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## DYEING OF COTTON FABRICS WITH SODIUM COPPER CHLOROPHYLL PAMUKLU KUMAŞLARIN SODYUM BAKIR KLOROFİL İLE BOYANMASI

Deniz Mutlu ALA<sup>1</sup> (ORCID: 0000-0002-5864-308X)  
Gamze GÜLŞEN BAKICI<sup>1\*</sup> (ORCID: 0000-0002-4241-7096)

<sup>1</sup> Çukurova Üniversitesi, AOSB Teknik Bilimler Meslek Yüksekokulu Tekstil, Giyim Ayakkabı ve Deri Bölümü, Adana, Türkiye

\*Sorumlu Yazar / Corresponding Author: Gamze GÜLŞEN BAKICI, gamzegulsenbakici@gmail.com

### ABSTRACT

The aim of this study is to determine the dyeing temperature at which cotton fabrics will absorb the most sodium copper chlorophyll (SCC) dyestuff. Bleached 100% cotton rib knitted fabrics were dyed with SCC at different times without mordanting. The dye absorption of the fabrics was determined by a spectrophotometric method depending on the dyeing time. Firstly, SCC solutions with known concentrations were measured and the calibration (absorbance-concentration) curve was obtained. Then, the SCC concentrations remaining in the dye liquors after dyeing at different times were determined. The dye exhaustion of the fabrics was calculated using the concentrations of the dye baths before and after dyeing. Color measurements and fastness tests of the dyed samples were performed.

**Keywords:** Sodium copper chlorophyllin, natural dyeing, color, UV-Vis

### ÖZET

Bu çalışmanın amacı, pamuklu kumaşların sodyum bakır klorofil (SCC) boyarmaddesini en fazla çekeceği boyama sıcaklığını tespit etmektir. Ağartılmış %100 pamuklu ribana örme kumaşlar, SCC ile mordanlama yapılmadan farklı sürelerde boyanmıştır. Kumaşların boya çekimi boyama süresine bağlı olarak spektrofotometrik yöntem ile belirlenmiştir. İlk olarak konsantrasyonları bilinen SCC çözeltileri ölçülünerek kalibrasyon (absorbans-konsantrasyon) eğrisi elde edilmiştir. Ardından farklı sürelerde yapılan boyama işlemlerinden sonra boya flotteleri içerisinde kalan SCC konsantrasyonları tespit edilmiştir. Kumaşların boya çekimi, boya banyolarının boyama öncesi ve boyama sonrası konsantrasyonları kullanılarak hesaplanmıştır. Boyanmış numunelerin renk ölçümleri ve haslık testleri yapılmıştır.

**Anahtar Kelimeler:** Sodyum bakır klorofil, doğal boyama, renk, UV-Vis

## INTRODUCTION

Natural dyes have been widely used in the coloring of natural fibers including wool, silk, and cotton since ancient times. Although at present synthetic dyes are dominating over natural dyes as they have advantages such as the complete range of colors and excellent color fastness, they have important disadvantages as they have adverse effects on human health and the environment (Mirjalili et al., 2022; Hossain et al. 2021; Jia et al., 2017). Whereas natural dyes are nontoxic, non-carcinogenic, biodegradable, and more eco-friendly compared with their synthetic counterparts (Baig et al., 2021; Tayade & Adivarekar, 2013). Concerning the growing awareness of environmental and health related problems, natural dyes have been receiving more attention for textile applications on account of their renewability, biodegradability, low toxicity, and environmentally friendly nature (Wang et al., 2017; Oh & Na, 2014; Islam et al., 2013). Many natural dye sources including plant, animal, and mineral extracts have been successfully applied for textile dyeing and printing (Ala & Gülşen Bakıcı, 2020; Rekaby et al., 2009; Karadağ, 2007).

Natural green dyes consisting of chlorophylls are used in many approaches as fabric dyeing colorants. Chlorophyll is the pigment that gives plants their characteristic green color. As a natural derivative of natural chlorophyll, sodium copper chlorophyll (SCC) is a natural edible pigment and can also be used as a green natural colorant for natural fibres (Liu et al., 2020; Hou et al., 2012; Ferruzi et al., 2002).

Recently, researchers have focused on investigating the natural dyeing of textile materials and their dyeability. Research has been conducted in the literature on “dyeing silk using Dwarf Elder fruit dye and Alkannin dye from *Alkanna tinctoria*” (Dayioglu et al., 2015; Adeel et al., 2022), “dyeing wool using *Chamaecyparis Lawsoniana* cone extract and madder and *resedaluteola*” (Kilinc et al., 2015; Hosseinezhad et al., 2022), “dyeing of banana fibers with natural dye extracted from the turmeric plant”(Canbolat et al., 2015), “dyeing of cotton with *Helichrysum Arenarium* extract and a natural dye extracted from *Syzygium cumini* fruits” (Akkaya & Eyupoglu, 2016; Periyasamy, 2022), “dyeing of mohair fibers with natural dye extracted from *Candelariella reflexa*” (Eyupoglu et al., 2022), “dyeing of organic cotton fabrics with gardenia yellow from *Gardeniae fructus* seeds” (Jiang et al., 2022), “dyeing hemp with extract dye from stinging nettle” (Gürbüz, 2022). In this study, natural dyeing was carried out using sodium copper chlorophyll. Summarizing the research conducted using SCC, it can be expressed as follows. Hou et al., (2012) investigated the kinetics and thermodynamics of SCC adsorption on silk in dye bath and they reported that SCC can be used for dyeing protein-based fibers. Tsatsaroni and Liakopoulou-Kyriakides, (1995) studied the fastness properties of natural fabrics dyed with the natural dyes chlorophyll and carmine after treatment with the enzymes. Yoo et al., (2013) investigated the possibility of dyeing wool and silk fabrics with chlorophyll without chemical alteration by using methanol, acetone, and water to extract spinach and mate powders. Usop et al., (2016) investigated the color stability of natural green dye coating films consisting of chlorophyll from *Cassia alata* leaves, which were exposed to ultraviolet A (UV-A). Zhao et al., (2020) used ethylene-diaminetetraacetic acid disodium salt and sodium citrate to treat the cotton fabrics dyed with SCC and gardenia yellow.

SCC, derived from chlorophyll, is a green natural dye extensively employed for coloring food and beverages (Zhong et al., 2020). Research on the use of SCC in the dyeing of textile materials is limited. The main objective of this study was to determine the dyeing temperature of SCC for dyeing cotton materials and to investigate its color and retention properties.

## EXPERIMENTAL STUDY

### *Material*

100% cotton 1 x 1 rib knitted fabric was produced on a 10 gauge flat knitting machine using Ne 20/1 cotton carded ring yarn. Commercial sodium copper chlorophyll dye ( $C_34H_{31}CuN_4Na_3O_6$ , SCC) liquid was provided from Tito Co. Ltd., Turkey.

### *Method*

#### *Pre-treatment*

The bleaching processes for cotton fabric were conducted within the laboratory type machine operating at 95°C. Employing a specific recipe represented in Table 1, the bleaching process involved the application of hydrogen peroxide alongside auxiliary chemicals. The bleaching process of the fabrics was done according to the exhaust method.

**Table 1.** Pretreatment Recipe and Diagram

Liquor ratio	1:30
Oil remover soap (g/L)	0.5
Anti-crease (g/L)	0.5
İon trapping (g/L)	0.8
Wetting agent (g/L)	0.5
Peroxide Stabilizer (g/L)	0.8
Sodium Hydroxide (46°Be) (g/L)	4
Hydrogen peroxide (%35) (%)	6
pH	10

After the bleaching procedures, the samples underwent rinsing and neutralization in a neutralizing solution containing 1 cm<sup>3</sup>/L of acetic acid, maintained at 50°C for a duration of 20 minutes. Subsequently, an anti-peroxide treatment was conducted in a solution composed of 1 g/L antiperoxide and 0.5 cm<sup>3</sup>/L acetic acid, also held at 50°C for 20 minutes. The process concluded with a final rinse before drying.

### **Dyeing**

The dyeing process was performed at a temperature of 60°C, maintaining a liquor ratio of 10:1. Upon reaching the prescribed dyeing temperature, cotton fabrics were immersed for varying durations ranging from 10 to 90 minutes, while the SSC remained constant at 7 ml/L throughout the dyeing processes. After dyeing, the samples underwent two consecutive rinse cycles before being air-dried under ambient conditions.

### **UV-Vis Spectroscopy**

A UV-Visible light spectrophotometer (UV-1800 Shimadzu) was employed to examine the dye exhaustion of the cotton fabric.

### **Color Measurement & Color Strength & Color Fastness**

The CIELab color values (L\*, a\*, b\*, ΔE) of the dyed samples were determined using a Datacolor 850 spectrophotometer from Eksoy Chemical Industry, Turkey, with illuminant D65 and a 10° standard observer. Colorfastness to rubbing and washing of fabric were determined according to TS EN ISO 105-X12 and TS EN ISO 105-C06, respectively. Additionally, the color strength of cotton fabric samples was assessed by obtaining K/S values at the maximum wavelength using the spectrophotometer (Equation 1).

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

Equation 1

where, R=reflectance, K=absorption coefficient, and S=scattering coefficient of dye (Periyasami, 2022).

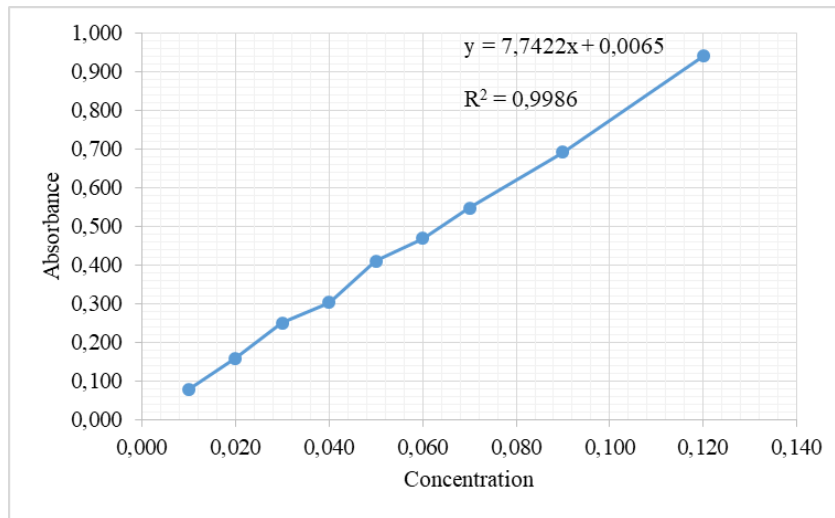
## **RESULTS AND DISCUSSION**

### **UV-Vis Spectroscopy**

#### **Calibration Curve**

To determine the concentration of dyestuff remaining in the dye bath after dyeing, the maximum absorbance wavelength of the sodium copper chlorophyll dyestuff was first determined with the UV-1800 Shimadzu Spectrophotometer device in the laboratory of Eksoy Chemical Industry, which can measure in the range of 190-1100 nm. For this purpose, a 0.1% dilute solution of copper chlorophyll dye was prepared. The absorbance wavelength measurement of the dyestuff was made from this dilute solution. The measured wavelength range was 400-800 nm, the photometric method was chosen, and  $\lambda_{max} = 626.5$  nm (Figure 2). To create the calibration curve, the wavelength was set as 626.5 nm and dye solutions (1, 2, 3, 4, 5, 6, 7, 9, 12) at 9 different concentrations were used.

The solutions containing specified dye concentrations were individually subjected to analysis within the UV-VIS spectrophotometer, whereupon the respective absorbance values were determined. Subsequently, a graphical representation delineating absorbance against concentrations, termed herein as the absorbance graph or calibration curve, was constructed (Figure 1).



**Figure 1.** Absorbance Graph (calibration curve) for SCC

### Examination of Dye Exhaustion Depending on Time

To determine suitable dyeing time for the natural dyeing process of cotton with SCC, dyeing processes were performed at 60°C with different dyeing times (10 to 90 minutes) by using the same concentration of SCC (7 ml/L) in the dyeing bath. The liquor ratio for dyeing was chosen as 10:1. Dye exhaustion was determined by the spectrophotometric method. Dye exhaustion was calculated according to the Equation (2).

$$\text{Dye Exhaustion (\%)} = (C_b - C_a)/C_b * 100 \tag{Equation 2}$$

where  $C_b$  is the concentration of the dye bath before dyeing and  $C_a$  is the concentration of the dye bath after dyeing (Yolaçan, 2009). The unexhausted amounts of SCC were determined by the difference in the dye concentration in the dye bath before and after dyeing.

As seen in Table 2, the dyeing time had a significant effect on the dye exhaustion. It is seen that approximately 15% of the dye in the dye bath is exhausted during the first 10 minutes. It is seen that the dye exhaustion value was gradually increasing up to 60 minutes of dyeing time. The opportunity for migration of the SCC molecules from the dyeing bath to the surface of the cotton fabric increases till it reaches the optimum dyeing time. The maximum value of dye exhaustion (%25.71) was obtained at 60 minutes of dyeing time. Further increase in the dyeing time is accompanied by either staying constant or even a decrease in dye exhaustion. Hence, it may be concluded that the optimum dyeing time is 60 minutes for reaching the dyeing saturation of cotton fabrics when it was dyed with SCC under the conditions in this study.

**Table 2.** Dye Exhaustion

Sample Code	Dye Exhaustion (%)	Unexhausted Dye (%)
P10	15.71	84.29
P20	17.14	82.86
P30	20.00	80.00
P40	21.42	78.58
P50	21.42	78.58
P60	25.71	74.29
P70	25.71	74.29
P80	22.85	73.15
P90	25.71	71.43

### Color Measurement & Color Strength

Color measurements were made to assess the color of dyed samples. The color values given as CIELab coordinates are shown in Table 3 when the sample P60 was selected as the reference. As seen in Table 3, dyeing time influences the color of dyed fabrics. L\* values decrease with increasing dyeing time, and the dyed samples are darker with longer dyeing time with regard the L\* coordinate. Negative a\* value means the greenness of a shade, and positive b\* is the yellowness of the shade. With the longer dyeing time, the changes in the values of a\* and b\* were not fluent. The results also revealed that the K/S value is affected by the dyeing time and increases with the longer dyeing time. The K/S value was improved from 0.76 to 1.16 over time from 10 minutes up to 90 minutes.

**Table 3.** Color Measurement Results

Sample Code	L*	a*	b*	$\Delta E$	K/S
P60	78.12	-8.80	9.09		1.08
P10	79.03	-6.58	8.94	1.99	0.76
P20	79.11	-6.75	9.36	1.95	0.81
P30	78.85	-8.38	8.96	0.45	1.02
P40	78.94	-8.82	8.75	0.43	0.97
P50	78.38	-9.05	8.83	0.37	1.11
P70	78.25	-8.36	9.16	0.41	1.12
P80	78.22	-8.39	9.20	0.40	1.12
P90	77.33	-9.21	9.26	0.46	1.16

### Color Fastness

Color fastnesses to rubbing and washing were determined (Table 4). The wet and dry rubbing fastness values of the samples dyed for 40-50-60-70-80 and 90 minutes were observed at a very good level (4 and 5). In the color fastness test to washing, level 4 staining was observed on the cotton fabric. It can be concluded that SCC dye exhibits good fastness to dry and wet rubbing and washing fastness.

**Table 4.** Color Fastness to Rubbing and Washing

Sample Code	Rub Fastness		Washing Fastness						
	Dry	Wet	Acetate	Cotton	Polyamide	Polyester	Acrylic	Wool	Fading
P10	4/5	4	4/5	4	5	5	4/5	5	4
P20	4/5	4	4/5	4	5	5	4/5	5	4
P30	4/5	4	4/5	4	5	5	5	5	4
P40	4/5	4/5	5	4	5	5	5	5	4
P50	4/5	4/5	5	4	5	5	5	5	4
P60	4/5	4/5	5	4	5	5	5	5	4/5
P70	4/5	4/5	5	4/5	5	5	5	5	4/5
P80	5	4/5	5	4/5	5	5	5	5	4/5
P90	5	4/5	5	4/5	5	5	5	5	4/5

### CONCLUSIONS

The objective of this investigation is to examine the impact of dyeing duration on the dyeing process of cotton utilizing sodium copper chlorophyllin. 100% cotton textiles underwent dyeing with SCC as a natural dye without mordanting, and the influence of dyeing duration on SCC exhaustion was evaluated using the UV-Vis spectrophotometer. In the dyeing of cotton with reactive dyestuffs, dye uptake increased rapidly for a certain period depending on the dyeing time and then increased with decreasing acceleration (Özdemir & Tutak, 2013). When the % dye exhaustion behaviour of SCC was examined in response to increasing dyeing time, a similar behaviour was observed. Spectrophotometric analysis reveals that the dye exhaustion value steadily rises up to 60 minutes of dyeing time, after which further increases do not significantly enhance dye exhaustion. This suggests that a dyeing time of 60 minutes is optimal for achieving maximum dye uptake when dyeing cotton with SCC.

Color strength increases with increasing dyeing concentration and time (Shahid et al., 2016; Kamel et al., 2007). K/S values of the dyed cotton samples exhibit a noteworthy increase from 0.76 to 1.16 as the dyeing time extends from 10 to 90 minutes, indicating improved color strength with prolonged dyeing time. Notably, it reveals a positive correlation between dyeing time, dye exhaustion, and color strength (K/S).

Additionally, the color measurements and the fastness properties were assessed. Both dry and wet rubbing, as well as washing fastness, are rated as good to excellent, underscoring the suitability of the dyeing process for practical applications. These findings collectively contribute to a better understanding of the dynamics involved in dyeing cotton with SCC, paving the way for enhanced practices in environmentally friendly textile production.

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