Functional Dyeing of Wool with Natural Dye Extracted from *Berberis vulgaris* Wood and *Rumex Hymenosepolus* Root as Biomordant

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ABSTRACTB: *In this study, a functional colorant, Berberine, extracted from Berberis vulgaris wood was applied onto wool fiber using the extract of roots of Rumex Hymenosepolus as biomordant. The effect of treatment variables on the color strength of dyed fibers was examined. The fastness properties of dyed wool against washing, light, dry and wet rubbing were evaluated. Dyed samples were tested for antibacterial activity using AATCC test method 100-2004. Tannin present in the roots of Rumex hymenosepolus when used as a biomordant on wool increased the color strength of the dyed goods. Increase in dyeing time, temperature and pH caused deeper shades. Biomordanting, increased light fastness, rub fastness and wash fastness of dyed samples. The dyed wool represented a high level of antibacterial activity. The extract of the Berberis vulgaris can be considered as a natural dye of acceptable fastness properties together with excellent antibacterial activity for woolen textiles.*

KEY WORDS: *Berberine, Natural dye, Biomordant, Antibacterial, Wool.*

INTRODUCTION

Dye substances of plant origin are present in many wild and cultivated species. Development of synthetic dyes in the last century reduced the use of natural dyes in modern dyeing [1,3]. Synthetic dyes are produced from cheap petroleum sources, and generally have easy dyeing with superior fastness properties. But there are drawbacks about synthetic dyes mainly toxicity and environmental pollution caused by waste water expelled from dye-houses.

Recently, a new tendency to natural dyes has risen mainly due to their environmentally friendly characteristics [1,2]. They are considered to give several advantages such as non toxic functions, specific medical actions and environmentally friendly finishes [2]. Natural dyes are clinically safer than their synthetic analogs in handling and use because of non carcinogenic and biodegradable nature [3-5].

Microbial growth on textile materials has been considered as a major cause of biodegradation of textile arts, particularly natural products, which has led to development of antimicrobial technologies for preservative purposes. The growth of microorganisms on textiles inflicts a range of unwanted effects not only on the textile itself but also

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on the wearer. These effects include the generation of unpleasant odor, stains and discoloration in the fabric, a reduction in fabric mechanical strength and an increased likelihood of contamination. Such results have further stimulated the research on antimicrobial textiles with focuses on development of durable and powerful antibacterial finishing technologies [1,6].

Dyeing and functional finishing are two necessary but traditionally separated processes employed in textile treatments which need repeated wet treatments and drying, and thus consume large quantities of energy and produce large amounts of wastewater. Simultaneous dyeing and finishing could reduce both the costs of production and waste.

Some natural dyes, when applied on textiles, show antibacterial effects, so can be considered as functional dyes with health care properties. Lawsone, a natural dye extracted from henna, when applied on wool fabric, has shown antibacterial effect [1]. In other researches [2,11] cotton and nylon fabrics dyed with berberine, a natural colorant extracted from Amur cork tree, has shown antibacterial activity. *Rhizoma coptidis* extract has been applied on wool fiber and antibacterial property has been observed too [7].

Berberis vulgaris is a shrub which extensively is implanted in south khorassan-Iran and many other places all over the world, for it's valuable fruit, barberry. For gathering barberry fruit when ripe, the branches of the shrub are cropped with the fruit and when the barberry fruit dried and removed from the wood, the wool is dispelled or used as fuel.

In this study, this woods which are generally considered as waste, has been used as a source of a natural colorant. There is a natural yellow dye in these woods named Berberine [8]. This natural cationic dye has been extracted and applied on wool fiber with the extract of *Rumex hymenosepolus* as biomordant, and the fastness properties of dyed fiber has been evaluated. The roots of *Rumex hymenosepolus* have large quantities of tannin (18-35 %), which is considered as a biomordant in wool dyeing [8]. Finally the antibacterial property of dyed fibers has been tested according to AATCC 100-2004.

EXPERIMENTAL SECTION

Materials

Woolen yarn (Nm= 400, 2 ply) was purchased from local wool spinning mill. To remove any natural or synthetic impurities, the yarns were scoured using 2 g/L non-ionic surfactant and 2 mL/L ammonia at 45°C for 30 minutes and then rinsed and air dried.

Berberis vulgaris wood and *Rumex hymenosepolus* roots were first washed and dried and then chopped and powdered. To prepare the original solution of the dye, each 200 g of powder was added to 1 liter of distilled water and boiled for 2 hours and then filtered. The biomordant solution was prepared by adding 100 g of roots powder to 1 liter of distilled water and then boiled for 1 hour and filtered. The concentration of the resultant solution is 10%. Acetic acid and sodium carbonate were analytical grade reagents from Merck.

Methods

1- Biomordanting: the scoured wool yarns were mordanted using different amounts of biomordant solution (5, 10, 15, 20, 25 and 30 %owf) at 80 °C and L:G= 30:1, for 45 minutes.

2- Dyeing: 10 mL of original dye solution was mixed with 90 mL of distilled water for each 5g of wool (40% owf, L:G= 20:1). pH of the dyebath was adjusted using acetic acid or sodium carbonate (pH=4, 5, 6, 7, 8, 9). The dyeing was started at 40°C and the temperature was raised to final temp. (60, 70, 80, 90 and 96 °C (boil)) at the rate of 2°C per minute. Then the samples remained in that condition for appropriate time (30, 45, 60, 75, 90 min), and then rinsed and air dried. All mordanting and dyeing processes were carried out using a laboratory dyeing machine made by Rissanj Co.-Iran.

3- Color measurements: the reflectance of dyed yarns and color coordinates CIE L*, a*, b* values were measured on a X-Rite CA22 spectrophotometer using illuminant D65 and 10°standard observer. Color strengths (K/S) of dyed samples were calculated using kubelka-munk equation:

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

Where R is the observed reflectance, K is the absorption coefficient and S is the light scattering coefficient.

4 - Color fastness tests: For fastness tests, wool samples were dyed to give 1/1 standard depth (1/1 SD). Color fastness to washing, light and rubbing was measured according to: ISO 105-C01: 1989(E), ISO 105-B02: 1994(E), ISO 105-X12: 1993(E), using launder-o-meter, light fastness tester and crock-o-meter laboratory equipments all made by Rissanj Co. -Iran, respectively.

Table1: Effect of Biomordanting on color coordinates of dyed goods (10% owf biomordant, pH= 6).

	ΔE	L*	a*	b*
Without Biomordant	30.08	79.14	1.86	43.31
Biomordanted	57.21	62.33	18.30	62.29

ΔE: Color Difference, L: Lightness, a*: Redness-Greenness of color, b*: Yellowness-Blueness of color*

5- Antibacterial test: the antibacterial property of dyed yarns was quantitatively evaluated according to AATCC 100-2004. The bacterial species used were: *Klebsiella pneumoniae* (Gram negative) and *Staphylococcus aureus* (Gram positive). The colonies of both bacteria before and after incubation on the agar plate were counted by microscope. The reduction in the number of bacteria which was calculated using equation 2 shows the efficacy of the antibacterial treatment.

$$E\% = [(N1-N2)/N1]*100 \quad (2)$$

Where N1 is the number of bacteria colonies at the beginning of the test and N2 is the number of bacteria colonies after 24 hours contact of dyed yarns [9,11].

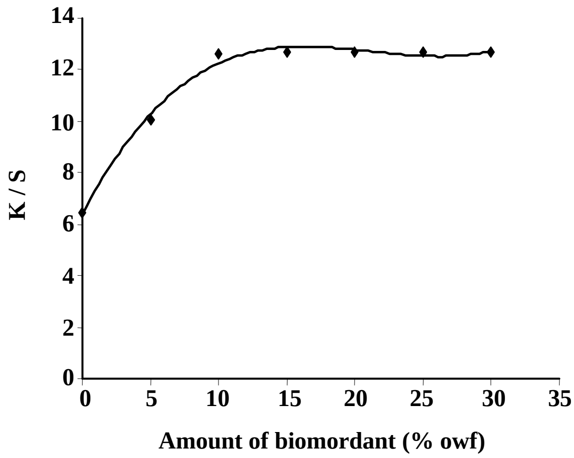

Fig. 1: Effect of the amount of biomordant on the color strength of dyed wool at pH= 5.

RESULTS AND DISCUSSION

Effect of Biomordant

Fig. 1 shows the effect of the amount of biomordant on the color strength of dyed wool at pH= 5. K/S increased when 5% owf of biomordant was applied. Increase in biomordant amount increased the K/S of dyed wool but addition of biomordant in excess of 15% had no significant effect on K/S. The root of *Rumex hymenosepolus* contains 18-35 % of tannin. Treatment of wool by tannin gives carboxylic groups (-COOH) to the fibers which in the dyeing stage, the ionic interaction between COO^- anion and cationic dye increases the absorption of the dye onto the fiber [12].

Table 1 shows that mordanting with the extract of Rumex hymenosepolus caused an increase in ΔE, a* and b* but a decrease in L* of dyed wool in comparison with the wool dyed without mordant. It means that the biomordanted wool has absorbed more dye and became brighter and yellower than non-mordanted one.

Effect of pH

As shown in Fig. 2, color strength increased with the increase of pH from 4 to 9. Over pH=9 there is serious damage to wool fiber. The native pH of the dyeing solution was 5 and around this pH the damage to wool fiber is at minimum level. In the alkali media, the number of anionic sites (COO^-) on wool fiber is relatively larger than that in acidic condition. Thus because of ionic interactions between cationic berberine dye and the COO^- sites in the wool fiber, the exhaustion increased in alkali media [1,13]. However many cationic dyes are not stable at alkaline pH; berberine dyes are stable under pH variations. Therefore, antimicrobial finishing of wool fibers with berberine dye is chemically feasible based on the above analysis [13].

Effect of Temperature

As shown in Fig. 3, color strength of dyed wool increased at higher temperatures. As the temperature rose, the wool fiber swelling and the breakdown of dye molecule aggregates in the solution became more, thus the diffusion of the dye molecules to the fiber became easier and thus the exhaustion and K/S increased. There was no significant increase in K/S when the temperature was raised from 90 ° C to boil (96 ° C).

Effect of dyeing time

Fig. 4 shows that the color strength increased when the dyeing time increased from 30 minutes to 75 minutes, after which the increase in time had no significant effect on dye absorption. It should be due to this fact that, dye in the fiber and the dyebath has reached to equilibrium after 75 minutes.

Fastness properties

Table 2 shows the fastness properties of biomordanted and non-mordanted dyed wool. All fastness properties of dyed samples, when biomordanted, were higher than when non-mordanted. This increase is due to increase in size of dye molecules when connected to tannin molecules into the fiber. Wet rub fastness was less than dry rub fastness because the water molecules can dissolve some of water-soluble dye molecules and make them easier to be removed from the fiber by rubbing. All fastness properties of biomordanted and then dyed wool fibers are generally acceptable.

Build up

Five wool samples were mordanted using 10 % owf biomordant and dyed at five depths, viz. 20, 40, 60, 80 and 100 % owf. (i.e. 5, 10, 15, 20 and 25 mL of original dye solution) at pH=5 and K/S of each dyeing was calculated. Fig. 5 shows that this natural colorant has good build up and color strength increased with increasing dye concentration.

Antibacterial activity

Table 3 shows the percent reduction in number of two bacteria after 24 hours incubation on the surface of undyed, only biomordanted and dyed (after biomordanting) wool. It is obvious that the dyed wool has excellent antibacterial activity against both bacteria used in this study. As shown in Fig. 6, the berberine colorant, is a quaternary ammonium compound, containing a positive charge on N atom that could destroy the negatively charged cell membrane of the bacteria by disturbing charge balances of cell membrane [1,10].

CONCLUSIONS

Berberine which is a cationic colorant present in the extract of *Berberis vulgaris* wood can be used as a natural dye for wool. There is more colorant in the roots of this plant but in this study the woods were used to prevent from wasting. When the wool was pre-mordant with the tannin present in the roots of *Rumex hymenosepolus,*

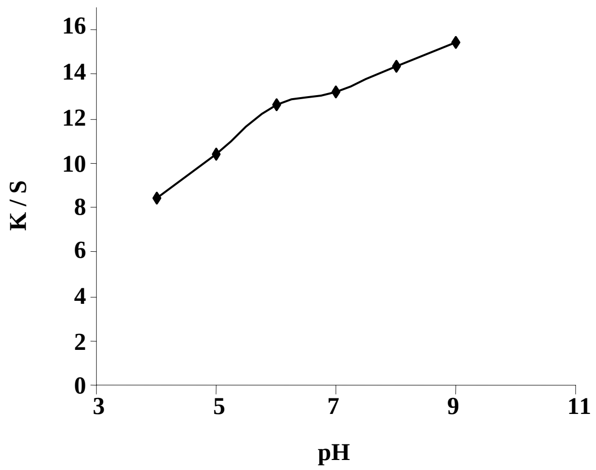

Fig. 2: Effect of pH on color strength of dyed wool.

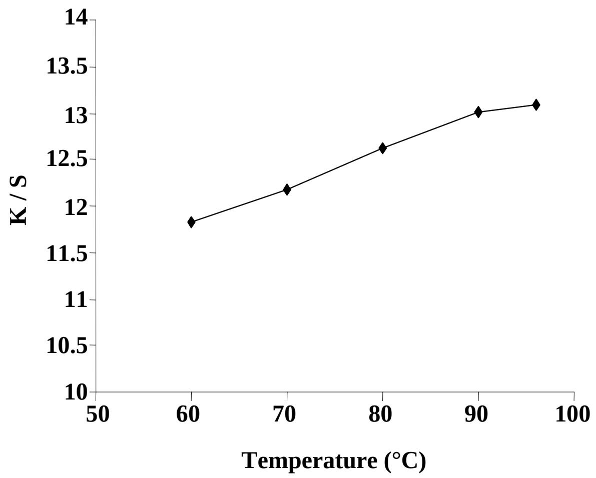

Fig. 3: Effect of Temperature on color strength of dyed wool.

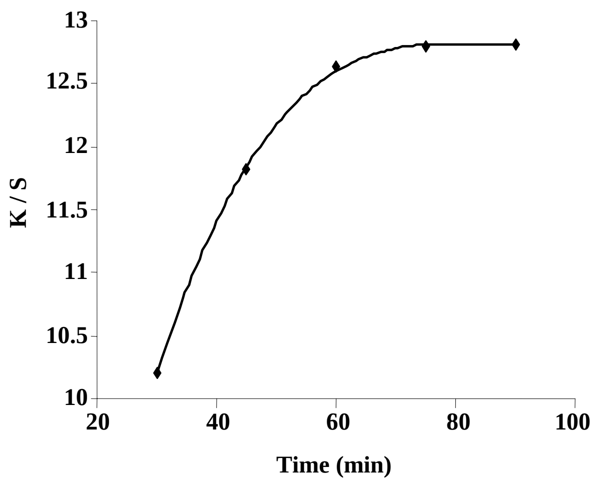

Fig. 4: Effect of dyeing time on color strength of dyed wool.

Table 2: Fastness properties of dyed wool without biomordant and with 10% owf biomordant.

	Light fastness	Wash fastness (color change)	Wet rub fastness	Dry rub fastness
Without biomordant	5-6	3	2-3	3-4
Biomordanted	5-6	4	3	4

Table 3: Reduction (%) of number of bacteria colonies after 24 hr incubation.

	Staphylococcus aureus	Klebsiella pneumoniae
Control	0%	0%
Biomordanted	<0.5%	<0.5%
Biomordanted and dyed	99.5%	99.4%

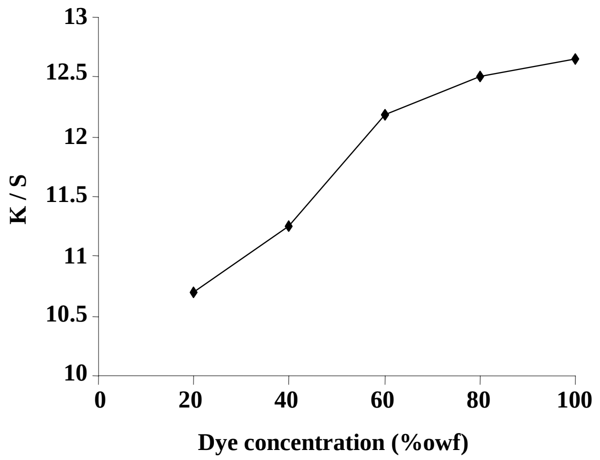

Fig. 5: Build up of used dye on wool fiber.

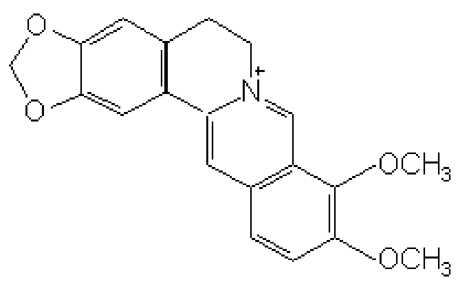

Fig. 6: Chemical Structure of Berberine.

the color strength and all fastness properties of dyed wool increased. Increase in dyeing time, pH and temperature, increased the color strength of dyed wool. In this study, the extract of two plants has been used to dye wool and no salt or other chemical has been used. This process is completely environmentally friendly and has the minimum pollution. Furthermore the dyed fiber has good antibacterial activity as another advantage of this natural dyeing process.

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