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INVESTIGATION ON COLOR DIFFERENCE OF DYED COTTON FABRICS WITH EXTRACTS FROM *Bixa orellana* SEEDS THROUGH CIELAB COLOR SPACE

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ABSTRACT

Natural dyes extracted from plants are eco-friendly and biodegradable pigments, using for coloring not only food industry but also textile materials but a lot of their mysteries have not been discovered. This work is to investigate the effects of bleaching agents, dye concentration, exhausting time, mordanting sequence, mordant types and mordant contents on the dyeability of cotton fabrics with *Bixa orellana* (BO) extracts through color difference (ΔE) obtained from the CIELAB color system. Based on such investigations, the bonding mechanisms between bixin/norbixin in BO extracts and the cellulose of cotton fibers were clarified. The results demonstrated that bleaching agent prevented the absorbance of dyes into fabrics and the dyeability improved better as increasing dye concentration and exhausting time. The mordant agents enhanced the dyeability of cotton samples with BO extracts, which obtained the highest ΔE values when dyeing and mordanting samples simultaneously. In fact, potassium aluminum sulfate ($KAl(SO_4)_2.12H_2O$) played a role as an efficient mordant which presented the higher ΔE value as compared to copper sulfate ($CuSO_4.5H_2O$). The color fastness of dyed samples achieved the grade 3 after a washing cycle and their color staining tests to rubbing were better as mordanting before dyeing process.

Keywords: Bixa orellana (BO) extract, bixin/norbixin, color difference, mordant, metal complex.

1. INTRODUCTION

Natural colorants possess many advantages owing to their great demands, health safety and environmentally friendly properties. Basically, natural dyes are sustainable because of their renewability and biodegradability [1]. Annatto or *Bixa orellana* (BO) is a tropical shrub popularly grown in many countries around the world. It is cultivated for orange-red pigments extracted from seeds which are widely used in foods, drugs and cosmetics [2]. BO is well-known as a condiment, generating not only colorant but also flavors and aroma for foods and beverage. Some highlighted biological properties of BO extracts are no stability to oxidants, no toxicity, and flexibility [3].

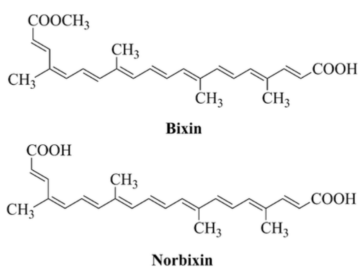


Figure 1. Chemical structure of bixin and norbixin in BO seeds

In recent years, BO seeds have been used in medicine to cure wounds, skin eruptions, burns, obesity and diabetes [4]. Besides bixin and norbixin content, the antioxidant and antibacterial activities of BO extracts and leaves were affirmed in some publications through the findings of phenolic compounds and flavonoid compounds [5-7]. The colorant is obtained from the outer layer of BO seeds which chemical compositions are a mixture of bixin (i.e., a monomethyl ester of dicarboxylic carotenoid and norbixin) [8]. The colorant extracted from BO seeds was also used to dye some natural textile fibers such as wool, silk and cotton [9-12]. Natural dyes from BO extracts were supposed to be a potential alternative to synthetic dyes for use in the textile industry [13]. It is evidenced that main non-toxic compositions including bixin and norbixin generated the characteristic color (orange-red) of BO extracts [14, 15]. Rare earth salts such as cerium nitrate, lanthanum chloride and yttrium chloride have also been used in dyeing textiles with BO extract with the significantly improved dye uptake, better fastness properties and less effluent pollution [16]. Some conventional mordants including copper sulfate and iron sulfate were applied to optimize the dyeability and washing fastness of treated silk with BO extracts [17]. Cotton and wool fabrics have been dyed with BO ultra-filtrated powders in the absence of mordants with optimum dyeing conditions [18, 19]. Accordingly, the previous authors proved that color strength (K/S) of wool and silk as dyed with BO extract presented better than that of cotton because of their high affinity [15]. Obviously, the dyeing ability of cotton fabric with BO extracts should be considered more to improve color fastness. The evaluation on dyeability of cotton with BO extracts in terms of color change should be carried out in various conditions.

In this work, BO extract was used to dye cotton fabrics including concentration, exhausting time, mordant types, mordanting sequence, washing and rubbing. Accordingly, the dyeability of cotton with BO pigment was evaluated and clarify based on color difference among different samples obtained from the specific values of CIELAB color system.

2. MATERIALS AND METHODS

2.1. Materials

Grey cotton woven fabrics (253 g/m^2) with 24 threads per cm and 32 threads per cm in lengthwise and crosswise, respectively. *Bixa orellana* (BO) seeds were purchased from Vinada shop. Mordants including aluminum potassium sulfate dodecahydrate ($KAl(SO_4)_2.12H_2O$) and copper sulfate ($CuSO_4.5H_2O$) which were purchased from AR (India) and Xylong (China), respectively, were used to enhance dyeability of cotton fabrics. Hydrogen peroxide (H_2O_2) and sodium hydroxide (NaOH, Tide) were used for bleaching grey fabrics and washing dyed fabrics.

2.2. Experimental methods

BO seeds were ground into small particles and extracted in distilled water. The cotton samples (15 cm x 15 cm) were prepared in the room condition within 24 hours before carried out all experiments. The effects of dye concentration, exhausting time, types and concentration of mordants on dyeability through color difference (ΔE) were investigated. Experimental instruments include an exhaust dyeing machine (Copower) with 24 cups, a magnetic stirrer (Velp Scientifica), an electronic scale (Ahaus, ±0.0001g). A portable colorimeter which color measurements were determined based on CIE Lab color space. Color difference (ΔE) of dyed samples was defined as a distance between two color coordinates (L_1, a_1, b_1) and (L_2, a_2, b_2) as follows:

$$\Delta E = \sqrt{(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2} \quad (1)$$

The color fastness and color staining to washing and rubbing were measured by a washer (ISO 105-C01) and a crockmeter (according to the AATCC 8). All results were examined by a grey scale with 10 evaluating grades including 1.0, 1.5, 2.0, 2.5, 3.0, 3.5, 4.0, 4.5 and 5.0.

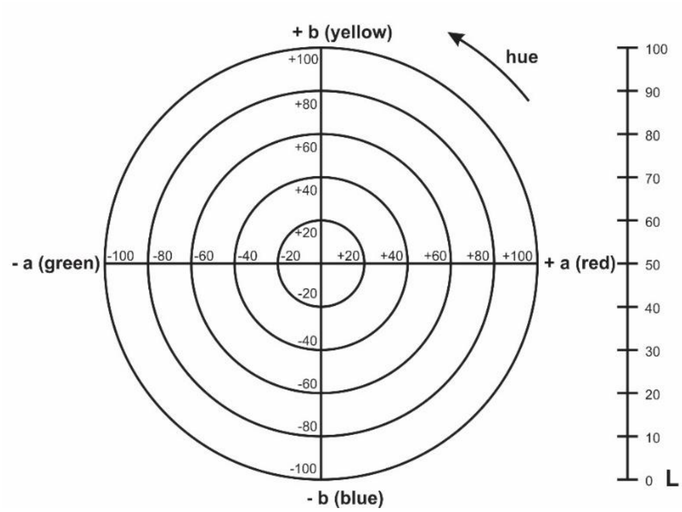


Figure 2. CIELAB color space

3. RESULTS AND DISCUSSION

3.1. Bleaching agents

Usually, grey cotton fabrics contain a significant amount of sizing agents formed in the spinning and weaving stages. Such compounds could obstruct the penetration of dyes into fabric structures. Furthermore, sizing agents are able to absorb a large amount of dyeing solution that can be removed from fibers, causing waste of dyes as well as negative impacts on the environment. Accordingly, before dyeing cotton fabrics, they are often scoured with bleaching agents to ensure that all sizing agents and impurities are eliminated. A remarkable color difference between the unbleached cotton sample and the bleached cotton sample were found when dyeing with BO extracts. It can be recognized with naked eyes that cotton fabric without bleaching pretreatment provided a darker color than that with bleaching pretreatment.

Table 1. ΔE of dyed cotton fabrics with and without bleaching pretreatments

Sample		L	a	b	C	ΔE*
Unbleached		42.75	44.8	43.63	62.53	5.64
Bleached		44.78	44.92	48.89	66.39	

As indicated in Table 1, the difference between bleached and unbleached samples dyed with BO extracts could be quantitively evaluated based on the CIE Lab color space, which the color distance (i.e., ΔE) was defined as 5.64. The results demonstrated that the cotton fabrics should be bleached carefully before dyeing with BO extracts to obtain their higher dyeability. Clearly, the dyes were distributed more uniformly throughout the fabric surface owing to the removal impurities. The removal of sizing and pretreating agents from the grey cotton fabrics is very important to ensure its dyeing quality.

3.2. Dye concentration and exhausting time

To evaluate the ΔE values among dyed samples at various dye concentrations in a specific condition (at 70ºC in 60 min), the cotton sample dyed with 2 g/20 mL of BO (SPC2) was used as a standard sample. The effects of dye concentration and exhausting time on the dyeability of dyed sample with BO extracts were examined as presented in Table 2 and Figure 2.

Table 2. The effect of dye concentration on color of dyed samples

Sample		Dye concentration	L	a	b	C	ΔE*
SPC2		2 g/20 mL (1:1)	55.42	33.32	53.96	63.42	-
SPC4		4 g/20 mL (2:1)	47.61	41.73	49.96	65.09	12.15
SPC6		6 g/20 mL (3:1)	44.78	44.92	48.89	66.39	16.54
SPC8		8 g/20 mL (4:1)	40.53	44.73	39.02	59.36	23.98

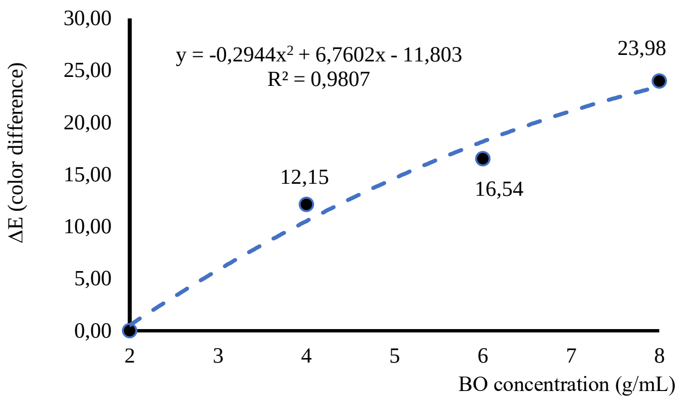


Figure 2. The curve of ΔE versus dye concentration on dyed cotton sample with BO extracts

The achieved results exhibited that the ΔE value rapidly increased with the dye concentration, being 12.15, 16.54 and 23.98 for SPC4 (4 g/20 mL), SPC6 (6 g/20 mL) and SPC8 (8 g/20 mL), respectively. It means that ΔE values among the dyed samples at various BO concentrations were distinguished, which the color of dyed fabrics was darker with higher BO content. The curve of ΔE against the BO concentration was described as a quadratic function with R-squared of 0.9807, meaning that the ΔE value of dyed sample with BO extracts approaches an extreme point (i.e., maximum BO concentration is 11.4812 g/20 mL) because excessively high dye content is difficult to diffuse deeply into textile fiber. Through the evaluation on the color difference between dyed samples with different dye concentrations, their dyeability was clarified. Similarly, the effect of exhausting time on color difference of dyed samples in 40 min (SP40MIN), 60 min (SP60MIN), 80 min (SP80MIN) and 100 min (SP100MIN) was investigated and presented in Table 3.

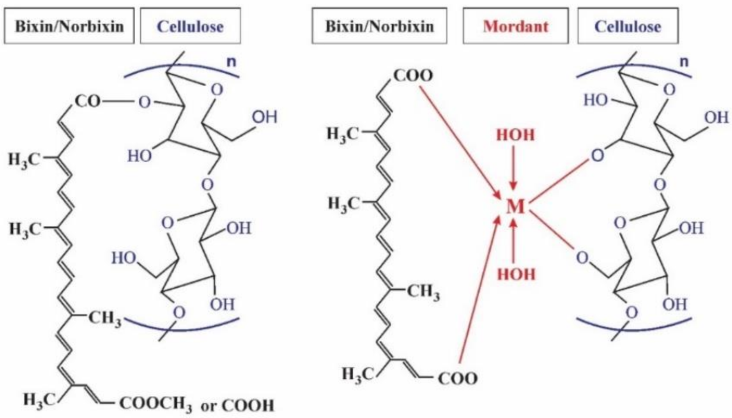
Table 3. The ΔE values of dyed sample with BO extracts in different dyeing times

Sample		Time (min)	L	a	b	C	ΔE*
SP40MIN		40	44.6	42.87	48.91	65.04	0
SP60MIN		60	44.78	44.92	48.89	66.39	1.56
SP80MIN		80	44.83	43.47	45.38	62.84	5.08
SP100MIN		100	45.2	39.79	48.08	62.41	6.66

The investigated results proved that a slight increase in the ΔE value was detected from 1.56 to 6.66 for cotton samples dyed in 60 to 100 min, respectively. Obviously, in terms of CIELAB color space, the color difference among colors of cotton samples dyed with various dyeing times was significant in range from 40 to 100 min. Accordingly, the dyeing process enhances the absorbance of dyestuffs into the textile fibers in which the intermolecular interaction between BO pigments extracts and cellulose molecules happens gradually in the amorphous region.

3.3. Mordanting agents

To enhance the color fastness of dyed fabrics with BO extracts, two different types of mordanting agents including copper sulfate ($CuSO_4.5H_2O$) and potassium aluminum sulfate ($KAl(SO_4)_2.12H_2O$) were added to the BO solution. A complex formation mechanism of bixin/norbixin - mordant - cellulose were proposed and illustrated in Figure 3.



a) Unmordanted b) Mordanted

Figure 3. Proposed bonding mechanism of bixin/norbixin to cellulose with (a) the absence and (b) the presence of metal salts.

As mentioned in the previous section, the natural main composition generating the characteristic colorant of BO seeds is originally bixin (i.e., red-orange hues, an oil soluble pigment) and norbixin (from yellow hue to orange hue, a water-soluble pigment). It can be seen that both bixin and norbixin are capable of binding to cellulose because of ester bonds between carboxyl groups (in bixin/norbixin compounds) and hydroxyl groups (in cellulose molecules).

Such bonding mechanism between bixin and cellulose has proved in the other work by the UV, IR and H-NMR spectra [20]. Table 4 shows the color difference among samples mordanted with 10% on weight fabric (owf) of $CuSO_4.5H_2O$ before (SPMB), while (SPMW) and after (SPMA) dyeing process as comparing to the untreated sample with mordanting agents (SPNO).

Table 4. Color difference among dyed samples with three types of mordanting sequence as before, while and after dyeing.

Sample		Mordanting sequence	L	a	b	C	ΔE*
SPNO		None	44.78	44.92	48.89	66.39	-
SPMB		Before	47.01	46.34	52.38	69.94	4.378
SPMW		While	38.71	43.59	41.43	60.14	9.709
SPLA		After	42.19	48.84	43.32	65.28	7.287

Clearly, the mordanting sequence (10% owf of $CuSO_4.5H_2O$) causes change in the dyeability of cotton fabrics with BO extracts. The highest ΔE value belongs to the cotton samples which were dyed and mordanted simultaneously (i.e., being 9.709). It means that the mordanting agent should be inserted while dyeing cotton fabrics with BO extracts through the exhaust method to obtain the best dyeability. Based on the findings of mordanting sequence, the effects of mordant types including $CuSO_4.5H_2O$ (SPCU) and $KAl(SO_4)_2.12H_2O$ (SPKA) were examined and reported in Table 5.

Table 5. The color variation of dyed samples mordanted with $CuSO_4.5H_2O$ (SPCU) and $KAl(SO_4)_2.12H_2$ (SPKA) as compared to the standard sample

Sample		Mordant	L	a	b	C	ΔE*
SPNO		None	44.78	44.92	48.89	66.39	-
SPCU		$CuSO_4.5H_2O$	49.37	41.26	49.96	64.79	5.97
SPKA		$KAl(SO_4)_2.12H_2O$	40.68	49.39	44.53	66.45	7.47

Accordingly, the ΔE value of SPKA is higher than that of SPCU, being 5.97 and 7.47, respectively. It can be confirmed that the dyeability of cotton fabrics dyed with BO extract was improved significantly owing to the presence of metal mordants such as copper sulfate and potassium aluminum sulfate. Certainly, the dyeability of cotton fabrics with BO extracts also depends on the added mordant contents. Whereby, 5, 10 (and 15% owf of $CuSO_4.5H_2O$ (also called as SPM05, SPM10 and SPM15, respectively) were inserted to the BO solutions at the same dyeing time (i.e., dyeing and mordanting processes are conducted simultaneously) and the obtained ΔE values were reported in Table 6.

Table 6. Color difference of dyed cotton samples at 5, 10 and 15% owf of $CuSO_4.5H_2O$

Sample		Content (% owf)	L	a	b	C	ΔE*
SPM05		5	44.06	48.9	44.46	66.09	-
SPM10		10	42.19	48.84	43.32	65.28	2.19
SPM15		15	39.94	47.45	39.62	61.82	6.52

Consequently, as comparing to the SPM05, the ΔE value increased with the mordant content, being 2.19 and 6.52 for SPM10 and SPM15, respectively. This is because the more attendance of mordant in forming complexes of cellulose - mordant and bixin/norbixin molecules as mentioned above. On the other hand, the dyeability of dyed cotton with BO extracts was improved significant as increasing the concentration of mordant.

3.4. Color fastness to washing and rubbing

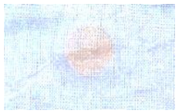
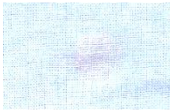
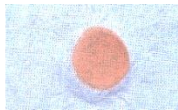
In order to examine the color fastness of dyed cotton fabrics with BO extracts to washing and rubbing, three different samples including SP00 (not mordanted), SPMB (mordanted with $KAl(SO_4)_2.12H_2O$ before) and SPMA (mordanted with $KAl(SO_4)_2.12H_2O$ after) were tested. The color fastness on dyed samples after a washing cycle according to the ISO 105-C01 standard method were indicated in Table 7.

Table 7. The color change of dyed samples with BO extract in the absence (SP00) and the presence of mordanting agents (SPMB and SPMA) after a washing cycle

Sample	Mordants	Color change	Unwashed and washed sample
SP00	None	3-4	
SPMB	$KAl(SO_4)_2.12H_2O$ before dyeing	3	
SPMA	$KAl(SO_4)_2.12H_2O$ after dyeing	3	

Based on the grey scale, grades of color change obtained from the pair of dyed samples (before and after washing) are 3-4, 3 and 3 for SP00, SPMB and SPMA, respectively, meaning that washing conditions cause a significant impact on the color of the dyed samples. The color change was less as mordanted with different sequences. Likewise, the testing results of color staining in terms of dry and wet rubbing for dyed samples in the presence of $KAl(SO_4)_2.12H_2O$ before and after dyeing process were evaluated and showed in Table 8.

Table 8. Color staining to dry and wet rubbing on dyed samples with BO extracts in the absence and the presence of mordants

Sample	Mordanting sequence	Color staining			
		Dry rubbing		*Wet rubbing*	
SPN00	None		3		1-2
SPMB	Mordanted with $KAl(SO_4)_2.12H_2O$ before dyeing		4		2-3
SPMA	Mordanted with $KAl(SO_4)_2.12H_2O$ after dyeing		3-4		1-2

The results demonstrated that the grade of color fastness of dyed samples with BO extracts to wet rubbing was less than that to dry rubbing. In general, color fastness of dyed samples with BO extracts to wet rubbing is quite low, which the grade is from 1 to 3. However, the sample mordanted before dyeing produced more color durability as compared to the samples mordanted after dyeing. It can be concluded that cotton fabrics should be dyed with BO extract and mordanted with aluminum potassium sulfate dodecahydrate before dyeing processes. It can be explained that bixin and norbixin have already linked directly to cellulose (i.e., SPMB). Therefore, adding $KAl(SO_4)_2.12H_2O$ to dyed fabric (SPMA) is difficult to form metal complex between dyes and celluloses.

4. CONCLUSIONS

In terms of the color difference in the CIELAB system, this work clarified the dyeability of cotton fabrics with BO extracts which the effect of bleaching agents, dye concentration, exhausting time, mordanting sequence, mordant types and mordant content were discussed. The parameters obtained from these investigations provided for more details to optimize the dyeability of cotton fabrics with BO extracts as comparing to the previous related works. Particularly, the ΔE value between bleached and unbleached cotton samples was 5.64. Besides, the dyed samples become darker with higher BO content and exhausting time. A bonding mechanism between bixin/norbixin and cellulose was proposed and revealed. The highest dyeability of cotton fabric with BO extracts was obtained as mordanted with $KAl(SO_4)_2.12H_2O$) and the dyeing efficiency of cotton fabrics was better when the metal mordants were inserted while dyeing process. Also, the color fastness to washing and rubbing was better as pretreated with mordants.

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TÓM TẮT

KHẢO SÁT ĐỘ LỆCH MÀU CỦA VẢI BÔNG NHUỘM VỚI DỊCH CHIẾT HẠT ĐIỀU NHUỘM QUA KHÔNG GIAN MÀU CIELAB

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Thuốc nhuộm tự nhiên chiết xuất từ thực vật là những chất màu thân thiện và phân hủy sinh học, sử dụng trong lĩnh vực nhuộm không chỉ thực phẩm mà còn vật liệu dệt nhưng còn rất nhiều bí ẩn của chúng vẫn chưa được khám phá. Nghiên cứu này khảo sát ảnh hưởng của chất tẩy, nồng độ nhuộm, thời gian tận trích, thứ tự cầm màu, loại và lượng chất cầm màu đến khả năng nhuộm vải bông với dịch chiết từ hạt điều nhuộm dựa trên độ chênh lệch màu (ΔE) thông qua hệ không gian màu CIELAB. Dựa trên các kết quả thực nghiệm, cơ chế liên kết giữa bixin/norbixin trong dịch chiết và cellulose của xơ bông được làm rõ. Các kết quả đã chứng minh rằng chất tẩy ngăn cản khả năng hấp thu thuốc nhuộm vào sâu trong cấu trúc vải, khả năng nhuộm của vải được nâng cao khi tăng nồng độ nhuộm và thời gian tiếp xúc. Các chất cầm màu giúp tăng cường khả năng nhuộm của vải bông với dịch chiết với giá trị đạt cao nhất khi nhuộm và cầm màu diễn ra đồng thời. Thực tế, muối nhôm ($KAl(SO_4)_2.12H_2O$) đóng vai trò như một chất cầm màu hiệu quả, cho giá trị lệch màu cao hơn so với muối đồng ($CuSO_4.5H_2O$). Độ bền màu của các mẫu nhuộm đạt cấp độ 3 sau một chu kỳ giặt và độ bền màu với ma sát đạt kết quả tốt hơn khi thực hiện cầm màu trước khi nhuộm.

Từ khóa: Dịch chiết hạt điều nhuộm, bixin, norbixin, độ lệch màu, chất cầm màu, phức kim loại.