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Utilization of Natural Dyes to Fabricate Multifunctional Silk

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VARIOUS wet processing operations of textiles from the initial preparatory process to final finished clothes are now focused for green technology. Several conventional non-ecofriendly chemicals are being replaced by natural based products that are safe to the environment and health during manufacturing and usage. Natural dyes were used instead of synthetic dyes due to environmental conditions. Natural dyes are very important for textiles as well as to replace synthetic dyes, because of non-polluting, non-carcinogenic and eco-friendly. Silk is the lightest, thinnest and softest natural fiber and has many applications in the textile field. In the present work the silk fabric pretreated with PEG then dyed with two different natural dyes, the fabric then compared with those dyed with natural dyes only and with those with dyes and mordents. protective properties such as UV- blocking give a remarkable improvement in UPF values. the antibacterial reduction percentage was also enhanced. significant enhancement in dyeability values K//S values are demonstrated. Also, the mixture with PEG showed good tensile strength, wettability Enhancement and also crease recovery angles.

Keywords: Antibacterial activity, Multifunctional finishing, Natural dyes, Silk, Ultraviolet protection.

Introduction

The public has been an attentiveness towards the environmental problems, which is represented in textile industry to manufacture products with the environmentally friendly profile [1, 2]. Recently, the concept of “natural life” has been introduced into textiles, which lead to a new upsurge in the use of natural fibers and natural dyes [3,4]. The protective properties of suitable clothes depend on fiber composition (natural, artificial or synthetic fibers), fabric construction (porosity, weight, and thickness) and the wet-processing history of the fabric (using dyes, ultraviolet absorbers, and other finishing chemicals) [5-9].

The natural dyes are obtained from plants and animals’ sources that pigmentary molecules, which impart color to the substrate. These molecules containing aromatic ring structure coupled with a side chain are usually required for resonance and thus to impart. There is a correlation of chemical structure with color, chromogen-chromophore with autochrome [10]. They are non-toxic, non-allergic to the skin, non-carcinogenic, easily available and renewable [11].

Silk the queen of textiles is characterized by luxury, elegance and comfort. silk is produced by the silkworm bombyx mori; it is the only organic fiber that is in the form of crystallinity filament” [1, 12]. Silk has special properties such as luminosity, great air permeability, eximious performance of absorbing moisture, water vapor transmission, thermo-insulating and adaptation to skin [13, 14, 15]. So, it is commonly used in apparel, modern industry and medical field. However, silk is a proteinc fiber and suffers from some disadvantages such as an existing clear deficiency in antibacterial, where all kinds of microbes can grow at a great speed in a suitable environment and produce abnormal stimulation even induce skin diseases. So, it is important to improve antibacterial silk fabric where aesthetic and hygiene are important, and safety is necessary [16-19].

Textiles form the most intimate covering

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of human skin, so it is necessary to support the shielding against UV. Many factors play a role for protection provided by fabrics such as type of fiber, fabric construction, stretching, wetting, fabric cover factor, concentration of dyestuff and types of UV finishing agents [20, 21]. UV finishing agent are divided into organic (soluble) UV absorbers, this type has chromophore systems that absorb the UV portion of sunlight and convert it into non-radiative thermal energy. And inorganic (insoluble) photoactive agents that have an absorption capacity in a UV wavelength between 280 and 400nm; reflects visible rays and infrared rays. UV absorbers based on Oxalamides are the most important groups of UV absorbing compounds [22, 23].

Glycols (Diols) are alcohols containing two hydroxyl groups it includes: ethylene glycol, propylene glycol, glycerol, and polyethylene glycols. When Polyethylene Glycol (PEGs) are cross-linked onto fabric they become water-insoluble and impart several improved functional and aesthetic properties to the fibrous substrate [24]. The Polyethylene Glycol (PEGs) are insolubilized and restricted to the fibers by physical and\or chemical attachment. crosslinked PEG treatment changes fabric properties relative to untreated fabric [25-27].

So the objective of this study was to investigate the effect of the pretreatment of silk fabric with Polyethylene Glycol (PEGs,) dyed with Natural dyes on color strength, Antibacterial activity and UV protection. Silk fabric was dyed with (Natural Red 25 and Natural Yellow 25) dyes using the conventional method.

Experimental Work

Materials

Fabric

100% Silk fabric (83g/m^2) has 32 ends and picks/cm. (1/1) plain weave, was purchased from Al-Gammal Company, Suhag, Upper Egypt.

Chemicals

Sodium sulfate, copper sulfate, potassium aluminum sulfate, polyethylene glycol (PEG) 600 M.W , citric acid and phosphoric acid and a non-ionic detergent were of laboratory grade chemicals.

Dyes

Commercial samples of natural dye powders were obtained from ALPS Industries Ltd, Sahibabad. Indian. Specifications of the dyes are listed in the Table 1.

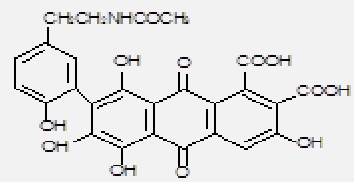
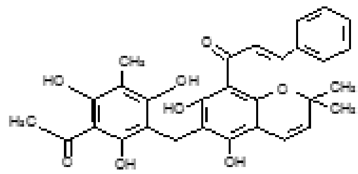
Microorganisms

Microorganisms *Staphylococcus aureus (S. aureus*) Gram-positive and *Escherichia coli (E. coli)* Gram-negative bacteria were used for estimation of antibacterial activities.

Media

Media Nutrient broth/agar medium: contains beef extract (3 g/l), peptone (5 g/l), for a solid medium (15 g/l) agar was added. This medium was sterilized for 20 min at 121°C under pressure

TABLE 1. Natural dyes used.

Common name	Botanical name	Commercial name	C.I.	λmax. (nm)	Chemical structure
Lac	*Lacifer lacca*	Rhine-M	Natural red 25	530	
Kamala	*Mallotus philippinensis*	Basant	Natural yellow 25	400	

Methods

Polyethylene glycol (PEG) treatment

The fabric was weighted and padded through two dips and two nips in the solution containing polyethylene glycol (PEG) concentration 30%. Citric acid concentration 15% to approximately 100% wet pick up on laboratory padder. The fabric was (dry-cure) one step at temperature 120 C° for time 2 minutes. then washed with 2g/L non-ionic detergent 40 C° for 8 min. and rinsed with hot then with tap water and air dry.

Dyeing

Silk fabric samples were dyed using two natural dyes (Natural Red 25 and Natural Yellow 25) and prepare 3 mixtures for each dye via the conventional method.

Natural Red 25 (mixture 1)

The silk fabric sample was immersed in a dye bath composed of the Natural Red 25natural dye shade 13% (o.w.f.), phosphoric acid concentration 4% (o.w.f.), sodium sulphate concentration 8% (o.w.f.),material to liquor ratio L: R (1:50) at temperature 100 C° for 80 minutes. Then The fabric was washed with 2g/L non-ionic detergent 40 C° for 10 minutes . And rinsed with hot water then with tap water and air dried.

Mixture (2): It represented mixture 1 + 8% mordant (copper sulfate).

Mixture (3): It represented mixture 1 + treated silk fabric with PEG.

Natural Yellow 25 (mixture 1)

The silk fabric sample was immersed in a dye bath composed of the Natural Yellow 25 natural dye shade 10% (o.w.f.), L: R (20:1), PH (9) at temperature 80 C° for 40 minutes. Then The fabric was washed with 2g/L non-ionic detergent 40 C° for 8 min. and rinsed with hot then with tap water and air dry.

Mixture (2): It represented mixture 1 +8% mordant (potassium aluminum sulfate).

A mixture (3): It represented mixture 1 +treated silk fabric with PEG.

Testing and analysis

Color strength (K/S)

Color strength (K/S) of the dyed samples was measured on the Mini Scan XE spectrophotometer using Hunter lab universal software, which based on the Kubelka – Munk equation. [28]

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

Where: K, S, and R are the absorption coefficient, scattering coefficient, and reflectance, respectively.

Ultraviolet protection factor (UPF) evaluation

In vitro testing measures ultraviolet (UVR) transmission and the ultraviolet protection factor (UPF) was calculated according to the Australian/ NewZealand Standard (AS/NZS-4399-1996) using UV-Shimadzu 3101-PC-Spectrophotometer. The following equation which based on the percent ultraviolet radiation transmittance through the specimen used to calculate the UPF [28-34].

$$UPF = \frac{\sum E\lambda \cdot S\lambda \cdot \Delta\lambda}{\sum E\lambda \cdot S\lambda \cdot T\lambda \cdot \Delta\lambda}$$

Where:

$S\lambda$ = Solar spectral irradiance *(W/cm^2/nm)
* (Is a function of the amount of solar energy that reaches the surface of the earth of each wavelength).

$E\lambda$ = Relative erythemal spectral effectiveness **
** (Is a weighting spectrum of the action of UVR on the skin for each wavelength).

$T\lambda$= Spectral transmittance of the specimen (measured)

$\Delta\lambda$= Measured wavelength interval or bandwidth (nm.)

Antibacterial activity

Antibacterial activity was quantitatively evaluated against, *Staphylococcus aureus* and *Escherichia coli* according to the AATCC 100 test method [35]. The reduction in numbers of bacteria colonies was calculated using the following equation:

Reduction rate (%) = (A-B)/A*100

Where: A = the numbers of bacterial colonies recovered from untreated fabrics and B = the numbers of bacterial colonies recovered from treated fabrics

Fastness properties evaluation

Lightfastness

The light fastness of dyed samples was determined according to the ISO test method. [36]

Wash fastness

The wash fastness of dyed samples was

determined according to the ISO test method [37].

Perspiration fastness

The perspiration fastness of dyed samples was determined according to the AATCC test method [38].

Physicomechanical measurements

Tensile strength and elongation at break

The tensile strength and elongation at break were determined according to ASTM procedure [39].

Crease recovery angles

The crease recovery angles (CRA) of fabrics were determined according to ASTM procedure [40].

Results and Discussion

From Fig. 1, and Table 2 it can be seen that the highest value of (K/S) was obtained by using Copper Sulfate as a mordant. The ability of copper to coordinate with the dye molecules is much greater than other mordant used in this study, this will lead to higher K/S values [41],as well as PEG modifications enhanced the ability of fabric to dyes i.e. more hydroxyl groups ,forming more hydrogen bonds that improve fabric dye-ability [42].

Fastness properties including washing, perspiration and light were carried out for dyed silk fabric with a different mixture. The data in Table 3 show enhancement in the fastness properties of silk dyed fabric. This may be attributed to that mordants act as a binder by making complex between silk fiber and dye [43].

The impact of different dyeing mixture on tensile strength, crease recovery and wettability of dyed silk fabric is represented in a Table 4. From this data, it can be concluded that the tensile strength of silk fabric has slightly enhancement. Crease recovery of dyed silk fabric enhanced significantly, especially with the PEG mixture.

According to rating scheme the protection categories are non-ratable protection (UPF <15), good protection (UPF >15), very good protection (UPF >30) and excellent protection (UPF >40, 50, 50+). It is supposed that UPF values should be at least 40 to 50+ for apparel and garment applications [44, 45]. In order to improve the UPF values of silk fabric, different dyeing mixtures were prepared and applied to silk fabric. The rate of UV protection of silk fabrics was quantified and expressed via UPF values in the Table 5. It can be seen from the data that there is a remarkable improvement in the UV- protection functionality of dyer silk with all mixtures. The imparted functional property depends on the inherent properties of the fiber, fabric, dye-fiber interactions as well as the absorption capacity of the dyed substrate to the harmful UV-B radiation [46].

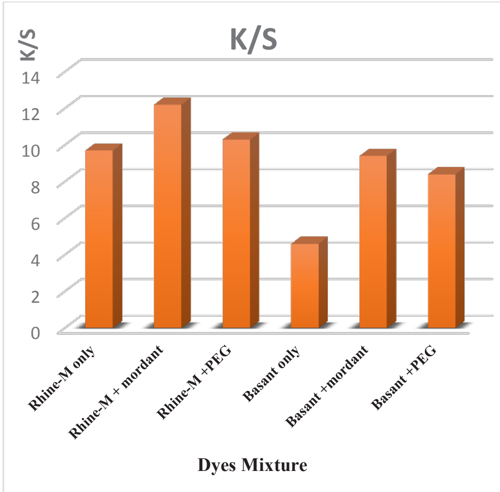


Fig. 1. Effect of the different mixture of dyeing bath on color strength (K/S) of dyed silk fabric.

TABLE 2. effect of the different mixture of color obtained.

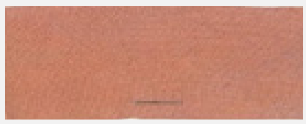
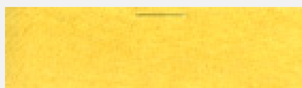
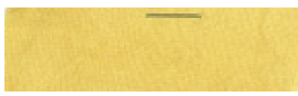
Dye mixture	Color obtained
Rhine M+ mordant	
Rhine M+PEG	
Rhine M Only	
Basant +mordant	
Basant +PEG	
Basant only	

TABLE 3. Effect of the different mixture of dyeing bath on fastness properties of dyed silk fabric.

Mixtures	Washing fastness			Lightfastness	Perspiration fastness					
					Alkali			Acid		
	Color change	Staining on polyester	Staining on wool		Color change	Staining on polyester	Staining on wool	Color change	Staining on polyester	Staining on wool
Natural Red 25 dye only	4-5	4	4	6	4	4-5	4-5	4	4-5	4-5
Natural Red 25 dye + mordant	4-5	4-5	4-5	7	4-5	4-5	4-5	4-5	4-5	4-5
Natural Red 25dye + PEG	4-5	4-5	4-5	7	4-5	4-5	4-5	4-5	4-5	4-5
Natural Yellow 25only	4-5	4-5	4	6	4-5	4-5	4-5	4-5	4	4
Natural Yellow 25+ mordant	4-5	4-5	4-5	7	4-5	4-5	4-5	4-5	4-5	4-5
Natural Yellow 25+ PEG	4-5	4-5	4-5	7	4-5	4-5	4-5	4-5	4-5	4-5

TABLE 4. Effect of the different mixture of dyeing bath on tensile strength and crease recovery of dyed silk.

Sample	Tensile strength (kg/ cm)	The elongation %	Crease recovery (CRA)^o
Blank sample	40.7	9.7	300
Silk fabric dyed with Natural Red 25only	40.5	12.3	320
Silk fabric dyed with Natural Red 25+ mordant	42.4	11.3	330
Silk fabric dyed with Natural Red 25+ PEG	42	12	350
Silk fabric dyed with Natural Yellow 25only	41.3	13.3	310
Silk fabric dyed with Natural Yellow 25+ mordant	41.2	11.7	310
Silk fabric dyed with Natural Yellow 25+ PEG	40.3	12.6	321

TABLE 5. Effect of the different mixture of dyeing bath on UPF values of dyed silk fabric.

Sample	UPF	Protection categories
Blank sample	15.70	good
Silk fabric dyed with Natural Red 25only	39.07	Very good
Silk fabric dyed with Natural Red 25+ mordant	47.35	Excellent
Silk fabric dyed with Natural Red 25+ PEG	38.84	Very good
Silk fabric dyed with Natural Yellow 25only	42.12	Excellent
Silk fabric dyed with Natural Yellow 25+ mordant	66.90	Excellent
Silk fabric dyed with Natural Yellow 25+ PEG	38.12	Very good

From the mentioned data in Fig. 2 we found that Natural Red 25dye has an antibacterial effect. This may be due to the Naphthoquinone pigment. Naphthoquinones are thought to function as an antibacterial by targeting surface. Exposed adhesions, cell wall polypeptides, and membrane-bound enzymes in the microbial cells leading to inactivation of protein and thus a loss of function. [47, 48]. It's presumably because there is a net-like structure between polyethylene glycol (PEG) and crosslinking agent and the fiber that would tightly bind the fiber and restrict the latter to loosen up during regular use [49]. Antibacterial activity, expressed as growth reduction of the microorganisms, could be explained as follows: The hydroxyl groups in PEG interfere with the bacterial metabolism by stacking at the cell surface and binding with DNA to inhibit m-RNA synthesis [50]. Silk fabrics treated with PEG and dyed with the Natural Red 25dye (natural dye+ PEG) display high growth reduction of bacteria.

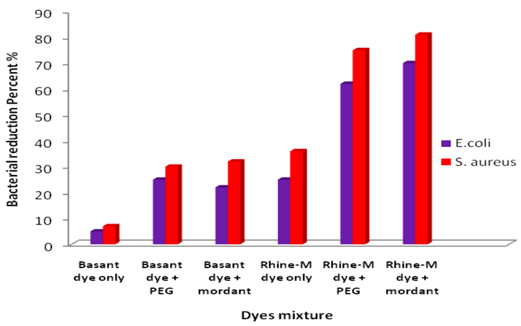


Fig. 2. Effect of the different mixture of dyeing bath on antibacterial properties of dyed silk fabric.

Conclusion

Recently the demand for natural fibers having various functionalities has increased considerably. To introduce functionalities such as UV protection function and antibacterial activity into the natural fabric like The queen of textiles silk, The present work shows that, when the fabric was dyed with a natural dye (Rhine-M, Natural Yellow 25 with mordants and PEG) to improve a number of different performance feature. The test results refer to significant enhancement of UPF values ("excellent" protection category), improved dyeability and possibility for minimizing cost and pollution through mordant free finishing and dyeing. The treatments applied also enhanced the light and wash fastness of dyed fabrics. Also, there was a good enhancement in the antibacterial activity especially with Rhine M + PEG. The final modified silk fabric obtained in this study could have possibility as apparel textiles showing protection against UV radiation and antibacterial properties.

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استخدام الأصباغ الطبيعية لصناعة الحرير متعدد الوظائف

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كلية البنات للآداب والعلوم والتربية - جامعة عين شمس - القاهرة - مصر.

ظهر مؤخرا الاهتمام في عمليات المعالجة الرطبة المختلفة للمنسوجات ابتداءً من العملية التحضيرية الأولية وحتى الملابس الجاهزة بما يسمى التكنولوجيا الخضراء. حيث يتم استبدال العديد من المواد الكيميائية التقليدية غير الصديقة للبيئة بالمنتجات الطبيعية الآمنة للبيئة والصحة أثناء التصنيع والاستخدام. فقد تم استخدام الأصباغ الطبيعية بدلا من الأصباغ الاصطناعية بسبب الظروف البيئية حيث تعتبر الصبغات الطبيعية آمنة بالنسبة للمنسوجات لتحل محل الأصباغ الاصطناعية ، بسبب عدم تلوثها ، وهي غير مسرطنة وصديقة للبيئة. يعتبر الحرير هو الأخف والأرق والأنعم بين الألياف الطبيعية وله العديد من التطبيقات في مجال الغزل والنسيج.

في هذا البحث، كان النسيج الحريري المعالج مسبقًا ب عديد الايثيلين جليكول مصبوغًا بصبغتين طبيعيتين مختلفتين، ثم قورنت الأنسجة مع تلك الصبغيات الأصباغ الطبيعية فقط ومع تلك الأصباغ والأعاصير. توفر الخصائص الوقائية مثل حظر الأشعة فوق البنفسجية تحسينًا ملحوظًا في قيم UPF. كما تم تعزيز نسبة تخفيض البكتيريا. تحسين كبير في قيم قابلية الصباغة تظهر قيم K / S. أيضا، أظهر المزيج مع عديد الايثيلين جليكول قوة شد جيدة، تعزيز قابلية التبلل وأيضا لاسترجاع زوايا التجعد.