COLOR STRENGTH AND COLOR DIFFERENCE OF COTTON AND SILK FABRICS DYED WITH EXTRACTS FROM BUTTERFLY PEA FLOWERS

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Natural dyes have always been of interest due to their non-toxicity and environmental benefits. In this work, cotton and silk fabrics were dyed with extracts from butterfly pea (BP) flowers. Accordingly, the effects of pH value, liquor ratio, dyeing concentration, temperature, exhausting time and mordanting agents on the color strength (K/S) and the color difference (ΔE) of cotton and silk fabrics dyed with BP extracts were investigated. The experimental results showed that the K/S and ΔE values of dyed cotton fabrics were much higher than those of dyed silk fabrics. The research also established the most suitable conditions for dyeing cotton and silk fabrics with BP extracts as being the following: pH of 6.0, liquor ratio of 1:5, dyeing time of 60 min, at 70 °C for cotton and 60 °C for silk. Notably, four different types of mordants, including potassium aluminium sulfate ($KAl(SO_4)_2.12.H_2O$), copper sulfate ($CuSO_4.5H_2O$), ferrous sulfate ($FeSO_4.7H_2O$) and tannin, were used to enhance dyeability of cotton and silk fabrics with BP extracts. There were slight differences in the ΔE values of mordanted and unmordanted samples, while the K/S values improved significantly, especially in the samples mordanted with tannins. Color fastness and color staining to washing and rubbing were also examined, indicating that treated silk fabrics achieved higher values than treated cotton fabrics.

Keywords: butterfly pea (BP), color difference (ΔE), color strength (K/S), dyeability, cotton fabric, silk fabric

INTRODUCTION

Butterfly pea (BP) – *Clitoria ternatea* – is known as an ornamental plant, as well as a type of vegetable, with a striking color of flowers, which is widely grown in South Asian and Southeast Asian (also called as Asian pigeon wings or blue pea). Many beneficial applications of butterfly pea plants have been explored in the agriculture and the medical fields, including as fodder, eco-insecticide and decorative plantings, as traditional cure in medicine, as well as in desserts, beverages, food colorants, *etc*.[1-3] The chemical composition of BP flowers mainly includes triterpenoids, flavonoid glycosides, anthocyanins and steroids.[4] Among them, anthocyanins are an important compound generating various colors and bioactive effects.[5]

In recent studies, two types of anthocyanins, including ternatin and delphinidin, have been successfully identified by ultra-performance liquid chromatography (UPLC) coupled with ultraviolet and mass spectrometry.[4,6] In previous works, excellent pharmacological effects of BP extracts were clarified, such as antimicrobial, antioxidant, hypolipidemic, anticancer and anti-inflammatory ones.[7-10] The methanol extracts of BP leaves, stems, flowers, seeds and roots were demonstrated to fight against several species of bacteria, yeasts and filamentous fungi, owing to bioactive compounds, such as tannin, flavonoid, anthraquinone *etc*.[11-14] It was demonstrated that BP extracts exhibited potent inhibitory effects, compared to other extracts. Also, the antioxidant activity of BP extracts *in vitro* was quantitatively assessed through total phenolic content (TPC) and total flavonoid contents (TFC), offering possible avenues for therapeutic applications.[15,16]

Moreover, the antioxidant activity was proved to be significantly influenced by the pH of the medium.

In recent years, the demand for natural colorants has increased because synthetic pigments have been shown to cause many serious problems for human health and the environment. In fact, BP powders are successfully commercialized as food and cosmetic colorants, and have quite good thermal stability due to the presence of poly-acylated anthocyanins, with multiple aromatic acyl groups (as shown in Fig. 1). Both anthocyanin and anthocyanidin are flavonoids, however, anthocyanidin is the sugar-free counterpart of anthocyanin, the latter being a soluble pigment (a subclass of the polyphenol family). The poor stability of anthocyanins leads to strong degradation and reduction, because anthocyanin molecules are modified by chemical processes, such as hydroxylation (generating blue with an increasing number of hydroxyl groups), methylation (causing red shifting), glycosylation (leading to hypsochromic shifting) and acylation (increasing structural stability and generating blue colour).[17,18]

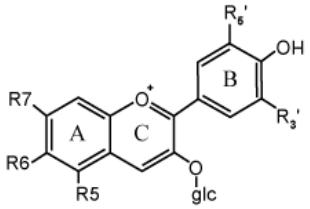

Figure 1: Basic chemical structure of poly-acylated anthocyanin[19]

Many studies demonstrated that BP extracts contain a significant content of anthocyanins. Accordingly, the pigments and the antioxidant ability of BP extracts were shown to be affected by pH, resulting in obvious color changes.[20] Thus, anthocyanins are known as pH indicators, due to their susceptibility to medium pH. It was reported that the optimum antioxidant activity is achieved in acidic BP extracts, namely at pH values from 3.2 to 5.2.[20] Interestingly, BP extracts were red in acidic solution, blue in neutral solution and green in basic solution.[21] The authors have also analyzed the dye concentration in petals of BP flowers through UV-Vis spectrophotometry to see if it can be influenced by various factors, such as extracting time, temperature, amount of flower and pH value.[22] In addition, the BP powders have been investigated as natural sensitizers in solar cells as anthocyanin pigments have been found to increase the power conversion efficiency and environmentally friendly features.[23]

Recently, several natural pigments, extracted from plants such as turmeric, onion, curry, neem, mango, grape, henna *etc*., have been investigated to color cellulose and protein fibers (especially, cotton, silk and wool), due to the biodegradability or lower environmental impact and health benefits (*e.g*., antibacterial, antioxidant, ultraviolet resistance) associated with natural dyes.[24-28] Some authors have reported on dyeing cotton, silk and synthetic fabrics with anthocyanin dyes in the presence of metal mordants, such as ferrous sulfate and copper sulfate.[29,30] However, to the best of the authors' knowledge, no work has been reported so far to tackle the dyeability of different fibers, specifically, protein and cellulose fibers, with BP extracts. In this study, cotton and silk fabrics were dyed with BP extracts, under varying conditions, and quantitative spectrophotometric measurements have been conducted to determine the dyeability of the fabrics, in terms of color strength (K/S) and color difference (ΔE).

EXPERIMENTAL

Pure cotton woven fabrics (200 grams per square meter) and silk woven fabrics (40 grams per square meter), with 24 and 45 threads per inch (TPI) lengthwise and crosswise, respectively, were used in this work. Both cotton and silk fabrics were first bleached and weighed before the dyeing process (2 g for cotton specimen and 0.5 g for silk specimen).

BP flowers were purchased from farms in Long An Province (in South Vietnam). BP flowers were separated and ground into small particles, dried and stored before extracting in ethanol solvent at various concentrations and liquor ratios. All BP powders were dispersed into distilled water and extracted through the microwave-assisted extraction methods to obtain the required mixtures.

Four common mordants, including potassium alum sulfate ($KAl(SO_4)_2.12H_2O$, copper sulfate ($CuSO_4.5H_2O$), ferrous sulfate ($FeSO_4.7H_2O$), and

tannic acid, were used to enhance the dyeability of cotton and silk fibers. The exhaust dyeing process with simultaneous mordanting was carried out (*i.e.*, mordanting while dyeing). The pH value, temperature, liquor ratio, dye concentration, exhausting time and mordant concentration were examined in the following ranges: 5.0 to 7.0, 60 to 90 °C, 1:5 to 1:20, 25 to 100%, 60 to 90 min and 1 to 3% owf of mordants, respectively.

The following experimental equipment was used: a dyeing machine (Dai Phuoc machinery Ltd.), a dyer (Gavazzi S.L.R.), a magnetic stirrer (Velp Scientifica), an electronic scale (Ahaus, ±0.001 g), a spectrophotometer (X-rite Color i5D, λ = 360-750nm, d=10 nm).

The dried petals collected from BP flowers (80 g) were separated, ground and immersed in 400 mL of ethanol (70%) for 60 min at 30 °C. The BP extracts were obtained after 2 filtering times.

Color measurements were carried out using the CIELAB color space, with three coordinates of L, a and b. Accordingly, color strength (K/S) was calculated by the Kubelka-Munk equation:

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

where R is the reflectance at the wavelength of 580 nm.

Color differences (ΔE) of the dyed samples were calculated according to the distance between two color coordinates in the CIELAB color space as follows:

$$\Delta E = \sqrt{(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2} \quad (2)$$

All the K/S and ΔE measurements were carried out in triplicate to obtain the average value; the ratio of standard derivation (σ) to the average value should be less than 0.05.

The color fastness and color staining to crocking and washing were examined according to the ISO 105 test method, using the grey scale with 10 evaluating levels (1.0, 1.5, 2.0, 2.5, 3.0, 3.5, 4.0, 4.5 and 5.0).

RESULTS AND DISCUSSION

Effect of pH value on K/S and ΔE of cotton and silk dyed with BP extracts

As mentioned in the previous section, the anthocyanin compound was established to be a pH indicator, due to its sensitivity, in terms of color changes, to the pH value.

Therefore, the dyeing medium pH was expected to strongly impact the color of cotton and silk fabrics dyed with BP extracts. Three varying levels of pH – from 5.0 to 7.0 – were prepared by adding 0.1M NaOH to the dye solutions, as reported in Table 1.

It was noted that, generally, at the same pH – in acidic medium, the K/S values of treated silk were lower than those of treated cotton. Notably, the K/S value of dyed cotton at pH 6.0 (*i.e.*, sample CTN-6) is 2.45, much higher than that of dyed silk (*i.e.*, K/S of only 0.76). When cotton fabrics were dyed in neutral medium (pH = 7), as well as in strong acid medium (pH = 5), their dyeability with BP extracts was quite low. Accordingly, the treatment of cotton fabrics with BP extracts should be carried out in slightly acid medium (pH = 6.0) to ensure the highest K/S value. Meanwhile, the dyed silk samples exhibited a slight change of K/S in the pH range from 5.0 to 7.0, particularly, obtaining the highest value at the pH of 6.0, similarly to the cotton samples. Obviously, the acidic medium somewhat loosens the molecular structure of cotton fibers (especially in the amorphous region), where the colorants could penetrate more deeply to form linkages. Reversely, such effects did not happen clearly in the silk structure because of its inherent amphoteric nature, where the acidity was more prominent.

When considering the effect of pH value on ΔE, both dyed cotton and silk at low acid level presented the highest ΔE values, of 33.60 and 15.25, respectively. Remarkably, the difference in ΔE between cotton and silk samples dyed with BP extracts was very high, up to 18.53, also indicating that the dyeability of cotton was better than that of silk (the optimized pH value is 6.0). Therefore, such pH value will be fixed in this study to evaluate the effects of other factors on the dyeability of both cotton and silk, due to their most obvious difference from reference samples.

Table 1
Effects of pH value on K/S and ΔE of dyed cotton and silk fabrics with BP extracts

Medium	pH	Sample	Average value				ΔE_{CTN}-ΔE_{SLK}	K/S_{CTN}-K/S_{SLK}
			ΔE	σ(ΔE)	K/S	σ(K/S)		
High acid	5.0	CTN-5	17.35	0.562	0.77	0.025	6.87	0.13
		SLK-5	10.48	0.402	0.64	0.029		
Low acid	6.0	CTN-6	33.60	1.176	2.45	0.093	18.35	1.69
		SLK-6	15.25	0.641	0.76	0.026		
Neutral	7.0	CTN-7	7.67	0.265	0.35	0.013	-1.11	-0.19
		SLK-7	8.78	0.360	0.54	0.022		

Table 2
ΔE and K/S of cotton and silk fabrics dyed with BP extracts at liquor ratios of 1/5, 1/10 and 1/20

Sample	Liquor ratio (LQ)	L	a	b	Average value			
					ΔE	σ(ΔE)	K/S	σ(K/S)
CTN1/5	1:5	-35.13	4.05	-20.31	33.60	1.176	2.43	0.078
SLK1/5	1:5	-15.70	0.68	-10.12	15.25	0.488	0.76	0.028
CTN1/10	1:10	-24.17	-1.99	-12.69	20.91	0.721	1.06	0.045
SLK1/10	1:10	-16.88	-1.85	-6.19	10.36	0.425	0.50	0.017
CTN1/20	1:20	-16.49	-0.12	-11.25	17.70	0.644	0.55	0.021
SLK1/20	1:20	-12.58	-1.63	-3.70	6.83	0.235	0.34	0.014

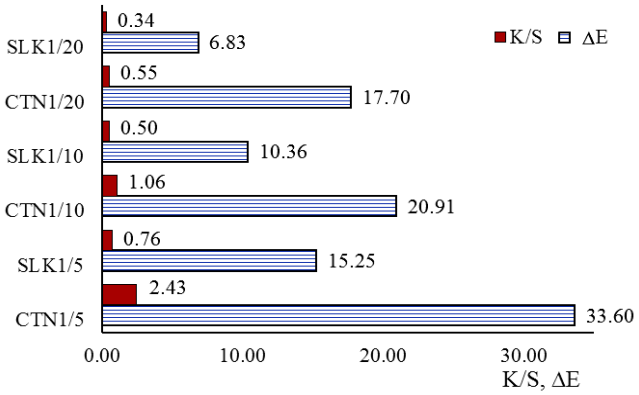

Figure 2: K/S and ΔE of cotton and silk fabrics dyed with BP extracts (70%) at various liquor ratios (1/5, 1/10 and 1/20)

Table 3
K/S and ΔE values of cotton and silk samples dyed with 25, 50, 75 and 100% of BP extracts at liquor ratio of 1/20 and pH of 6.0

Sample	Dye concentration (%)	Average value			
		ΔE	σ (ΔE)	K/S	σ (K/S)
CTNC25	25	29.37	0.952	1.19	0.050
SLKC25	25	10.38	0.399	0.38	0.016
CTNC50	50	31.95	1.118	2.21	0.071
SLKC50	50	16.41	0.689	0.76	0.028
CTNC75	75	35.12	1.212	3.56	0.150
SLKC75	75	18.84	0.772	1.01	0.035
CTNC100	100	32.94	1.199	3.26	0.123
SLKC100	100	17.29	0.595	0.99	0.041

Effect of liquor ratio on K/S and ΔE of cotton and silk dyed with BP extracts

To observe the color changes of dyed cotton and silk with BP extracts in ethanol, the liquor ratios (LQ) of 1/5, 1/10 and 1/20 were investigated. The obtained results for K/S and ΔE values of the dyed fabrics are presented in Table 2 and Figure 2. Certainly, the K/S and ΔE values rose with increasing LQs due to higher contents of BP colorants, achieving 2.43 and 33.6 for cotton, and 0.76 and 15.25 for silk at LQ of 1/5, respectively. Moreover, it can be seen that all K/S and ΔE values of cotton were higher than those of silk, showing that the dye absorbance of cotton fibers was better owing to higher amorphous regions, as well as a larger amount of hydrogen bonds. Such difference was most clearly expressed at the LQ of 1/5. Consequently, the colorants (mainly, anthocyanins) in the BP extracts penetrated into the cellulose structure more deeply, compared to the protein structure.

Effect of dye concentration on K/S and ΔE of dyed cotton and silk

Similarly to the liquor ratio, the difference in dye concentration significantly impacted on the dyeability of dyed cotton and silk fabrics with BP extracts, as shown in Table 3. Anthocyanins are

polycyclic organic compounds, which are well soluble in ethanol, resulting in high extraction efficiency, with a reasonable concentration to ensure high polarization. Therefore, various dye concentrations could cause differences in the diffusion of anthocyanins in aqueous extracts. In the present work, the effects of varying dye concentration were investigated while the other reaction conditions were kept constant at: pH 6.0, liquor ratio of 1/20, 80 °C for 60 min. The findings showed that the maximum K/S and ΔE reached 3.56 and 35.12 for cotton, and 1.01 and 18.84 for silk, respectively, meaning that the optimum concentration to dye cotton and silk, in such conditions, should be 75%. A higher increase in dye concentration had insignificant effects on the dyeability of textile materials, as over-saturation obstructed the penetration of dye molecules into the fiber structure. The results in Table 3 also reported a rapid decline of K/S with decreasing dye concentration below 75%. This could be due to the formation of hydrogen bonds between anthocyanins and cellulose in the amorphous domain. Such behaviour was not obvious in the fibroin structure of silk, owing to its highly crystalline structure (*i.e.*, denser arrangement).

Color change of dyed cotton and silk by temperature and exhaust time

It is known that protein fibers possess lower heat resistance than cellulose fibers, therefore the dyeing temperature of silk should be lower than that of cotton. Silk can be destroyed or hydrolyzed in aqueous dye solution at high temperature, while cotton could be treated at higher temperature to enhance the dyeing efficiency.[17,18] In this work, the effects of dyeing temperature was investigated in range from 60 to 90 °C for both silk and cotton, at a stable pH of 6.0 and 75% dye concentration.

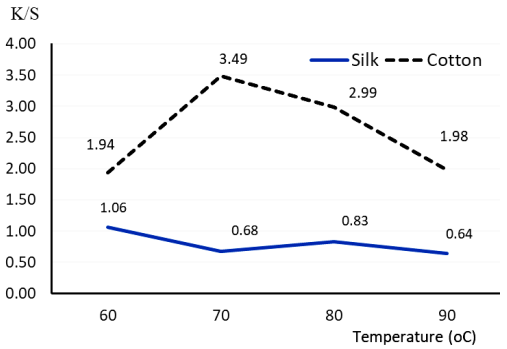

Figure 3: K/S versus temperature (60 to 90 °C) during dyeing cotton and silk with BP extracts (pH 6.0, dye concentration of 75%)

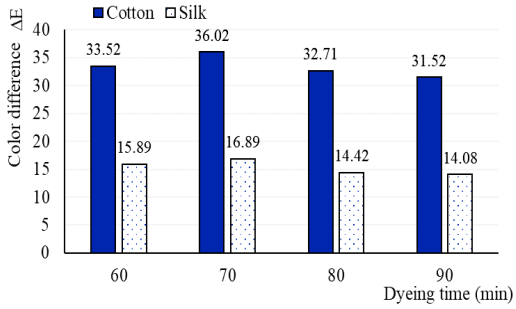

Figure 4: Effect of dyeing time on ΔE values of cotton and silk samples dyed with BP extracts

Table 4
Results of K/S measured on dyed cotton and silk with BP extracts in various exhausting times at pH 6.0, 70°C and 75% of BP

Sample	Time (min)	K/S (average)	σ (standard deviation)
CTNT60	60	2.28	0.074
SLKT60	60	0.78	0.025
CTNT70	70	3.32	0.119
SLKT70	70	0.97	0.030
CTNT80	80	2.30	0.074
SLKT80	80	0.77	0.027
CTNT90	90	2.84	0.108
SLKT90	90	0.81	0.031

Figure 3 shows that, at the same pH value and BP pigment concentration, the optimum dyeing temperatures are 70 °C and 60 °C for cotton and silk, respectively, at which the respective K/S values of the fabrics were 3.49 and 1.06. It should be noted that the K/S values of cotton show more obvious variation as a function of temperature, compared to those of silk. This can be explained

by the fact that, although at high temperature textile fibers have a looser structure, allowing the penetration of anthocyanins, in the case of silk, its more crystalline structure makes difficult the formation of intermolecular interactions with the dyes.

Similarly, Figure 4 and Table 4 present the results obtained for the ΔE and K/S of cotton and silk samples dyed with BP extracts during 60, 70, 80 and 90 min reaction time, while the other parameters were fixed (pH 6.0, 70 °C and 75% BP concentration). It can be affirmed that the increase in dyeing time caused a slight change in K/S for treated cotton and silk, the best values being achieved for a reaction time of 70 min. Furthermore, the ΔE results for the treated samples also reveal a similar trend (Fig. 4).

Role of mordants in dyeing cotton and silk with BP extracts

As known, most natural dyes require the use of mordants in order to enhance the color fastness for treated fabrics. After finding the most suitable (optimum) parameters for dyeing cotton and silk with BP extracts in the previous steps, four different types of mordants, namely potassium alum sulfate ($KAl(SO_4)_2{\cdot}12H_2O$, copper sulfate ($CuSO_4{\cdot}5H_2O$), ferrous sulfate ($FeSO_4{\cdot}7H_2O$) and tannic acid, were added in various amounts to the BP solution, to assess their effects on the dyeability of the fabrics. Thus, 1, 2 and 3% owf (on weight fabric) of mordant were used to evaluate the impact of mordant concentration on the dyeability of cotton and silk with BP extracts. Simultaneously, two cotton and silk samples, without the use of mordants, were used as controls. A proposed complex forming mechanism of anthocyanin to fibroin molecules or cellulose molecules, in the presence of metal mordant (*e.g.*, potassium alum sulfate, copper sulfate and ferrous sulfate), is displayed in Figure 5.

In general, the achieved colors on both dyed silk and cotton do not vary much with the type and the content of the mordants. Anthocyanins were able to form metal complexes with iron, copper and aluminum ions, to generate a typical blue color, but fading occurred.

It can be clearly seen a descending order of mordanting efficiency, in terms of the ΔE value of cotton dyed with BP extracts, as follows: tannins > copper sulfate > potassium alum sulfate > ferrous sulfate. Similarly, the mordanting efficiency decreased for silk dyed with BP extracts in the following order: tannins > potassium alum sulfate > copper sulfate > ferrous sulfate. As shown in Figure 6, the highest ΔE values were 37.35 for dyed cotton mordanted with 2% owf tannins, and 18.73 for dyed silk mordanted with 2% owf tannins. Reversely, the lowest ΔE values were 31.36 for dyed cotton mordanted with 1% owf ferrous sulfate, and 8.21 for dyed silk mordanted with 3% owf ferrous sulfate.

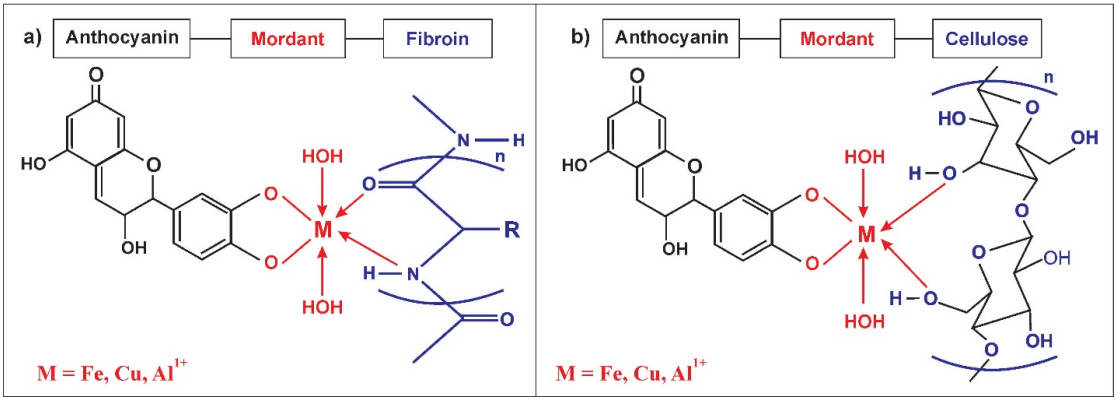

Figure 5: Proposed complex forming mechanism of anthocyanin, metal mordant and (a) silk, or (b) cotton fibers

However, in terms of K/S value, these mordants played a decisive role in retaining a specific content of colorants (anthocyanins) in the textile fibers, especially, in the cellulose structure, as displayed in Figure 7. At the same concentration of mordant (2% owf), the K/S values of dyed cotton were remarkably improved and achieved the highest value (5.57) for CTN-TA2 sample (*i.e.*, cotton mordanted by tannins). On the other hand, the K/S values obtained for

dyed silk fabrics mordanted with the metal salts (PA, FS and TA) do not seem to improve the color depth (lower color effects of the dyes on protein fibers), as compared with the unmordanted samples (*i.e.*, CTN-NO and SLK-NO). Accordingly, when dyeing natural fibers with BP extracts, the use of mordants brings noticeable effects for cellulose fibers, such as cotton or viscose.

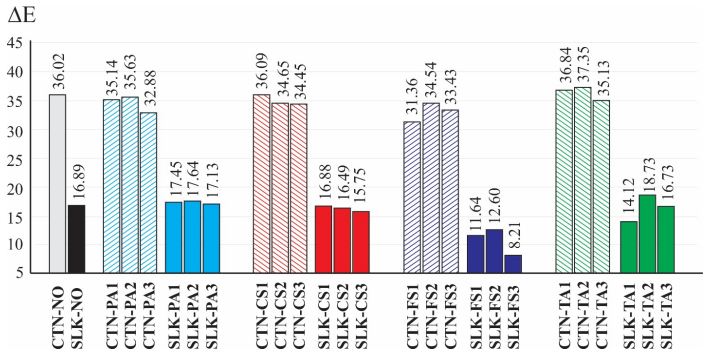

Figure 6: ΔE of cotton and silk dyed with BP extracts, unmordanted (CTN-NO, SLK-NO) and mordanted with 1, 2 and 3% owf of potassium alum sulfate (CTN-PA1, PA2, PA3 and SLK-PA1, PA2 PA3), copper sulfate (CTN-CS1, CS2, CS3 and SLK-CS1, CS2, CS3), ferrous sulfate (CTN-FS1, FS2, FS3 and SLK-FS1, FS2, FS3) and tannin (CTN-TA1, TA2, TA3 and SLK-TA1, TA2, TA3)

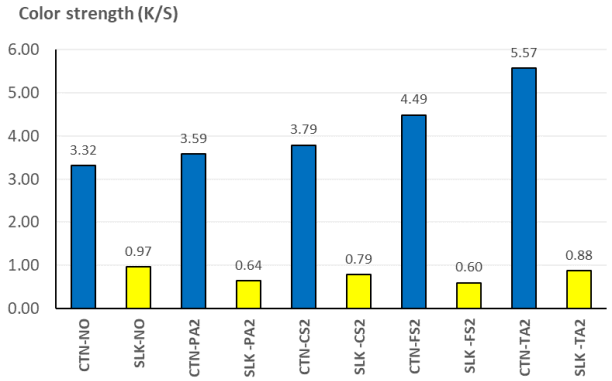

Figure 7: K/S values of dyed cotton and silk without mordants (CTN-NO, SLK-NO) and with 2% owf potassium alum sulfate (CTN-PA2, SLK-PA2), copper sulfate (CTN-CS2, SLK-CS2), ferrous sulfate (CTN-FS2, SLK-FS2) and tannin (CTN-TA2, SLK-TA2)

Table 5
Results of color fastness and color staining to crocking and washing for cotton and silk dyed with BP extracts in the absence of mordant (CTN-NO, SLK-NO) and with 1% owf $CuSO_4.5H_2O$ (CTN-CS1, SLK-CS1) and 2% owf tannin (CTN-TA2, SLK-TA2)

Sample	Mordant	Color fastness			Color staining					
		Washing	Dry rubbing	Wet rubbing	Acetate	Cotton	Nylon	Polyester	Acrylic	Wool
CTN-NO	None	2-3	3	2-3	5	4-5	4-5	5	5	4-5
CTN-CS1	$CuSO_4.5H_2O$, 1%	2-3	5	2-3	5	4-5	4-5	5	5	4-5
CTN-TA2	Tannin, 2%	2-3	4-5	2-3	5	4-5	4-5	5	5	4-5
SLK-NO	None	4-5	5	5	5	5	5	5	5	4-5
SLK-CS1	$CuSO_4.5H_2O$, 1%	4-5	5	5	5	5	5	5	5	4-5
SLK-TA2	Tannin, 2%	4-5	5	5	5	5	5	5	5	4-5

Color fastness to crocking and washing of dyed cotton and silk

To evaluate the color fastness to crocking/rubbing and washing of the treated fabrics, the cotton and silk samples were dyed with BP extracts in the absence of mordant (CTN-NO and SLK-NO) and in the presence of mordant (1% owf of $CuSO_4.5H_2O$ and 2% owf of tannin). The thus-mordanted cotton and silk samples were denoted as CTN-CS1, CTN-TA2 and SLK-CS1, SLK-TA2, respectively, as listed in Table 5. All the measurements of color fastness to crocking and washing were carried out according to the ISO 105 test method (using a grey scale).

Clearly, in the absence of mordant, the color fastness to washing for the cotton sample exhibited lower values, compared to those of the silk sample. Specifically, the color fastness grades obtained for CTN-NO and SLK-NO are 2-3 and 4-5, respectively. Similarly, the grades of color fastness to dry and wet rubbing obtained for silk are higher than those for cotton, being 3 and 2-3 for cotton, 5 and 5 for silk, respectively. However, the grades of color staining for both cotton and silk are very close, being 4-5 to 5. As compared with the unmordanted samples, the grades of color fastness to washing of the samples dyed in the presence of mordants remain the same, being 2-3 for cotton and 4-5 for silk (Table 5).

CONCLUSION

This work investigated the optimum dyeing conditions for cotton and silk fabrics with BP extracts, in terms of color strength and color difference. The effects of various pH values, liquor ratios, dye concentrations, temperature, dyeing time, types of mordant and content of mordant were observed. The optimum parameters for dyeing cotton and silk with BP extracts were found the following: 70 °C for cotton and 60 °C for silk, at pH of 6.0, liquor ratio of 1/5 and dyeing time of 60 min. The evaluations on color fastness, including color change and color staining, under washing and rubbing conditions, were performed, indicating that the color fastness of dyed cotton was lower than that of silk (2-3 and 4-5, respectively). To conclude, the K/S and ΔE values of cotton were higher than those of silk samples, however, their results of color fastness and color staining to washing and rubbing were reversed (*i.e*., silk had higher color fastness).

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