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Dyeing quality of walnut shells on polyester and polyester/viscose blended fabrics

Ferda Eser[a], Ebru Aktas & Adem Onal
Natural Dyes Investigation & Application Center, Gaziosmanpasa University, 60240, Tokat, Turkey

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This paper reports an investigation on the dyeing efficiency of walnut (*Juglans regia*) shells for dyeing of polyester and polyester/viscose blended fabrics. Various extraction conditions, such as material - liquor (M : L) ratio, extraction temperature, extraction time and *p*H have been studied in order to obtain highest color depth. Optimal production of natural dyes from walnut shells with extraction is achieved at 80°C for 75 min keeping the M : L ratio as 1:30 at *p*H 2. The effects of dyeing time, mordant and dyeing method are investigated in terms of color strength (*K*/*S*). Optimum dyeing with *Juglans regia* shells is achieved with meta-mordanting method for 90 min treatment using $AlK(SO_4)_2 \cdot 12H_2O$ (alum) mordant for polyester fabric. For polyester/viscose blended fabric, best dyeing conditions are achieved in the presence of $FeSO_4 \cdot 7H_2O$ mordant, using meta-mordanting method for 90 min treatment. The fastness properties are evaluated with respect to light, washing and rubbing. The results show that walnut shell extract with metal mordants has potential applications on polyester and polyester/viscose blended fabrics.

Keywords: Dyeing, *Juglans regia*, Polyester, Polyester / viscose blend, Walnut

1 Introduction

Natural dyes have been used to dye carpets, rugs and clothes and are obtained from various parts of plants such as roots, stems, barks, leaves and flowers since ancient times. People have also used seashells and some insects in the coloration of these materials[1]. Natural dyes and pigments can be considered as an important alternative to harmful synthetic dyes and generally give soft and lustrous pastel colors[2]. Hue of the color can be varied, depending on some factors such as concentration of dye, mordant type, dyeing method, dyeing time, temperature and *p*H.

It is known that synthetic dyes are derived from petrochemical sources that results in chemical substances which are hazardous to human health and environment. Thus, there is a growing interest to natural dyes due to their biodegradable, less toxic and eco-friendly properties in recent years[3,4]. Therefore, the necessity of low cost natural dye production encouraged the people to use dyestuff containing wastes such as food, beverage and aerial parts of plants[5]. The huge amount of such wastes can be evaluated as an important raw material for textile dyeing industry[6].

The introduction of natural dyes into modern textile dye houses requires some aspects, such as product classification, standardized quality in terms of color depth and shade of the dyeing[7]. Hence, it is important to determine efficient extraction conditions in order to extract the colorants with high yield. Additionally, determination of optimum dyeing conditions is of considerable interest of textile dyers so as to utilize the natural dye source efficiently in the dyeing of fibres.

Walnut (*Juglans regia* L.) belongs to the Juglandaceae family and is mostly grown in un-reclaimed and poor soil. Various parts of walnut tree are used for dyeing and tanning[8]. There are number of studies about natural dyeing for different types of fibres, such as cellulosics, lingo cellulosics, proteinic and synthetics[9-12]. Bhattacharya and Lohiya[13] investigated the dyeing of cotton and polyester fibres with pomegranate rind, catechu, nova red and turmeric using high temperature high pressure (HTHP) dyeing method. Gupta and Gulrajani[1] studied the kinetics and thermodynamics of juglone on five fibrous substrates, namely wool, human hair, silk, nylon, and polyester. Mirjalili and Karimi[14] investigated the extraction and characterization of natural dye in walnut shells and application of its dyeing on polyamide fibres with respect to antibacterial properties. Ghaheh *et al*.[15]

[a]Corresponding author.
E-mail: ferda.eser@amasya.edu.tr
Present address: Suluova Vocational Schools, Amasya University, 05100, Amasya, Turkey.

evaluated the effect of different mordants on the dyeing potential of wool fabrics in terms of color strength and antibacterial activity. Tutak and Korkmaz[16] investigated the usage of walnut shell in the dyeing of organic cotton fabrics. Silk dyeing potential of walnut husks was evaluated by Mirjalili[17]. It is reported that juglone (5-hydroxy-1,4-naphtalenedione) is the main coloring component which is found in walnut leaves[18,19], husks[20-22], inner root bark[23] and stem bark[24].

It is important to develop a knowledge base on dye chemistry of juglone and its effects on dyeing processes. However, it is important to study different types of natural dyes and fibres/fibre combinations in order to obtain high color yield in an economical way. Appropriate and standardized dyeing techniques need to be adopted in order to enhance commercial use of natural dyes[14-24]. Therefore, the present study focuses on the determination of optimum extraction and dyeing conditions for *Juglans regia* shells and investigation of the dyeing potential of the extracted natural dyes on dyeing of polyester and polyester/viscose blended fabrics. For this purpose, various extraction conditions are applied for walnut shells in order to obtain the highest color depth. Different factors effecting dye ability such as dyeing time, mordant type, dyeing method are also investigated.

2 Materials and Methods

2.1 Fabrics

Plain weave polyester fabric having 230 ends/dm, 314 picks/dm and weighing 70 g/m^2 on the average; and polyester/viscose blended fabrics (60/40) having 260 ends/dm, 190 picks/dm and weighing 190 g/m^2 were used for the natural dyeing of walnut shell.

2.2 Chemicals

All chemicals were analytically graded. The following mordants were used without further purification: stannous chloride dihydrate ($SnCl_2{\cdot}2H_2O$, Aldrich), alum ($AlK(SO_4)_2{\cdot}12H_2O$, Aldrich), potassium dichromate ($K_2Cr_2O_7$, Aldrich), ferrous sulphate heptahydrate ($FeSO_4{\cdot}7H_2O$, Aldrich) and copper sulphate pentahydrate ($CuSO_4{\cdot}5H_2O$, Aldrich). Acetic acid (CH_3COOH) and NaOH were used for *p*H adjustment and supplied from Merck (Germany).

2.3 Test Methods

Dyeing was performed with a Termal HT 610NHT model dyeing machine. For washing and crocking fastness Atlas Weather-ometer equipment and 255 model Crock-meter were used separately. Color characteristics of dyed samples were determined with Premier Colorscan SS 6200A Spectrophotometer using illuminant D65 and 10° standard observer in terms of CIELab values (L*, a*, b*, C*) and color strength (*K*/*S*). UV-visible spectrum of walnut shell water extract (50 mg/L) was recorded on a Perkin Elmer Lambda 35 model spectrophotometer.

Color strength in visible region of the spectrum (400-700 nm) was calculated based on the Kubelka-Munk equation.

Dyed samples were exposed to bare sunlight for 200 h. After the exposure, light fastness ratings of dyed samples were given on 1-8 grey scale. For determination of wash fastness, dyed samples were heated in a soap solution for 30 min at 45°C, keeping liquor - material ratio as 50:1. Wash fastness values were established according to ISO 105-C06 standards. Dry and wet crock fastnesses of dyed fabrics were tested according to ISO 105-X12 method[25].

2.4 Optimization of Extraction Conditions

Shells of *J. regia* were dried at room temperature, crushed and then soaked in distilled water. After extraction, the extract was filtered through filter paper and the filtrate was used in the dyeing experiments. Extraction of natural dye with distilled water from walnut shells was performed using various material - liquor (M : L) ratio, such as 1:10, 1:20, 1:30, 1:40 and 1:50. The extractions were performed at different temperatures (20-100°C) using 20°C increase. Various extraction times such as 30, 45, 60, 75, 90 and 120 min were also used in terms of color strength. The effect of *p*H was also studied. For this purpose, before dyeing processes, *p*H values of the dye solution were adjusted to 2, 4, 6, 8 and 10 with CH_3COOH or NaOH solution. Polyester and polyester/viscose blended fabrics were dyed separately with each extract. Color strength (*K*/*S*) values were further utilized to optimize the dyeing conditions.

2.5 Optimization of Dyeing Conditions

Polyester and polyester/viscose blended fabrics were dyed with optimized walnut extract at different durations, such as 30, 60, 90 and 120 min. To determine the effect of mordant, stannous chloride, alum, potassium dichromate, ferrous sulphate and copper sulphate were used. Effect of dyeing methods (pre-mordanting, meta-mordanting and post-mordanting) were also investigated. Dyeing of polyester fabrics was performed at 130°C. Polyester/viscose blended fabrics were dyed at two different temperatures, namely 130°C and 75°C respectively (130°C for polyester and 75°C for viscose fabrics).

For pre-mordanting method, polyester fabric was first immersed into 0.1 M mordant solution (100 mL) and heated for 30 min at 130ºC. After the cooling of the sample, it was rinsed with distilled water and put into the dye-bath solution (100 mL). It was dyed at 130ºC for 1 h. At the end, the dyed material was removed, rinsed with distilled water and dried.

In meta-mordanting method, both mordant (in solid form that is equal to 0.1 M mordant solution) and the dye residue were transferred to a conical flask and polyester fabric was poured into the mixture. Then the mixture was heated at 130ºC until 1 h. Then it was cooled and washed with distilled water, squeezed and finally dried.

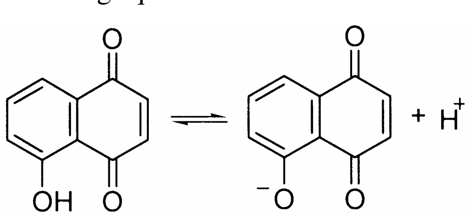
For post-mordanting method, the non-colored polyester material (1 g) was firstly treated with the dye solution for 1 h at 130ºC. Then the material was cooled, washed twice with distilled water and poured into 0.1 M mordant solution (100 mL). It was heated for 30 min at 130ºC. After the end of the process, the dyed fabrics were rinsed with distilled water.

Same mordanting methods were applied to polyester/viscose blended fabrics with small modifications in heating temperature. All heating processes were performed at 130°C followed by 75°C for the dyeing of polyester/viscose blended fabrics,

3 Results and Discussion

3.1 UV-Visible Spectrum

The UV spectrum of the crude walnut extract is given. Absorption of walnut dye was observed in the UV-C region (200-290 nm). The spectrum shows absorption at 263 nm which may be attributed to the electronic transitions of benzene and its derivatives[26,27]. UV-VIS spectra of walnut shells was examined in previous studies[28,29]. Strong signals have been obtained between 240-290 nm which complies with naphthtoquinones spectra, as shown in following equation:



… (1)

Ionized and unionized species are involved in *p*H-dependent equilibrium (Eq. 1). The reason for ionized the different color is protonation or deprotonation of the dye molecule causing a different electron configuration. The visible color change of the dye is mostly based on a ring opening of the dye molecule upon deprotonation, or on a tautomerism, as tautomers have different colors and tinctorial strengths[30]. The tautomer excess in acidic solutions is therefore more deeply colored than the ionized species associated with high *p*H values.

3.2 Optimization of Extraction Conditions

Figure 1 shows the *K*/*S* values of dyed fabrics under different conditions. Various M:L ratios are studied in order to find out the best color depth. Maximum color strength is obtained at 1:30 (M:L) for walnut shell water extract [Fig. 1 (a)]. It is observed that *K*/*S* values of dyed fabrics decrease after 75 min [Fig. 1 (b)]. This result may be explained by the fact that the decrease in the absorbance of the extract is caused with the prolonged extraction time which may lead to dye degradation. Effect of extraction temperature is also investigated. Previous studies reveal that the extraction efficiency of natural dye depends on temperature. Hence, it is important to determine optimum extraction temperature. It is clear that *K*/*S* value keeps increasing with increase in extraction temperature up to 100°C and attains the highest value at 100°C [Fig. 1 (c)]. This can be attributed to the decreasing amount of colorant uptake by fabric with increasing temperature which indicates an exothermic process[30-33]. Effects of different *p*H values between 2 and 10 are examined and it is concluded that *p*H of the dye bath have considerable effect on the color strength of dyed fabrics. This can be explained by the correlation between the dye molecule and the fabric. Best performance on dyeing of polyester and polyester/viscose blended fabrics is achieved best at *p*H 2 [Fig. 1 (d)]. Lower color strength values of dyed fabrics with higher *p*H values (*p*H>6) might be due to the destruction of the conjugate structure of the natural dyes[34,35].

Considering overall results, best extraction of walnut shells is performed at 100°C for 75 min, keeping the M:L ratio as 1:30 at *p*H 2. Walnut shell extract is prepared freshly before dyeing experiments.

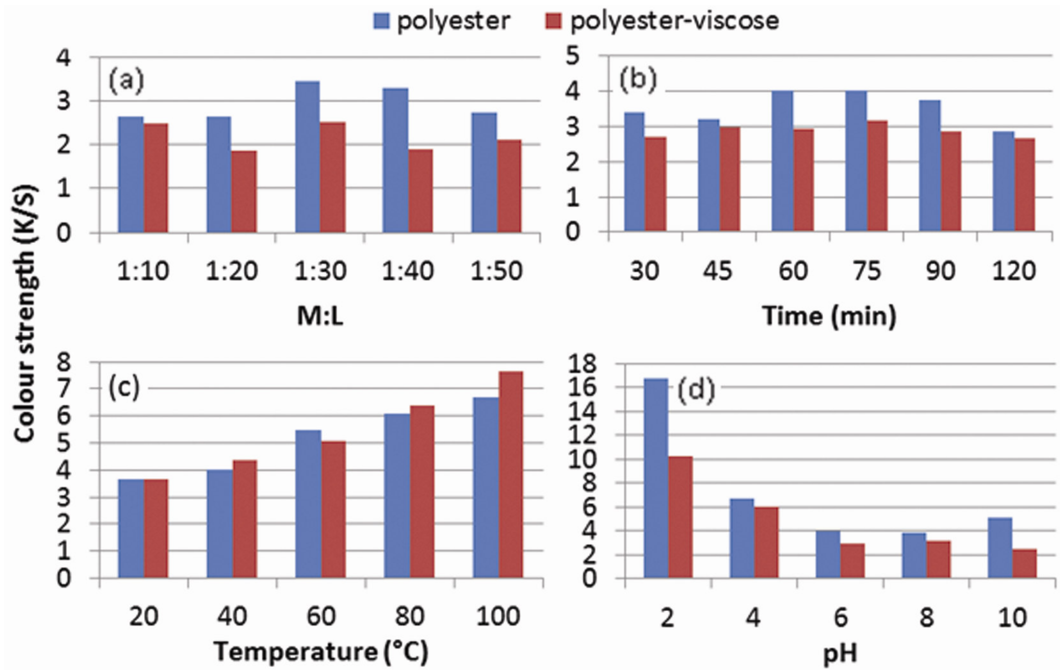


Fig. 1—*K*/*S* values of dyed polyester and polyester/viscose blended fabrics with walnut shell extract for different extraction variables [(a) M:L ratio, (b) extraction time, (c) dyeing temperature, and (d) *p*H]

3.3 Optimization of Dyeing Conditions

3.3.1 Effect of Dyeing Time

Effect of dyeing time on *K*/*S* value is demonstrated in Fig. 2. It is clear that there is no considerable difference between the color strength values of dyed polyester/viscose blended fabrics which are dyed at different time durations. Sharp increase in *K*/*S* value is observed for polyester fabric at 90 min dyeing. Color strength shows decrease in time 120 min. This decline may be attributed to the shift in equilibrium of dyestuff from fabric to the dye bath solution at a longer dyeing time[35].

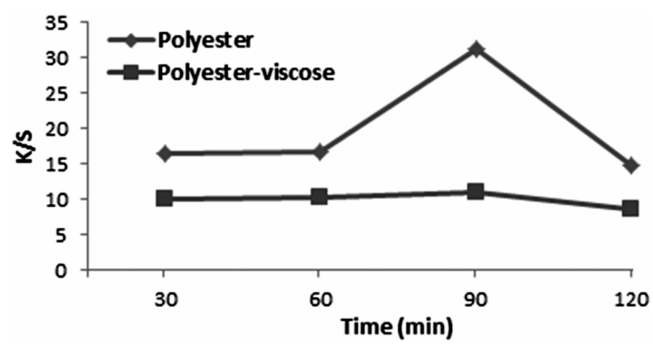


Fig. 2—Effect of dyeing time on color strength of dyed polyester and polyester/viscose blended fabrics [mordant– $AlK(SO_4)_2{\cdot}12H_2O$, dyeing method– meta-mordanting for polyester fabric; and mordant– $FeSO_4{\cdot}7H_2O$, dyeing method– meta-mordanting for polyester/viscose blended fabric]

3.3.2 Effect of Mordant

Influence of various mordants, such as stannous chloride, alum, potassium dichromate, ferrous sulphate and copper sulphate, has been investigated for polyester and polyester/viscose blended fabrics (Table 1). The highest *K*/*S* value (22.8) is obtained in the presence of alum mordant for polyester fabric. The results also indicate that some mordants (alum, copper sulphate and ferrous sulphate) help to increase color strength of dyed samples while others (stannous chloride and potassium dichromate) give rise to decrease when compared with undyed sample. It is found that *K*/*S* values increase in the order of Al>Cu>Fe>control>Sn>Cr for polyester; and Fe>Cu>Al>control>Sn>Cr for polyester/viscose blended fabrics. It is known that metal salts of mordants are water soluble and thus they act as bridge between the fibre and the dyestuff at molecular level[36-38]. The results of mordant effect on polyester/viscose blended fabrics are well-matched with previous study carried out by Mirjalili & Karimi[14]. They studied the effect of mordants including alum, copper sulphate and ferrous sulphate on the dyeing of polyamide with the shells of walnut. They found that the effect of mordants is increased in the order of Fe>Cu>Al based on their *K*/*S* values.

Table 1—Color characteristic values of the polyester and polyester/viscose blended fabrics dyed with walnut shell with different mordants

Mordant	Polyester					Polyester/viscose				
	*L**	*a**	*b**	*h*	*K/S*	*L**	*a**	*b**	*h*	*K/S*
Control	37.3	4.4	11.0	68.3	9.9	41.7	4.2	11.5	70.0	7.8
Stannous chloride	63.4	3.0	18.8	81.1	4.3	63.7	3.0	18.6	80.8	4.3
Alum	33.6	4.6	11.1	67.6	22.8	39.7	4.4	12.5	70.5	9.5
Potassium dichromate	89.8	-1.2	5.7	101.9	0.8	86.9	-2.1	14.7	98.2	0.9
Ferrous sulphate	36.4	3.3	10.2	72.1	13.8	36.1	2.4	8.6	74.7	9.8
Copper sulphate	32.3	4.5	10.6	66.9	16.7	36.9	4.0	8.9	65.5	9.6

The results of this study indicate that the type of shades produced and the level of absorption of dye are affected by different mordants whose chemical reaction with the natural dyes depends on the unique structures of the colour components and on the strength of the metal dye coordination complex formed during the dyeing process. Aluminium metal forms weak coordination complexes with the dye, having tendency to form quite strong bonds with the dye but not with the fibre. This is in accordance with the observed highest *K*/*S* values in presence of aluminum[38]. Sn forms the coordinate bonding with the fibre, but does not show the good result. This may be due to some blockage of salt particle between the dye molecule and the fibre.

3.3.3 Effect of Dyeing Method

Polyester fabrics are dyed according to the optimized conditions (i.e. with alum mordant for 90 min). It is observed that the dyeing method has significant effect on color strength of dyed polyester fabric (Fig. 3). Effect of dyeing method is increased in the sequence of meta-mordanting> pre-mordanting>post-mordanting for polyester fabric and meta-mordanting>post-mordanting>pre-mordanting for polyester/viscose blended fabric. In meta-mordanting method, mordant interacts with the fabric to lead unoccupied active sites that provide opportunity for the absorption of natural dye. Thus, more stable fabric-mordant-dye complex is formed.

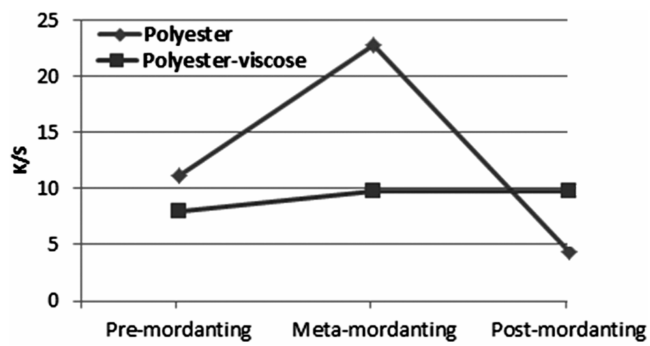


Fig. 3—Effect of dyeing method on the color strength of dyed polyester and polyester/viscose blended fabrics [mordant– $AlK(SO_4)_2{\cdot}12H_2O$, dyeing time– 90 min for polyester fabric; and mordant– $FeSO_4{\cdot}7H_2O$, dyeing time– 90 min for polyester/viscose blended fabric]

3.4 Color Coordinates of Dyed Fabrics

Evaluation of color parameters is performed using CIELab system. Dyeing of two types of fabrics is carried out at optimized conditions except mordant. Results are given in Table 1. Lightness-darkness values of dyed fabrics are symbolized with 'L' and these values vary between 100 and 0, representing white to black; + values of a* and b* indicate redness and yellowness shade respectively. Additionally, - values of a* and b* refer to greenness and blueness color tones respectively. Lightness values of dyed polyester and polyester/viscose blended fabrics are found low (<42), except stannous chloride and potassium dichromate mordants. Mink color and color tones are obtained with pre-mordanting method. Dark brown is acheved in meta-mordanting method while light brown is obtained with post-mordanting method for polyester and polyester/viscose blended fabrics respectively. As shown in Table 1, different mordants not only affect the hue of the color but also color strength of the dyed fabrics.

3.5 Fastness Properties of Dyed Fabrics

Fastness values of dyed fabrics are given in Table 2. Color fastness to washing with soap is very good for both types of fibres. Rub fastness of the dyed samples is determined in both dry and wet form. It is observed that rub fastness values are found higher in the dry form than in the wet form. Additionally, high rub fastness rates are obtained with post-mordanting method both for polyester and polyester/viscose blended fabrics. The light fastness values of the dyed fabrics range between 3 and 5, i.e. moderate

Table 2—Fastness values of the polyester and polyester/viscose blended fabrics dyed with walnut shell extract

Dyeing method	Fabric type	Fastness			
		Wash	Rub		Light
			Wet	Dry	
Pre-mordanting	Polyester	5	4	5	3
	Polyester/viscose	5	4	5	4
Meta-mordanting	Polyester	4/5	4	4	5
	Polyester/viscose	5	4	5	5
Post-mordanting	Polyester	5	4	5	4
	Polyester/viscose	5	5	5	5

to good. Polyester/viscose blended fabrics exhibit higher light fastness values than polyester fabrics (Table 2).

Fastness properties of cotton fabric dyed with walnut bark has already been investigated by Sharma and Grover[8]. We also obtained similar results in terms of rub and wash fastness values. On the other hand they obtained higher light fastness values ranging between 6 and 8 in the presence of alum, copper sulphate and ferrous sulphate mordants[8]. This may be explained by the interaction of the dye molecule and type of the fibre.

3.6 Dyeing Mechanism of Polyester Fabric

Polyethylene terephthalate (PET) or polyester fibres have been widely used in textile industries due to their low production costs, high strength, easy-care properties, high thermostability and chemical resistance[39]. Polyester built up from multiple chemical repeating units linked together by ester (CO-O) groups. Therefore, it is expected that chemical interactions between walnut shell dye and polyester fabric occur between –OH (hydroxyl) group of the dye molecule and oxygen atom of carbonyl group of polyester fabric via H-bonding [Fig. 4(a)]. On the other hand, Fe^{2+} and Cu^{2+} mordants are able to chelate with the dye to form coordinate complexes [Fig. 4(b)]. The coordination numbers of Fe^{2+} and Cu^{2+} are 6 and 4 respectively. So, after the interaction of the metal with the fibre, some coordination sites remain unoccupied. These sites may be occupied with the functional groups of the fibre such as carboxylic groups[40]. As the main constituent of walnut shell is juglone, the predicted mechanism that may occur between polyester fabric and walnut shell extract can be considered (Fig. 4).

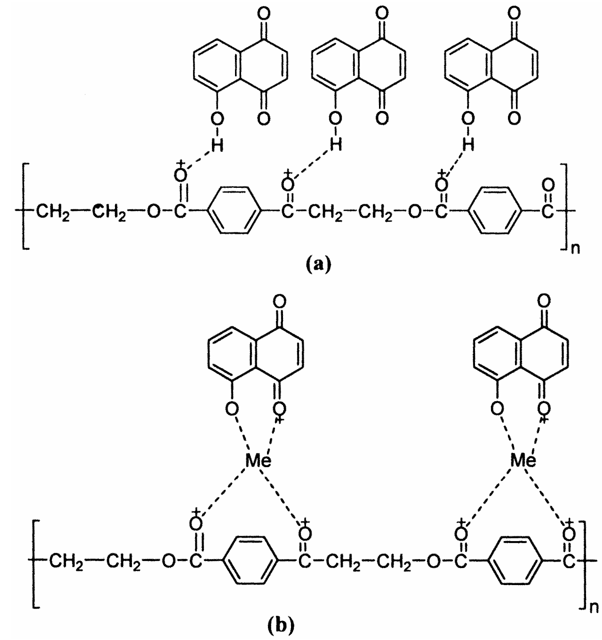


Fig. 4—Proposed mechanism of polyester dyeing (a) unmordanted and (b) in the presence of metal mordant (Me– Cu^{2+}, Fe^{2+}, Al^{3+})

4 Conclusion

This study mainly focuses on the evaluation of walnut shells in the dyeing of polyester and polyester/viscose blended fabrics. Furthermore, optimum extraction and dyeing conditions are determined based on their color strength values. For the extraction of walnut shells, best color depth is obtained under the following conditions: 1:30 M:L ratio, 100°C treatment temperature 75 min extraction time and *p*H 2. Polyester and polyester/viscose blended fabrics are dyed with optimized walnut shell extract. Effect of dyeing parameters such as mordant, dyeing method and dyeing time are also investigated and it is found that highest color strength values are obtained with 90 min dyeing in the presence of alum mordant using meta-mordanting method for polyester fabric. For polyester/viscose blended fabrics, highest *K*/*S* values are achieved with 90 min dyeing, in case of ferrous sulphate using post-mordanting method. The dyed polyester and polyester/viscose blended fabrics show brown and mink hue. It is concluded that walnut shells can be successively used in the natural dyeing of polyester and polyester/viscose blended fabrics to get good color strength and acceptable fastness properties.

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