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Eco-dyeing of Wool Fabrics with Natural Dye Extracted from Leaves of Neem

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Authors' contributions

This work was carried out in collaboration among all authors. All authors read and approved the final manuscript.

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ABSTRACT

More powerful of extraction natural dye from Neem leaves (*Azadirachta* indica) with respect to different dye bath concentrations in the range 20-80 g/L on wool fabrics was the aim of the present work. Also, the effect of artificial daylight up to 160 hours on the wool fabrics was evaluated. The optical properties of the blank and dyed unexposed and exposed fabrics were determined using the spectrophotometric technique. From the obtained reflectance spectra, the CIE tristimulus values, the color parameters, the absorption coefficient, optical band gap energy, extinction coefficient and color strength were calculated as well as the washing fastness of the fabrics was tested. The results noted that wool fabrics were depended on both the dyeing concentration and the exposure to artificial daylight. The locations of blank and Neem dyed wool fabrics unexposed and exposed on the chromaticity diagram were different and confirmed the change in the spectral colors of the fabrics. The obtained changes in the values of the optical parameters may be attributed to the induced change in the molecular configuration and variations in the chemical bonds in the wool fabrics. Also, the results obtained indicate that the natural dye extracted from Neem leaves has good potential in textiles dyeing. Also, excellent colorfastness properties were obtained indicated that the dyed wool fabrics have good washing fastness properties.

Keywords: Azadirachta indica; neem leaves; wool fabrics; spectrophotometeric technique; optical properties.

1. INTRODUCTION

Wool is one of the earliest natural fibers for texts. Wool is a popular natural fiber and very important for human beings to live in the eco-friendly world which exhibits the characteristics of feeling soft. heat retention, and comfortable to wear [1,2]. Wool was usually used in quality texts and handwoven carpets [3]. Wool behaves as an ampholyte because it contains both acidic and basic groups, namely carboxylic and amino groups (expressed as NH₂-P-COOH, where P is wool) [4].

Natural pigments and dyes may be a real alternative in a variety of applications at food, cosmetic, pharmaceutical and textile industries [5]. Natural dyes were extracted from plants, animals, bacteria and fungi. A dye is a colored substance that chemically bonds to the substrate to which it is being applied. This distinguishes dyes from pigments which do not chemically bind to the material they color. Dyes possess color due to 1) they absorb light in the visible spectrum; 2) they have chromophores at least one (color-bearing group); 3) they have a conjugated system, i.e., a structure with alternating double and single bonds; and 4) they exhibit resonance of electrons, which is a stabilizing force in organic compounds. Dyestuff is a substance yielding a dye or that can be used as a dye, especially when in solution. To analyze dyestuffs several points may be viewed. The dyestuff may be considered as any other organic body. The analysis of dyestuff may have reference to the determination of the amounts of the chemical elements present in it. This character of dyestuff analysis has no importance to the dyer or textile chemist in the manufacture and synthesis of dyestuffs. Recently, people have more interest in natural dyes, on the ground of their better biodegradability, compatibility with the environment, lower toxicity and allergic reactions [6,7]. Also, natural dyes were suitable and successfully applied to natural fabrics such as cotton, silk, flax and wool, because of their generally low toxicity, reduced pollution and biodegradable nature [8,9].

Unfortunately, the broad development of natural dyes in dyeing was specified by their high molecular mass which disfavors their penetration into the inner wool fiber, resulting in the decrease adsorption of dyes on the fibers and increase

desorption of dyes from the fibers. Nowadays, dyeing mordants have been used to improve dye on wool fiber and its dyeing fastness, resulting in the significant improvement of coloration and fastness [10].

Azadirachta indica was commonly known as "Neem", "Nimtree" and "Indian Lilac". It belongs to the Meliaceae botanical family and being abundantly available [11]. *Azadirachta indica* is one of two species in the genus Azadirachta and is native to India and the Indian subcontinent. Neem is a fast-growing tree and can grow in many different types of soil. The pinnate leaves are 20–40 cm long with 20 to 31 medium to dark green leaflets about 3-8 cm long. Uses of purified and fresh Neem leaves used in medicine are [12]: 1) traditional detox benefits of chewing Neem regularly - medicine used in treatment of viral disease, cardiac arrest, fungal disease, malaria, cancer, arthritis, poison, oral issues; 2) directly apply on skin for oil protection and beautiful glow; 3) apply as facial mask by mixing with water/milk etc. mix with body wash; and 4) glows hair too when applied to scalp.

In the present work, study of the optimization of extraction natural dye from Neem leaves with respect to four different dye bath concentrations (20, 40, 60 and 80 g/L) on wool fabrics was investigated before and after exposing to artificial daylight for 160 hours. The effect of the dye bath concentration on the reflectance spectra was followed by using spectrophotometric technique. The CIE tristimulus values $(x_r, y_r \text{ and } z_r)$, the color parameters $(L^*, a^*, b^*, W, C^*, H^*$ and ΔE^*), absorption coefficient (*α*), optical band gap energy, extinction coefficient (K) and color strength (K/S) were determined as well as the washing fastness of the fabrics was tested.

2. MATERIALS AND METHODS

2.1 Materials

Pure wool fabric (100%) of 137 g/m^2 in weight and 0.35 mm in thickness, number of threads in weft direction 25 and in warp direction 30 was supplied from Goldentex Co. (Cairo, Egypt). The fabric was scoured with a solution containing 0.5 g/L sodium carbonate and 2 g/L non-ionic detergent using liquor ratio 1:50 at temperature 60ºC for 15 minutes. Then, the samples were thoroughly washed with water and dried at ambient conditions.

2.2 Extraction and Preparation of the Dye

Dried leaves of Neem plant (*Azadirachta indica*) were used to obtain Neem dye without the application of any mordant in the dyeing process. After crushing the Neem leaves, 50 g were immersed in 500 mL of distilled water and boiled for 1 hour. Double Beam Spectrophotometer with standard illuminant C (1174.83) model V-530 (Shimadzu, Japan) with bandwidth 2.0 nm and of accuracy ±0.05% was used to determine the absorbance of the extract solution after filtration in the range 250-700 nm. Fig. 1 shows the absorption spectrum of the aqueous extract of the Neem leaves. The absorbance value was taken as a measure of Neem dye concentration. As shown, three peaks were presented at 320 nm of absorbance 3.765; at 370 nm of absorbance 3.402; and at 400 nm of absorbance 3.569. Increasing the number of dried leaves from 20 to 80 grams per liter water boiled for 1 hour was accompanied by the increase in color strength and depth in color.

2.3 Dyeing and Fixing Method

The wool fabrics under test were dyed by the preextracted dye in a laboratory dyeing apparatus by applying the conventional exhaust dyeing method [13] and by using a liquor ratio of 1:50 at pH 5, and at temperature 60ºC for 45 minutes for each concentration [14]. Different shades were obtained by using different concentrations (20, 40, 60 and 80 g/L). The

samples were washed with cold water and dried at ambient temperature.

2.4 Measurements

The blank and dyed wool fabrics with different Neem dye bath concentrations were exposed to artificial daylight by using Tera Light Fastness Tester [Patent No. 15182, Egypt, 1981] for 160 hours at 25 \pm 2°C and at a relative humidity of 65 \pm 5%. A standard blue scale was hanged alongside the samples (ISO 105- B02) to estimate the light fastness of the fabrics, i.e., the resistance of the fabric to a change in its characteristics as a result of light exposure.

The reflectance spectra in the visible region from 400 to 700 nm for all wool samples was carried out by using a Shimadzu Vis-Double Beam Spectrophotometer with standard illuminant C (1174.83) model V-530 and bandwidth 2.0 nm covers the range 200-2500 nm with accuracy ±0.05%.

The effect of different Neem dye concentrations on the color properties on the wool fabrics was performed using the CIE Colorimetric System, CIE 1931 2-degree Standard Observer. The tristimulus values (x_r, y_r, z_r) , the relative brightness (L*), color constants (a* and b^*), whiteness index (W), chroma (C^*) , hue (H*), tint (T) and color difference (∆E*), were calculated from the obtained reflectance values [15,16]. Also, the absorption coefficient (α), optical band gap energy, extinction coefficient (K) and the color strength (K/S) of the wool fabrics were determined by using the relations [17-22]:

Fig. 1. The absorption spectrum of the aqueous extract of the neem leaves

$$
\alpha = (1/d) \ln [(1-R)^2/T]
$$
 (1)

$$
K = \alpha \lambda / 4\pi \tag{2}
$$

 $K/S = (1-R)^{2}/2R$ $/2R$ (3)

Where R is the reflectance, T is the transmittance ($\approx 10^{-3}$), and d is the thickness of the sample (about 0.01 cm). The absorption coefficient, *α,* depends on the dyestuff and S the scattering coefficient, S, depends on the substrate.

The band tail energy value, E_b , in the normally forbidden band gap was determined by applying Urbach relation as [17-22]:

$$
\alpha = \alpha_o \exp \left[h \sqrt{E_b} \right] \tag{4}
$$

Where α_0 (= constant) is the absorption coefficient at photon energy (h*ν*) = 0 and ν is the frequency of radiation. The optical band gap energy (E_q) were calculated from the absorption coefficient data according to Tauc's model as [17-22]:

$$
\alpha h v = B (h v - E_g)^n \tag{5}
$$

Where B represents the band tail parameter in the range from 10^5 to 10^6 cm⁻¹.eV⁻¹ calculated from the slope of Tauc's edge and n is the electronic transition responsible type for absorption equals ½ and 2 for the allowed direct and indirect transition, respectively.

2.5 Fastness Properties

The change in color and staining of the dyed samples were evaluated for color fastness to washing according to the conditions mentioned in the AATCC Standard method (AATCC, 1997 & 1996). A specimen of the wool fabric in contact with one or two specified adjacent fabrics was mechanically agitated under specified conditions for 30 minutes and at 50° C for temperature in a soap solution without a fluorescent whitening agent (WOB), then rinsed and dried. The change in the color of the specimens and the staining of the adjacent fabric were determined concerning the original fabrics using the Grey scale of washing fastness for color change as: from 1 (very poor), 2 (poor), 3 (fair), 4 (good) to 5 (excellent).

A schematic diagram was drawn to represent the process of the present experimental section.

Schematic diagram represents the process of the experimental work

3. RESULTS AND DISCUSSION

Fig. 2 shows the reflectance (R%) as a function of wavelength in the visible region (400-700 nm) for unexposed (a) and exposed (b), blank and Neem dyed wool fabrics with different dye bath concentrations. It was observed from the figure that the behaviors of the unexposed and exposed dyed wool fabrics completely change in comparison with the undyed fabrics. Also, R% values increase with increasing wavelength for all fabrics and decrease gradually with increasing the dye bath concentration up to 80 g/L in comparison with the blank fabric. The observed variations indicate high color change by increasing Neem concentration. Similar behaviors and trends with more differences in R% values were observed for all samples after exposing to artificial daylight for 160 hours.

From the reflectance spectra, the CIE tristimulus values $(x_r, y_r,$ and z_r); the relative brightness (L^*) ; color constants (a* and b*); whiteness index (W); chroma (C^*) , hue (H^*) , tint (T) and color difference (∆E), of all fabrics were calculated. The tristimulus reflectance values; (x_1) , (y_1) and (z_r) ; for the blank wool fabric (Fig. 3a,b) as well as the unexposed (a) and exposed (b) Neem dyed wool fabrics with different dye bath concentrations were illustrated in Figs. 4, 5 and 6. It was observed that the behaviors of x_r , y_r , and z_r for all samples are similar and there is a shift in the peak positions (about 10 nm) for the Neem dyed wool fabrics in comparison with the blank one either without or with the exposure to artificial daylight.

Table 1 illustrates the maximum tristimulus reflectance values at their peak positions for

Fig. 2. The reflectance, R%, spectra as functions of wavelength (*λ***) in the visible region of blank and Neem dyed wool fabrics with different dye bath concentrations before (a) and after (b) exposure to artificial daylight for 160 hours**

Fig. 3. Dependence of the tristimulus reflectance values [x^r (▲), y^r (■) and z^r (●)] on wavelength for blank wool fabrics before (a) and after (b) exposure to artificial daylight for 160 hours

Fig. 4. Dependence of the tristimulus reflectance value (xr) on wavelength for blank and Neem dyed wool fabrics with different dye bath concentrations before (a) and after (b) exposure to artificial daylight for 160 hours

Fig. 5. Dependence of the tristimulus reflectance value (yr) on wavelength for blank and Neem dyed wool fabrics different dye bath concentrations before (a) and after (b) exposure to artificial daylight for 160 hours

Fig. 6. Dependence of the tristimulus reflectance value (zr) on wavelength for blank and Neem dyed wool fabrics with different dye bath concentrations before (a) and after (b) exposure to artificial daylight for 160 hours

blank and Neem dyed wool fabrics. It was noticed that the tristimulus values decrease with

increasing the Neem dye bath concentration. Also, the tristimulus values of the exposed wool fabrics to artificial daylight were higher than those of the unexposed values. In addition, the x_r , y_r , and z_r values for all samples either unexposed or exposed to artificial daylight were remarkably lower than that of the blank wool fabric.

The color parameters such as: the relative brightness (L*), color constants (a* and b*), whiteness index (W), chroma (C*), hue (H*), tint (T) and ∆E* were analyzed using the CIE Colorimetric System, CIE 1931 2-degree Standard Observer and were tabulated in Table 2. It was noticed that: The values of the relative brightness, L*, decrease with increasing Neem concentration which means that the fabrics become fader in color. The values of the color constant, a*, increase by increasing Neem concentration which indicates that, there is an increase in a red component instead of a green one. The values of the color constant, b*, also increase by increasing Neem concentration which indicates that there is an increase in a yellow component instead of a blue one. The detection of the whiteness index (W) and tint, T, values indicate opposite behavior in comparison with the color constants, a* and b*. The observed decrease in the values of the whiteness index, W, and the results of the color scales, C^{*} and H^{*}, indicate that variations in color difference between the blank fabrics and the Neem dyed were occurred due to the different Neem

concentration. Similar trends were detected in the values of the color parameters when the fabrics exposed to artificial daylight in comparison with that of the unexposed samples. In addition, as noticed from the table, the observed changes in the values of the color
parameters by either increasing Neem parameters by either increasing Neem concentration and/or exposure to artificial daylight for 160 hours may be due to the change in the physical bonds and therefore, changes in the molecular configuration of the fabric. These variations may lead to the formation of new color centers. Also, the obtained results of the color parameters were of great importance to improve the optical properties of the fabrics.

The chromaticity coordinates x [= $X/(X + Y + Z)$] and y $[= Y/(X + Y + Z)]$ were calculated for the fabrics according to the CIE Colorimetric System [15,23,24]. Fig. 7a,b shows the locations of the blank and dye wool fabrics with different dye bath concentrations before and after exposure to artificial daylight for 160 hours on the chromaticity diagram and their distances to the white point. It was clear that the chromaticity coordinate values (x) and their distances to the white point gradually increase with increasing Neem concentration which means that the locations of the fabrics concentrations were different. This observation confirmed that change in the spectral colors of a

(#) Variation in ΔE due to exposure to artificial daylight for 160 hours*

wool network was occurred either by dyeing or exposure to artificial daylight.

Briefly, dyed wool with different concentrations of dyeing presented diverse colors and might be attributed to the changes in Neem chemical groups [25]. As the surface of wool fibers covered with lamellar scales which mean that they contain lots of –S-S- bonds, therefore, with the interaction of Neem dye, the –S-S- bonds could be broken and –S-H- bonds were constructed, resulting in the formation of a loosened structure of wool fibers. So, the adsorption amounts and rate of dyes on wool fiber were improved in the presence of Neem dye.

The absorption coefficients (*α*) of the blank fabric as well as the unexposed and exposed wool fabrics dyed with different concentrations were calculated by using equation 1 and the reflectance values (Fig. 2a,b) and were represented as functions of wavelength (Fig. 8a,b) and photon energy (Fig. 8c,d). It is clear from the figures that a remarkable decrease in the absorption coefficient values with increasing the wavelength for all samples. Also, α values increase with increasing Neem concentration

through the whole wavelength and photon energy ranges for both the unexposed and exposed samples to artificial daylight.

As reported previously [26], the change occurs in the absorption coefficient may be due to the difference in the chemical bonds between the fabric and the dye which forms other molecular species and leads to the formation of new color centers. The detected increase in absorption coefficient values may be attributed to the change in the molecular configuration of the fabric which may indicate the formation of new color centers. The data also indicate that dyeing with Neem and exposure with daylight leads to the rupture of the bonds and formation of free radicals. These observed variations may be due to modification in molecular structure introduced as a result of the degradation process. Also, the dye components role was to strengthen the linkage between the reactive species of the wool fabric chemical groups and their polar groups.

Fig. 9a,b shows the dependence of ℓn *α* on h*ν* for blank and Neem dyed wool fabrics with different dye bath concentrations before and after exposure to artificial daylight for 160 hours. It

Fig. 7. Chromaticity diagram of blank and Neem dyed wool fabrics with different dye bath concentrations before (a) and after (b) exposure to artificial daylight for 160 hours

Fig. 8. Dependence of absorption coefficient values of blank and Neem dyed wool fabrics with different dye bath concentrations on wavelength and photon energy (hν); before (a and c) and after (b and d) exposure to artificial daylight for 160 hours, respectively

was clear from the figure that, each curve could not be represented by straight lines relation which means that the absorption does not follow the quadratic relation for inter-band transitions to verify Urbach rule [21]. On other hand, as observed from Fig. 10 that the variation between $ln \alpha$ and hy showed complicated behaviors which may due to the change in the total number available states caused by either increasing Neem concentration and/or exposing to the

artificial light according to the compromise between the degradation and/or cross-linking processes [27]. Also, the observed changes may be due to the variation in the internal fields due to the interaction between Neem and wool fabrics chemical groups.

Fig. 10a,b shows the variations of (*α*h*ν*) ²with hν for blank and Neem dyed wool fabrics with different dye bath concentrations before and after

Fig. 9. Variation of ℓn α with hν for blank and Neem dyed wool fabrics with different dye bath concentrations before (a) and after (b) exposure to artificial daylight for 160 hours

exposure to artificial daylight for 160 hours. Table 3 tabulates the values of allowed direct energy gap (E_d) calculated by extending the linear parts of the curves to zero absorption. It was illustrated that the E_d values decrease with increasing the dye bath concentration and nearly no variation was observed between the exposed and unexposed fabrics. These decreases indicate that the change in E_d shows the dependence on the Neem concentration and creation of localized states in the band gap and then structural changes were occurred [26].

The optical properties of the fabrics to light can be presented by calculating their extinction coefficient (K) [19]. Fig. 11a,b shows the

dependence of extinction coefficient (K) as a function of wavelength for blank and Neem dyed wool fabrics with different dye bath concentrations before and after exposure to artificial daylight for 160 hours. It was clear from the figure that, similar behaviors of the extinction coefficients for all samples were observed through the whole wavelength range. Also, the values of the extinction coefficient were found to be in order 10^{-3} which indicates that wool fabrics may be considered as a semiconducting material at room temperature [28]. Also, the increase in the extinction coefficient values with increasing the concentration of Neem dye without or with exposure to artificial light shows that the fraction of light lost could be due to scattering.

Fig. 10. Variations of (*α***h***υ***) ²with photon energy (h***ν***) for blank and Neem dyed wool fabrics with different dye bath concentrations before (a) and after (b) exposure to artificial daylight for 160 hours**

Fig. 11. Dependence of the extinction coefficient on wavelength for blank and Neem dyed wool fabrics with different dye bath concentrations before (a) and after (b) exposure to artificial daylight for 160 hours

Table 3. Values of the direct energy gap (Ed) for blank and Neem dyed wool fabrics with different Neem concentrations before and after exposure to artificial daylight for 160 hours

Direct energy gap (E_d) (eV)		
Before exposure	After exposure	
2.18	2.12	
1.67	1.67	
1.65	1.65	
1.63	1.63	
1.62	1.62	

The amount of dye absorbed by the wool fabric was obtained by determining the color strength (K/S) value of the dyed material using equation 3. The effect of the increase in the Neem dye bath concentration on the color strength of the blank and wool fabrics with different dye bath concentrations before and after exposure to artificial daylight for 160 hours was illustrated in Fig. 12a,b. It is clear that the values of the color strength decrease sharply with increasing wavelength for all samples. Also, K/S values increase with increasing Neem concentration up to 80 g/L for both unexposed and exposed samples. This means that higher the concentration of Neem higher is the coloring component extracted and higher is the color strength of the fabric dyed with these extracts which mean that occurrence of deepest shade.

Table 4. Color fastness grades for blank and neem dyed wool fabrics with different dye bath concentrations

The effect of the dye bath concentration can be attributed to the correlation between dye and wool fabrics. Since Neem dye is a water-soluble dye and containing anionic groups; these anionic groups interact ionically with the protonated terminal amino groups of wool fabrics through ion exchange reaction. Due to this ionic interaction, the dye-ability of the fiber would increase with increasing the Neem dye concentration may be due to i) the enhanced desorption of the dye; and ii) the greater availability of the dye molecules in the vicinity of the fiber. The present data are in good agreement with the previously reported results [29].

Table 4 above represents the color fastness properties of the blank and Neem dyed wool fabrics with different dye bath concentrations. Washing fastness results were estimated with respect to Grey scale and the results for light fastness were evaluated with respect to blue wool scale. Washing fastness values of the fabrics dyed with different dye bath concentrations show comparable results. In case of washing, the results were expressed in terms of change in color and staining of the neighboring cotton and wool fabrics. It was clear that as the Neem dye concentration increase, the samples give good rating (4) to very good rating (4/5). This is because the ionic bonds between dye molecules and the carboxyl groups in the wool fibers are strong. The washing fastness grade for color staining gives very good rating (4/5) to excellent rating (5) with increasing Neem dye concentration up to 80 g/L which means that the dyed wool fabrics have good washing fastness properties.

From Table 4 it was also noticed that the light fastness grade of the wool fabrics increase from 4 to 4/5 (good to very good) by increasing the Neem concentration up to 80 g/L was 4/5 which indicate very good grade. The results indicate

that light fastness was increased with increasing the dye concentration which may be due to the enhancement in dye aggregation inside the fabrics. Considerable influence in light fastness was exerted due to the polar groups of the substrate and its physical structure which tend to neutralize the polar group of the dye molecules in contact with the substrate by columbic attraction and then decreasing fading [29]. Also, exposure of wool fabrics to light may result in changes to color and fiber properties [29,30].

4. CONCLUSION

In the present study, leaves of Neem can be used as a dye for coloring wool fabrics. It was concluded from the obtained data that, the tristimulus reflectance values, color parameters values, the absorption coefficient, extinction coefficient and color strength, were found to be influenced by either the concentration of the Neem dyeing bath concentration and/or exposure to artificial daylight for 160 hours concerning the chemical structure of the used dye. Also, the Neem plant shows good shade reproducibility with significant fastness properties. It was found that Neem dye can be successfully used for dyeing of wool fabric to obtain a wide range for soft hell shades. The process of extraction was simple and environmentally friendly. The natural colorant obtained from Neem leaves was successfully used as an eco-friendly dye to obtain different shades of yellow. Also, the data obtained indicate that the increasing of the concentration of the natural dye extracted from Neem leaves has good potential in textiles dyeing.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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