	5	5	5	4-5	4-5

4. Conclusions

A clean procedure involved natural components being treated in a green dyeing process in the production of green Iranian carpet was practiced in this work. A relatively high K/S value was attained when oak extract was utilized alone and in combination with alum green mordant in dyeing wool with natural dyes. In ATR FT-IR spectra used to evaluate chemical bonds between the mordants and yarns, C-N peaks disappeared due to strengthened interactions between the yarn, mordant and dye molecules. In addition to the

colorimetric attributes of dyed wool yarns influenced by natural mordant, the use of green mordanting enhanced the wash, light and rubbing fastness properties. Such a green procedure seemed promising because of the abundance of the local natural dyes, which allows for clean production of Iranian carpet.

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