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Cleaner Production of Dyed and Functional Polyester Using Natural Dyes vis-a-vis Exploration of Secondary Shades

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ABSTRACT

Natural dyes are known for their added benefits over synthetic dyes in terms of health and ecology. They are considered to present a limited range of shades, which limits their use in high-end value-added products. Thus, to explore natural dyes on large scales, it is essential to generate a wide range of shades like synthetic dyes. Therefore, it is important to explore the secondary shades of natural dyes especially those which can act as primary colors in the recipe (red, yellow and blue). In the present work, three natural dyes, namely indigo, pomegranate peels and kumkum were used to dye the polyester fabric. Along with color strength at different dye concentrations, functional properties such as antioxidant, antibacterial, and UV protection were also evaluated. The dyed polyester showed good color values (K/S>1.7) and also displayed satisfactory color fastness to light, washing and rubbing. The polyester fabric dyed with pomegranate and kumkum in self-shades displayed a variety of functional properties including a bacterial reduction of more than 90%, radical scavenging activity of greater than 90% and also displayed excellent UPF values (UPF > 270). Indigo-dyed samples displayed antibacterial activity and UV-protection; however, their antioxidant activity was poor. The secondary shades of these dyes were produced on polyester fabric. Prog. Color Colorants Coat. 14 (2021), 121-128© Institute for Color Science and Technology.

1. Introduction

Textile coloration using natural dyes is gaining momentum due to revival in the interest regarding sustainable chemical processing and awareness regarding toxic effects associated with some of azo dyes, especially those based on banned amines. This has led to increased exploration of natural resources for the coloration of textiles. Most of the studies in this area were done on cotton, wool, silk, nylon, jute, linen etc. Utilization of natural dyes for dyeing and functional finishing of textiles is widely reported in the literature [1–10].

Punica Granatum, which is commonly known as pomegranate, belongs to the Punicaceae family. It is a traditional medicine for diarrhoea and respiratory pathologies [11, 12]. Its basic components are gallocatechin, delphinidin, cyanidin, gallic acid, ellagic acid, pelargonidin and sitosterol [13]. Pomegranate and its chemical components offer many useful properties, including antibacterial activity [14], anti-inflammatory activity, antioxidant activity [15], anti-angiogenesis, and anti-cancer properties [13]. *Mallotus philippensis,* commonly known as Kumkum or Kapila, is a traditional drug used in India for the treatment of

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typhoid and stomach disorders [16, 17]. The basic component present in the dye is Rottlerin (1-[6-[(3-acetyl-2,4,6-trihydroxy-5-methyl phenyl) methyl]-5,7-dihydroxy -2, 2-dimethyl - 2H-1-benzopyran -8 -yl] -3-phenyl - 2 propen- 1 -one). It also contains different compounds, such as diterpenoids, phenols, flavonoids, steroids, coumarins, and triterpenoids [16]. Interesting biological activities of kumkum such as anti-oxidant activity, anti-microbial activity, cytotoxicity, antiviral activity, immunoregulatory activity, and anti-inflammatory activity were reported [17, 18]. *Indigofera Tinctoria,* also known as indigo blue, is a traditional dye associated with denim clothes and blue jeans [19]. The basic components present in *Indigofera tinctoria* leaf extract are tannins, phenols, saponins, terpenes, flavonoids, anthroquinonesence and steroidal. These chemical compounds possess various pharmacological properties such as anti-oxidant activity, anti-bacterial activity and cytotoxicity activity [20].

Polyester, being a synthetic fiber, is a less expensive option and has many advantages over other fibers like wrinkle-resistance, quick-drying and better resistance to microbial attack. Polyester fiber is widely used in apparels and technical textiles and the functional coloration of such substrate using natural dyes would be interesting. Sen et al. attempted dyeing of various polyester fibres like poly-trimethylene terephthalate (PTT) and poly-lactic acid (PLA), and poly-ethylene terephthalate (PET) with lac dye in presence of catechu as a bio-mordant [21]. Elnagar et al. dyed the polyester and nylon fabrics with curcumin and saffron natural dyes using UV/ozone pretreatment to activate fiber and improve the dyeability of the fabrics [22]. Bhuiyan et al. dyed the polyester fiber with henna as a natural dye without using metallic mordants [23]. Arain et al. provided an eco-friendly dyeing process for dyeing of polyester fabric with henna dye using microwave-assisted technology [24]. Abate et al. used supercritical CO_2 for bio-activation and coloration of polyester fabric with curcumin without mordant and achieved an excellent color strength and fastness properties [25]. Shahidi et al. modified the polyester fabric by dielectric barrier discharge and the dyeability of modified fabric with natural dyes was studied [26]. Zohdy used gamma radiations to improve the dyeability of polyester [27]. Some researchers have also used various novel techniques for natural dyeing of natural and synthetic fibers [28–32].

Even though natural dyeing of textiles is reported widely, the reports regarding simultaneous dyeing and functional finishing of polyester are reported to a limited extent. The mixing of natural dyes to obtain a variety of shades on polyester to overcome the limitation of natural dye in terms of availability of limited shades is not reported. In the present work, three natural dyes namely indigo, kumkum and pomegranate were utilized for simultaneous dyeing and functionalization of polyester. The coloration and functional properties imparted to polyester using an application of such natural dyes were explored. Secondary shades of these dyes were also produced on polyester fabric to explore different shades.

2. Experimental

2.1. Materials

Polyester fabric was obtained from Toyota Water Jet machine with specifications 3937 ends/m and 2520 picks/m. The fabric was scoured using 2gpl detergent and 1gpl soda ash and further used for dyeing. Three natural dyes used were indigo (*Indigofera Tinctoria*), pomegranate peel (*Punica Granatum*) and kumkum (*Mallotus philippensis*). All were purchased in powder form from an Oriental drug store. The natural dye extraction was carried out in ethanol using soxhlet extraction for 12h. The dye powder was obtained by evaporating ethanol from the extract.

2.2. Methods

2.2.1. Dyeing of polyester fabric using natural dyes

The dyeing process was carried out via high-temperature high pressure (HTHP) method at 130 ^{o}C for 45 min in the Infrared Dyeing Machine. The polyester samples were dyed using different natural dyes (in various shade %) in acidic pH (4.5). The material to liquor ratio was kept as 1:10 and dispersing agent (1%) was used to prepare the dispersion of natural dye powder. Secondary shades were produced by mixing the natural dyes in weight proportions and maintaining the total shade percent as 1, 5 and 10%.

2.2.2. Measurement of color values

The color strength of dyed samples was compared by measuring K/S values [33] which were evaluated on

Gretag Macbeth Color Eye 7000A Spectrophotometer. The K/S values were assessed using the Kubelka-Munk equation (Eq. 1):

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

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

2.2.3. Estimation of color fastness

Color fastness of the dyed fabrics to washing, rubbing and light was evaluated using ISO105-C06, ISO-105X12, and ISO105B02 test methods, respectively [34].

2.2.4. Evaluation of functional properties

The dyed polyester fabrics were analyzed for antioxidant activity as per the method reported in the literature [7], UV-protection as per AS/NZS 4399:1996 standard [35], and antibacterial activity against *S. aureus* as per AATCC-100 test method [36].

3. Results and Discussion

3.1. Color fastness of dyed polyester

Fastness to light, rubbing and washing (color change and staining) were evaluated for all the dyed samples (Table 1). As shown in Table 1, all the dyed samples showed significant light fastness, with ratings of 5 and more. It shows a good resistance of dye on dyed polyester against fading in the presence of light. All the dyed samples showed excellent washing fastness in terms of both color change and staining, as indicated by ratings of 4 to 5. The rubbing fastness was also found in the range "good" to "very good". The results show satisfactory color fastness to light, washing and rubbing which might be attributed to the mechanism of dyeing of polyester using HTHP dyeing process. The dyes were applied on polyester through dispersion using HTHP method of dyeing. HTHP method utilizes high temperature to open the fibre structure which allows the dye to penetrate inside the core of the fibre. After dyeing, the bath is cooled which ensures the dye getting entrapped inside the fibre. This is very much similar to solid solution dyeing in case of disperse dyeing of polyester. The surface dye was removed by reduction clearance process after dyeing. This provides excellent resistance to color-bleeding in the presence of washing liquor, abrasive rubbing and light.

3.2. Functional properties of the dyed polyester

The polyester dyed with kumkum and pomegranate dyes showed excellent antioxidant activities, as shown in Table 2. The antioxidant activity of DPPH is based on the stability of the material. The odd electron of DPPH is responsible for a strong absorption at 517 nm. DPPH can accept electron donated by an antioxidant compound which further decolorizes the DPPH. The change in absorbance of decolorized DPPH can be quantitatively measured at 517 nm using UV-Visible spectrophotometer [37]. The dyed polyester, especially dyed with kumkum and pomegranate, exhibited excellent antioxidant property which might be because of the presence various compounds in natural dyes which are able to stabilize DPPH. They are electron-rich resonating centers which tend to avoid further oxidation and change the dye color. Indigo-dyed polyester showed significantly lower antioxidant activity as compared to other dyed samples.

Table 1: Color fastness of fabric samples dyed with indigo, kumkum and pomegranate dyes (5% shade).

S. No	Samples	Light Fastness	Rubbing Fastness	Washing Fastness	
				Staining	Color change
1	Kumkum	5	3-4	4	5
2	Indigo	5-6	3-4	4	4
3	Pomegranate	5	4	4	4

Table 2: Functional properties of polyester dyed with indigo, kumkum and pomegranate dyes (5% shade).

S. No	Samples	Radical scavenging activity (%)	UPF	Antibacterial activity (%)
1	Control	Negligible	42.97	-
2	Kumkum	91.44	322.07	85.87
3	Pomegranate	93.97	277.30	90.55
4	Indigo	43.82	198.40	83.82

The UV-protection properties of dyed fabrics were evaluated by measuring the UV-protection factor (UPF). UPF of the polyester fabric increased after dyeing with the three dyes, as shown in Table 2. As opposed to the UPF value of 42.97 for undyed fabric, dyed fabrics showed significant improvement in the UV-protection property. This behavior might be related to the introduction of dyes inside the fiber structure which produce hindrance in the ease of transmittance of UV radiation. UPF value of undyed polyester was also obtained which clearly indicates the dependence of UPF on the fiber properties. The presence of aromatic ring in the backbone of the polymer and the additive, which are added during fiber manufacturing, impart good UV-protection to the polyester. The antibacterial property of all the dyed samples was determined using the AATCC 100 method.

Table 2 shows the reduction of bacterial colonies by each dyed sample. All the dyed samples showed considerable antibacterial properties with the bacterial colony reduction of higher than 83%. The site and the number of hydroxyl groups on the phenolic components of dyes may increase the toxicity against the microorganisms. The phenolic groups in the dyes interfere with bacterial metabolism and inhibit their growth [17, 38]. Hence, all the dyed samples displayed a satisfactory antibacterial activity.

3.3. Primary and secondary shades of natural dyes on polyester

3.3.1. Color strength

Table 3 summarizes the L^*, a^*, b^* and K/S values of dyed samples at 5% shade. Here, L^* is lightness (0 = Black; 100 = White), a* is redness-greenness and b^* is yellowness-blueness.

In the secondary mixture of kumkum and indigo, the increase in kumkum concentration at the expense of indigo resulted in a decrease in L^* value indicating the increase in darkness of shade. This also resulted in a decrease in b* indicating the increase in the blueness of shade. All the mixed shades of kumkum and indigo showed a negative value of a* indicating the greenness of the obtained shades. The mixing of dyes resulted in beautiful shades with varying colour values. In most of the secondary shades, the dye present in a higher proportion showed the color dominance of the dyed fabric. The variation in dye concentration in binary mixture resulted in variations of tones and the lightness of shade (Table 3).

As shown in Table 1, the K/S values varied with varying the dye concentrations in the mixtures. The secondary mixture of pomegranate and kumkum showed higher K/S values as compared to other mixtures. In general, it can be concluded that the various dyes used for obtaining secondary shades showed good compatibility which resulted in a variety of shades on polyester fabric. Hence the suitability of these dyes for dyeing polyester in primary and secondary shades is confirmed.

3.4. Shade card formation

Nine mixtures were prepared with given dye proportions [(25% K, 75% I), (50% K, 50%I), (75% K, 25% I), (25% K, 75% P), (50% K, 50% P), (75% K, 25% P), (25% P, 75% I), (50% P, 50% I), (75% P, 25% I)]. All these dye mixtures were used to dye the polyester fabrics at different shade percentages, i.e. 1, 5 and 10%. Consequently, several different colors were observed in shade cards. Table 4 (a, b, c, and d) shows the shade cards for secondary shades, where the color of the fabric is gradually changing with a change in dye concentration. These colors and compositions are important for obtaining various desired shades. As shown in Table 4, the color changes into more bluish as indigo concentration increases; and it changes into brownish when the concentration of pomegranate dominates. Similar results were obtained for other combinations, and consequent shade cards with various shades were prepared with various shades.

Moreover, it was observed that the mixing of these dyes showed uniform and homogenized colors and shades without getting any patches, dark and dull places. Thus, it can be concluded that using a mixture of natural dyes at different concentrations and proportions may generate a huge number of colors, shades and tones. Mixing of primary colors to get a variety of shades is a general practice in the textile industry. Natural dyes were used as primary colors to produce a variety of shades. This is particularly encouraging as various natural dyes can be mixed to get a wide spectrum of shades on textile materials.

Table 3: Color values and colour strengths of dyed samples at 5% shade.

S. No	Dye concentrations	L*	a*	b*	K/S
1	(100% I)	57.79	-6.15	-5.19	1.75
2	(100% K)	70.81	1.71	25.84	8.80
3	(100% P)	57.04	4.79	15.94	4.40
4	(25% K, 75% I)	70.05	-8.87	-5.04	1.78
5	(50% K, 50% I)	60.47	-12.13	-0.31	1.71
6	(75% K, 25% I)	54.89	-5.94	2.32	1.81
7	(25% P, 75% I)	57.37	-6.57	-5.13	1.75
8	(50% P, 50% I)	55.75	-5.95	1.17	1.80
9	(75%P, 25% I)	50.34	-4.02	4.14	2.26
10	(25% P, 75% K)	63.39	2.99	21.03	8.01
11	(50% P, 50% K)	61.00	2.75	16.97	5.05
12	(75% P, 25% K)	65.94	1.98	20.50	6.80

I: Indigo, K: Kumkum, P: Pomegranate

Table 4: Shade Cards for secondary shades

a): Primary and secondary shade with 1% owf dye concentration

S. No	Dye Mixture	(100, 0)	(75, 25)	(50, 50)	(25,75)	(0, 100)
1	Indigo + Pomegranate					
2	Indigo + Kumkum					
3	Kumkum + Pomegranate					

b): Primary and secondary shade with 5% owf dye concentration

S. No	Dye Mixture	(100, 0)	(75, 25)	(50, 50)	(25,75)	(0, 100)
1	Indigo + Pomegranate					
2	Indigo + Kumkum					
3	Kumkum + Pomegranate					

c): Primary and secondary shade with 10% owf dye concentration

S. No	Dye Mixture	(100, 0)	(75, 25)	(50, 50)	(25,75)	(0, 100)
1	Indigo + Pomegranate					
2	Indigo + Kumkum					
3	Kumkum + Pomegranate					

4. Conclusions

The polyester fabrics were successfully dyed with natural dyes. The results showed that the natural dyes showed excellent compatibility with polyester fibers. The polyester fabrics dyed with kumkum and pomegranate dyes showed significant antioxidant properties (radical scavenging >90%), UV-protection (UPF>270), and also exhibited appropriate antibacterial activity against *S. aureus* (bacterial colony reduction >85%). The polyester dyed with indigo dye also displayed significant UV-protection and antibacterial properties; however, it showed inferior antioxidant properties compared to polyester dyed with kumkum and pomegranate dyes. The polyester fabrics dyed with indigo, kumkum, and pomegranate dyes demonstrated good washing (rating 4 and above), rubbing (ratings 3-4 and above) and light (rating 5 and above) fastnesses. At a higher level, these dyes can be used with their sustainable nature, unique shade character and functionalities, which are also compatible with strong, wrinkle-resistant and economically feasible polyester. A variety of colors can be obtained by changing the percentages and proportions of the dyes.

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