

12-1-2011

Section: Chemistry

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IBRAHIM, N.; EL-ZAIRY, W.; EL-ZAIRY, M.; and GHAZAL, H. (2011) "DYEING AND FUNCTIONAL PROPERTIES OF POLYAMIDE-6 FABRIC DYED WITH NATURAL DYES," *Al-Azhar Bulletin of Science*: Vol. 22: Iss. 2, Article 15.

DOI: <https://doi.org/10.21608/absb.2011.7017>

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DYEING AND FUNCTIONAL PROPERTIES OF POLYAMIDE-6 FABRIC DYED WITH NATURAL DYESN. A. IBRAHIM^{1*}, W. M. EL-ZAIRY², M. R. EL-ZAIRY² and H. A. GHAZAL²*1 National Research Centre, Textile Research Division, Dokki, Cairo, Egypt.**2 Faculty of Applied Arts, Printing, Dyeing and Finishing Dept. Helwan Univ., Cairo, Egypt.***Abstract**

There is increasing interest in adding value to polyamide-6 fabrics via upgrading their functional properties. In the present study, selected natural dyes namely madder and safflower yellow dyes as well as mordants namely alum, Zn-sulfate and tannic acid have been used to identify the proper dyeing conditions for attaining high quality natural dyeings along with imparting multifunctional properties, i.e. anti-UV and anti-bacterial properties, to the dyed polyamide-6 fabric. The results demonstrate that the improvement in dyeing properties, i.e. color yield as well as fastness properties, along with the enhancement in the imparted functional properties are governed by the type and concentration of natural dye, kind of mordant, dyeing sequence and conditions. The dyes / mordants examined exhibited high fastness properties and offering better UV-protection function and antimicrobial activity against G-ve bacteria (*E-coli*).

Key Terms: Polyamide-6, Natural dyes, Mordants, Anti-UV, Anti- microbial.

1. Introduction

Recently, there has been a growing interest in using the non-toxic, eco-friendly coloring materials for textile coloration to avoid and/or minimize the negative impacts of intermediates, auxiliaries as well as chemicals used in synthetic dyes.¹⁻² Accordingly there is a great potential for using natural dyes in textile applications instead of the synthetic ones taking in consideration the environmental concerns and technical drawbacks of natural dyes.³⁻⁵ Applications of natural dyes from plant sources is preferable because of their very low toxicity.

On the other hand, many of the plants used for natural dye extraction, i.e. from renewable resources, have recently been shown to possess remarkable antimicrobial activity and/or can absorb significant amount of harmful UV-radiation, especially UV-B.⁶⁻⁸

The current research work examines the enhancement effect of natural dyeings on both the UV-protection and anti-bacterial function of polyamide-6 fabric.

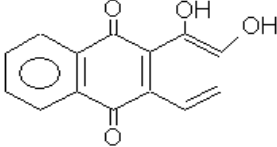
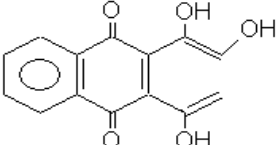
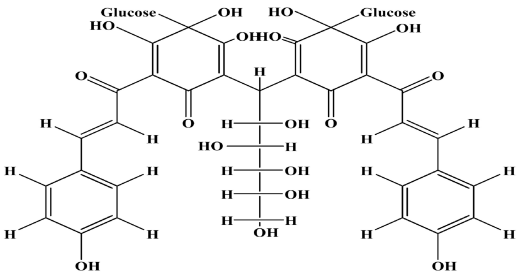
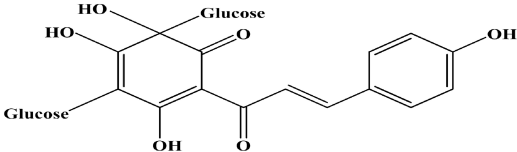
2. Experimental

2.1. Materials

Mill-scoured and bleached knitted polyamide-6 fabric of 58g/m² was used in this study. Commercial-grade natural dyes were purchased from local market.

Table 1 shows the chemical structures of the natural dyes used in the experiment.

Table 1. Natural dyes used

Natural dye	Chemical structure
1. Madder ⁹	<div style="display: flex; justify-content: space-around; align-items: center;">  <div style="text-align: right;">alizarin</div> </div> <div style="display: flex; justify-content: space-around; align-items: center; margin-top: 10px;">  <div style="text-align: right;">purpurin</div> </div>
2.Safflower yellow dye ¹⁰	<div style="display: flex; justify-content: center; align-items: center; margin-bottom: 20px;">  </div> <div style="text-align: center;">Safflower yellow B</div> <div style="display: flex; justify-content: center; align-items: center; margin-top: 20px;">  </div> <div style="text-align: center;">Safflomin</div>

Analytical reagent-grade chemicals were used for the dyeing of polyamide-6 with the nominated natural dyes.

2.2. Methods

2.2.1. Properties of colorants

The used natural colorants were extracted from their dried-powder forms in a material-to-liquor ratio (LR) of 1/100 in distilled water at the boil for 2hrs. The cooled-extracted dye solutions were used as a stock solutions after filtration.

2.2.2. Dyeing

Pre-mordanting, using Zn-sulfate, alum or tannic acid as a mordant, was done at a temperature of 90°C, LR (1/30); time (30 min.) in the presence of the used mordant (1-3% owf).

Dyeing of the pre-mordanted fabric samples was carried out at (60°-90°C), for (30-60 min.), using 1/30 LR at different pH's. After dyeing, the samples were soaped at 60°C for 15 min and thoroughly rinsed in water. Both pre-mordanting and subsequent natural dyeing were carried out in the shaking water bath. Typical formulations used in this study are given in the text.

2.2.3. Testing

Color strength, K/S of the obtained natural dyeings was measured and evaluated at the λ max of the used natural dye, using the color-Eye 3100® spectrophotometer and the Kubelka- Munk equation: $K/S = (1-R)^2 / 2R$ (where K: absorption coefficient, S: scattering coefficient, and R: reflectance)¹¹.

Fastness properties to washing, crocking and perspiration were assessed according to AATCC test methods (61-1972), (8-1972) and (15-1973) respectively.

UV-protection factor (UPF) was assessed according to the Australian/ New Zealand standard (AS/NZS 4399-1996).

Antimicrobial properties of the obtained natural dyeings against Gram- negative bacteria (*E-coli*) were examined for a clear zone of inhibition according to AATCC 100-1999. All the determinations in the present work were performed in triplicate.

3. Results and Discussion

In this article, attempts have been made to examine the suitability of using natural dyes in the dyeing of pre-mordanted polyamide-6 fabric, taking into consideration the environmental aspects, as well as to study the anti-microbial and UV-blocking properties of the obtained dyeings. Results obtained along with their appropriate discussion are as follows.

3.1. Dyeing temperature

For a given set of pretreatment and subsequent natural dyeing conditions, Fig. 1 illustrates that: i) raising the dyeing temperature from 60° up to 90°C results in an improvement in the color depth, K/S, of the obtained madder (Fig. 1.a) and safflower- (Fig. 1b) dyeings, ii) this enhancement in the extent of coloration reflects the positive impact of increasing the dyeing temperature on reducing the conglomeration of colorants molecules, enhancing the swellability of the pre-mordanted substrate, and increasing the accessibility and availability of the dye-sites thereby leading to easy and rapid diffusion into the swelled substrate as well as more dye uptake and fixation into and/or onto the substrate, i.e. higher K/S values,^{8,10} and iii) the extent of improvement is determined by the type of the natural dye, i.e. chemical structure, nature/position of substituent groups on the dye chromophore, molecular size, dye aggregates and extent of fixation or embedded firmly into the substrate,^{1,4,8} as well as kind of mordant ,Zn-sulfate > alum > tannic acid, regardless of the used natural dye, and differences among them in facilitating the formation of the dye-mordant-fiber bonds.^{1,2,4,8}

3.2. Dyeing time

The obtained data, Fig. 2, showed that: i) prolonging the dyeing time of premordanted substrates is accompanied by an increase in the extent of dye uptake as well as the color depth of the obtained madder (Fig. 2a) and safflower (Fig. 2b) dyeings regardless of the used mordant, ii) this increase in K/S values is a direct consequence of enhancing the extent of diffusion, penetration as well as fixation of the disaggregated dye molecules onto and/or within the available dye sites of the swelled substrate, and ii) both the type of natural dye as well as the kind of mordant have practically a governing effect on the extent of dyeing, expressed as K/S values, as discussed before.

3.3. Dye concentration

It is clear, Fig. 3, that: i) increasing the dye extract % from 0 up to 100% results in a sharp increase in the color depth of the obtained madder. (Fig. 3a) and safflower- (Fig. 3b) dyeings, irrespective of the used mordant, ii) this enhancement in the K/S values most probably is attributed to the greater availability and accessibility of dye molecules onto and/or within the vicinity of easily accessible attachment sites,^{8,10} and iii) the variation in K/S values of the obtained dyeings upon using the nominated mordants reflects the differences among them in molecular weight, chemical composition, location and extent of deposition onto and/or within the substrate, extent of modification of the pre-mordanted substrate, affinity to the used natural colorants as well as extent of fixation via the formation of dye-mordant-fiber bonds.⁸

3.4. Dyeing pH

It is well known that the pH of dyeing bath is one of the most important parameters affecting the extent of exhaustion and final fixation of the used natural dyes. For this reason, we studied the effect of pH, keeping other conditions constant, on the K/S of the obtained natural dyeings. Fig's 4a and 4b show that: i) the enhancement in K/S values of the obtained natural dyeings is determined by the pH of the dyeing bath and follows the decreasing order $\text{pH}2 > \text{pH}6$ regardless of the used natural dye, ii) this enhancement in K/S values at pH2 could be attributed to the electrostatic forces between the protonated amino ($-\text{NH}_2$) and amido ($-\text{CONH}-$) groups of the nylon fibre and the negatively charged dye molecules in aqueous medium, and the behavior of the used natural dyes like disperse dyes under the used acidic pH, thereby giving higher dye uptake, cannot be ruled out,^{4,12-13} and iii) the change in K/S of the obtained dyeings is governed by the extent of exhaustion and fixation of the used natural dyes as well as the extent of modification of the pre-mordanted substrate.

3.5. Functional and dyeing properties

As far as the changes in the dyeing and functional properties of the obtained natural dyeings as a function of kind of natural dye as well as type of mordant, the data in Table 2 signify that: i) premordanting using the nominated mordants brings about a remarkable improvement in the K/S of the obtained dyeings regardless of the used natural dye and mordant, ii) the extent of improvement in K/S value as well as fastness properties of the obtained natural dyeings are governed by the nature of the dye as well as the efficiency of the used mordant as discussed before, iii) pre-

mordanting followed by natural dyeing results in an improvement in both the UV-protection properties against the harmful UV-B radiation as well as in the antibacterial activity against G-ve bacteria (*E-coli*), iv) the UPF value depends on the dye-fibre interaction, the size and geometry of the dye molecule,¹⁴ as well as the ability of the used natural dye to absorb the harmful UV- B radiation, v) the improvement in the UPF of pre-mordanted fabric samples most probably attributed to the increased extent of dye exhaustion on fabric, formation of deeper shade and/or the different light absorption characteristics of the natural dye-mordant complex^{7,8}, and vi) the increase of anti-microbial activity of pre-mordanted fabric samples reflects the inhibiting effect of the mordant, natural dye and/or both of them synergistically via binding the microbial proteins,^{7,8} ability to reduce microbial growth⁶, as well as ability of the used mordants to react with the thiol groups thereby leading to cell inhibition or cell inactivation.¹⁵

4. Conclusions

This study was aimed to investigate the possibility of upgrading the functional properties of polyamide-6 fabric via natural dyeing. The multifunctionality of the obtained natural dyeings was evaluated by analyzing its UV-protection efficiency and antibacterial activity against G-ve bacteria (*E-coli*). The obtained results signified that multifunctional properties can be obtained by post- dyeing of premordanted polyamide- 6, with alum, Zn-sulfate or tannic acid, with madder and safflower yellow natural dyes. The enhancement in K/S and fastness properties of the obtained dyeings as well as the improvement in the imparted UV-protection and anti-bacterial properties are determined by the type of both the natural dye and the used mordant.

Table 2. Dyeing and functional properties of obtained natural dyeings.

Mordant	Natural dye	K/S	Fastness Properties								UPF	Z.I (mm)
			WF		RF		PF					
			St.	Alt.	Dye	Wet	Acid		Alkali			
							St.	Alt.	St.	Alt.		
None	Madder	3.61	4-5	4-5	4-5	4	3-4	4	4	4	19	3.7
	Safflower	4.02	4-5	4-5	4-5	4	4-5	4-5	4-5	4-5	13	2.8
Alum	Madder	7.72	5	5	4-5	4-5	4-5	4-5	4-5	4-5	25	8
	Safflower	11.81	5	5	5	4-5	4-5	4-5	4-5	4-5	22	5
Zn-Sulfate	Madder	12.20	5	5	4-5	4-5	4	4-5	4	4-5	40	14
	Safflower	13.32	5	5	5	5	4-5	4-5	4-5	4-5	35	9
Tannic acid	Madder	6.91	5	5	5	5	4	4-5	4	4-5	37	11
	Safflower	5.52	5	5	5	4-5	4-5	4-5	4-5	4-5	30	6

Pre-mordanting: mordant (3% owf); LR (1/30); at 90°C for 30 min.

Natural dyeing: dye stock solution (50%); nonionic wetting agent (2g/L); pH(2); LR (1/30), at 90°C for 60 min.

K/S: Color strength, WF: wash fastness; RF: rubbing fastness; PF: perspiration fastness; UPF: UV-protection factor; ZI: zone of inhibition.

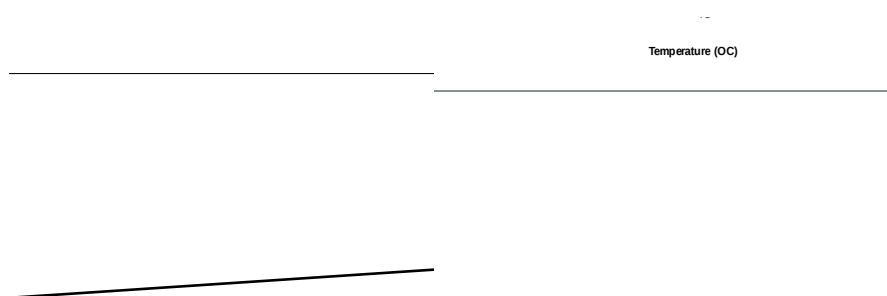


Fig.1. Effect of dyeing temperature on the K/S values of the obtained madder-dyeings (Fig. 1a) and safflower-dyeings (Fig. 1b). Zn-sulfate (□); Alum (●); Tannic acid (×).

Pre-mordanting conditions: mordant (3% owf) at 90°C for 30 min., LR (1/30)
 Natural dyeing conditions: dye stock solution (50%); nonionic wetting agent (2g/L); LR (1/30), time (60 min.); pH (2).

Madder
(a)

Fig. 2. Effect of dyeing time on the K/S values of the obtained madder-dyeings (Fig. 2a) and safflower-dyeings (Fig. 2b). Zn-sulfate (□); Alum (●); Tannic acid (×).

Pre-mordanting conditions: mordant (3% owf) at 90°C for 30 min., LR (1/30)

Natural dyeing conditions: dye stock solution (50%); nonionic wetting agent (2g/L); LR (1/30), at 90°C; pH (2).

Safflower
(b)

Fig.3. Effect of dyeing stock solution on the K/S of the obtained madder-dyeings (Fig.3a) and safflower-dyeings (Fig. 3b). Zn-sulfate (□); Alum (●); Tannic acid (×).

Pre-mordanting conditions: mordant (3% owf) at 90°C for 30 min., LR (1/30)

Natural dyeing: nonionic wetting agent (2g/L); LR (1/30), at 90°C for 60 min.; pH (2).

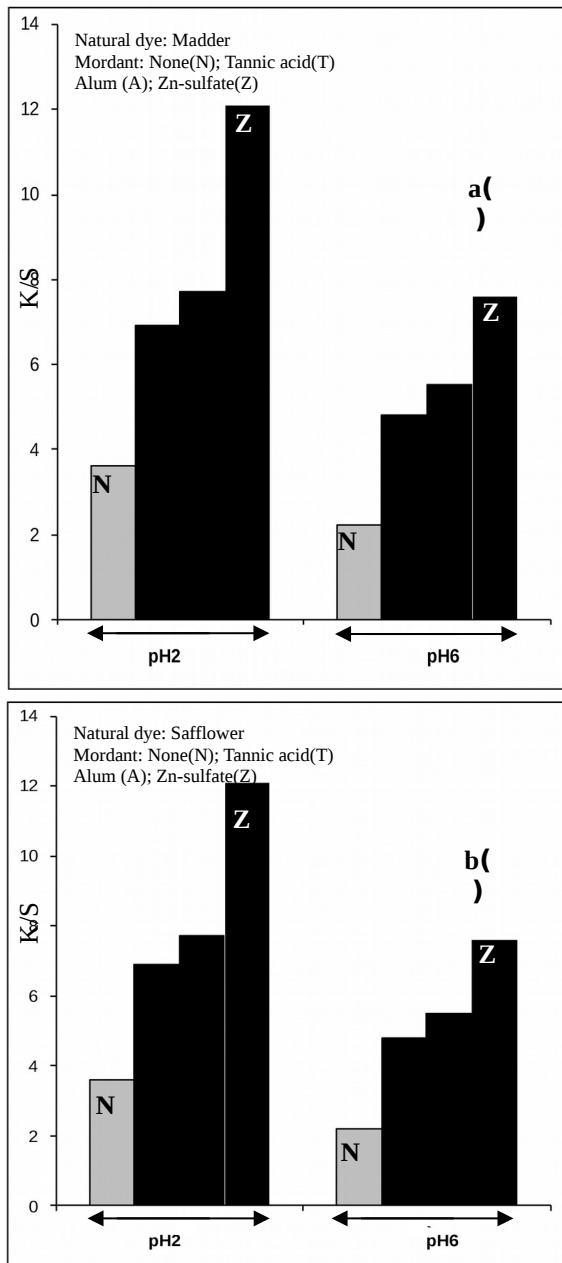


Fig. 4. Effect of the dyeing bath pH on the obtained Madder – dyeings (Fig 4a) and Safflower dyeings (Fig 4b).

* Pre-mordanting: mordant (3% owf); at 90°C for 30 min, LR (1/30).

* Natural dyeings: dye stock solution (50%); nonionic wetting agent (2g/l); LR (1/30), at 90°C for 60 min.

Fig. 4. Effect of the dyeing bath pH on the obtained Madder – dyeings (Fig 4a) and Safflower dyeings (Fig 4b).

* Pre-mordanting: mordant (3% owf); at 90°C for 30 min, LR (1/30).

* Natural dyeings: dye stock solution (50%); nonionic wetting agent (2g/l); LR (1/30), at 90°C for 60 min.

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AHIM, *et al*