

Simultaneous Coloring and Antibacterial Finishing to Cotton Through Environmentally-Friendly Way

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ABSTRACT

The present paper aimed to color 100% cotton fabrics while also ensuring antibacterial activity in an eco-friendly manner. For this purpose, cationized and pre-mordanted fabrics were dyed by using everlasting flower extract as a natural dye source. After dyeing process, antibacterial activity, CIEL^a*b^{*} color values, color strength (K/S), and fastness properties (washing, perspiration, rubbing) of the dyed fabrics were investigated, as well as the bursting strength of the dyed and undyed fabrics was also examined. The results showed that it was possible to ensure simultaneous coloring and antibacterial activity on the cotton fabrics by using a methanol extract of the everlasting flower as a natural dye source. In addition, applying the cationizing and mordanting processes to cotton fabrics before dyeing provided higher antibacterial activity, better fastness properties, and a darker color.

1. INTRODUCTION

Due to the public's enhanced environmental awareness and health concerns, sustainable, ecologic, nontoxic, and biodegradable products have recently gained more popularity [1]. Normally, textile dyeing and finishing processes include a number of steps in which hazardous chemicals are used. Scientists and textile industry firms have labored to reduce pollution caused by synthetic dyes and toxic chemicals currently used in textile applications [2,3]. Interest in the potential use of natural dyes has been growing, because of being safe, non-toxic, non-carcinogenic and biodegradable, having high compatibility with the environment, and being available in a range of natural shades as compared with synthetic dyes [3-6].

Natural dyes mainly contain various natural bioactive compounds, such as phenolic acids, flavonoids, alkaloids, terpenoids, essential oils, and natural color. These bioactive compounds have antimicrobial, antioxidant, anti-inflammatory activities and they can be used for hygiene, medical and pharmacological applications [2,4,7].

Antimicrobial textile materials have gained considerable popularity all over the world [4,8], since the textile

materials provide suitable environment for growth and multiplication of pathogenic microbes, leading to unpleasant odor, dermal infection, weakening of the substrate, discoloration, allergies, and other related diseases [7]. Various methods have been developed or are under development to give antimicrobial activity to textiles [5]. Although synthetic antimicrobial agents such as triclosan, metal and their salts, organometallics, phenols, etc. show good inhibition against bacteria, using natural eco-friendly agents such as natural dyes can eliminate their environmental risks [4]. For the reasons mentioned, with an ecological view, extract of everlasting flower (*Helichrysum armenium* DC. subsp. *araxinum* (Kirp.) Takht) was used as natural dye and antimicrobial agent in the present study.

The genus *Helichrysum*, belonging to the family of Asteraceae, consists of a few hundred species that are widespread throughout the world [9]. Plants of the genus *Helichrysum* are fertile producers of a host of secondary metabolites, including flavonoids, acetophenones, phloroglucinol, pyrones, triterpenoids and sesquiterpenes [10]. *Helichrysum* species are commonly used in Turkey and other parts of the world for their various biological properties, such as anti-inflammatory, antioxidant, and antimicrobial activities

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[11]. Albayrak et.al (2010) showed the antibacterial activities of methanol extract of *Helichrysum armenium* subsp. *araxinum* against *Aeromonas hydrophila*, *Bacillus brevis*, *B. cereus*, *Klebsiella pneumoniae*, *Pseudomonas aeruginosa*, and *Staphylococcus aureus* bacteria [11].

In the literature, there are a lot of studies about coloring of some textile materials with natural sources. When the studies are focused, it can be seen that most of them have interested in dyeing of cationic textile materials such as wool, silk and polyamide [1,3-8,12-14] since the cationic fibers can be dyed easier than anionic ones. Although cotton is the most preferred natural fiber [15], there are limited studies about natural dyeing of cotton materials [16,17]. A cationization process should be applied to the cotton materials before natural dyeing in order to achieve better dyeing.

The most common methodology for cationization is to introduce amino groups into the fiber [18]. There have been various reported cationic agents for the modification of cotton in the literature, such as polyamide-based epichlorohydrin type of polymers, dendritic polymers, quaternary ammonium-based compounds, glycidyltrimethyl ammonium chloride (Glytac), choline chloride, N-methylolacrylamide, and biopolymers like chitosan [18,19], starch and their derivatives, keratin hydrolysate from chicken feather, horn and hoofs [19]. In the present study, chitosan, one of the biopolymers, was preferred for cationization. A renewable polysaccharide-based cationic biopolymer, chitosan (CH), is the deacetylated derivative of chitin [20] and is an important alternative to commonly used compounds to functionalize cotton because of its protonated amino groups [21]. Chitosan is inexpensive, abundant, non-toxic, biocompatible, and eco-friendly material [21,22].

Due to the low affinity of most natural dyes to textile fibers, mordants are used to increase their affinity and color fastness [12]. Mordants are commonly metallic salts such as tannic acid, alum, chrome alum, sodium chloride [24] which can be fixed on the cationic fibers and interact with dye through coordination bonds [4,12]. Mordanting can be achieved in three ways as pre-mordanting (before dyeing), simultaneously mordanting, and post-mordanting (after dyeing). In this study, the pre-mordanting process was carried out with using aluminum potassium sulphate as mordant agent due to being non-harmful to the environment.

In the experimental study, it was aimed to ensure simultaneous coloring and antibacterial activity to 100% cotton fabrics in an eco-friendly way. For this purpose, the methanol extract of the everlasting flower was used as a natural dye source. The cationized, pre-mordanted 100% cotton knitted fabrics were dyed with the extract. After the dyeing process, CIEL*a*b* color values, color strength values, fastness properties (washing, perspiration, rubbing), and antibacterial activity of the dyed fabrics were

investigated, as well as the bursting strength tests were also applied to dyed and undyed fabrics.

2. MATERIAL AND METHOD

2.1. Material

Chitosan (medium molecular weight, Sigma-Aldrich), aluminium potassium sulphate dodecahydrate ($KAl(SO_4)_2 \cdot 12H_2O$, Merck), Rucon LFF (cross-linker, Rudolf Duraner) methanol (Isolab), rotatory evaporator (Scilogex), ultrasonic bath (Isolab), and water bath (Isolab) were used. The $KAl(SO_4)_2$ was chosen as mordanting agent since it is known as green agent [25]. The chemicals were used without any further purification.

The aerial parts of the everlasting plant used in the study were collected from Bingöl Metan Mountain in Turkey on June, 2021. The photo of the plant can be seen in Figure 1. The collected sample were dried in the shade and stored in a clean and dry environment. Plant identity was verified by Dr. Ömer Kılıç and the prepared herbarium sample is kept in Hacettepe University Faculty of Pharmacy Herbarium with the number HUEF-21042.



Figure 1. *Helichrysum armenium* subsp. *araxinum* in its habitat

100% cotton knitted fabric (Ne 30/1, 1*1 rib knit, 212 g/m²) was used in the experiments. The fabric was scoured and bleached by the supplier (İskur Group, Kahramanmaraş). A laboratory-type fulard (Termal), an IR dyeing machine (Termal), and a steamer (Ataç) were also used.

2.2. Method

2.2.1. Preparation of everlasting flower extract

The powder of the everlasting flower was extracted with methanol via the maceration process. The ratio of plant:solvent was determined as approximately 1:10 (w/v) in the water bath at 40 °C. The maceration process was

repeated three times for three hours for each one, and the combined solutions were filtered through filter paper, and they were then evaporated to dryness under vacuum at 40 °C with a rotary evaporator. In order to preserve the structure of such chemical substances in the plant, the temperature was not exceeded by 40 °C during both extraction and evaporation.

The photo of the produced extract is given in Figure 2. The yield of the extract was calculated as 13.79%.



Figure 2. The methanol extract of everlasting flower

A stock solution of the everlasting flower extract was prepared in distilled water at the concentration of 20 mg/ml by using ultrasonic bath. The prepared stock solution was stored in the freezer at -24 °C for further usage.

2.2.2. Cationization process of the cotton fabrics

The cotton fabrics were cationized with chitosan. The concentration of chitosan was chosen at two different levels as 10 g/L and 20 g/L and it was applied to cotton samples by pad-dry-cure method. In order to prepare the application solution, the chitosan was dissolved in a %1 (w/v) acetic acid solution, and 50 g/L cross-linker agent was added to the solution. The fabric samples were squeezed to 96% pick up at fulard, then dried at 100 °C and cured for 2 minutes at steamer at 110 °C. Some of the fabric samples were saved without cationizing to investigate the effect of the cationization process on the results.

2.2.3. Mordanting process

The pre-mordanting process was carried out with 5 g/L concentration of $KAl(SO_4)_2$ at 98 °C for 60 minutes with a liquor ratio of 1:10 by using IR dyeing machine. After the process, the samples were rinsed with cold water and dried at 100 °C. In order to research the effect of the mordanting process on the results, some of the fabric samples were saved without mordanting.

2.2.4. Dyeing process

The different pretreated fabric samples (only cationized, only mordanted, both cationized and mordanted, and neither cationized nor mordanted) were dyed. The dyed

fabric samples can be seen in Table 1. The dyeing processes were carried out with the stock extract solution with a liquor ratio of 1:5 at 40 °C for 30 minutes by using an IR dyeing machine. After the dyeing, the samples were washed with cold water for 10 minutes and then dried at the room temperature.

Table 1. The dyed fabric samples

| Sample Number | Chitosan concentration (g/l) | Mordant concentration (g/l) |
|---------------|------------------------------|-----------------------------|
| 1 | 0 | 0 |
| 2 | 10 | 0 |
| 3 | 20 | 0 |
| 4 | 0 | 5 |
| 5 | 10 | 5 |
| 6 | 20 | 5 |

2.3. The research methods

Bursting strength: The test was applied to dyed and undyed samples according to the ISO 13938-2:2019 standard [26] as three replications and the average of the results was calculated.

CIEL^a*b^{*} color value and color strength (K/S) value:

The CIEL^a*b^{*} values and the reflectance (R) values at wavelengths ranging from 400 to 700 nm of the dyed samples were measured. The measurements were carried out under D65 daylight and an aspect of 10° with a spectrophotometer (Datacolor SF 600 model). The results were measured over three replications, and the average of the results was calculated. The K/S values were calculated using the Kubelka-Munk equation by using the R-value at the wavelength (410 nm) at which each fabric sample had maximum absorption.

Color fastness test: In order to investigate the color fastness results of the dyed samples, fastness tests to washing [27], acidic and alkaline perspiration [28], and wet and dry rubbing [29] were carried out.

Antibacterial activity: The antibacterial activities of the everlasting flower extract and the fabric samples were investigated against Gram-negative bacteria (*Escherichia coli* ATCC 8739) and Gram-positive bacteria (*Staphylococcus aureus* ATCC 6538), since they are the major microorganisms responsible for 25% of hospital infections and popular test organisms which are resistant to common antimicrobial agents [30]. The tests were carried out in Ege University's Microbiological Analysis Laboratory (Egemikal). While the antibacterial activity of the extract was investigated through the disc diffusion method, it was researched via the AATCC 100 method for fabrics. The tests were applied as three replications.

3. RESULTS AND DISCUSSION

The antibacterial activity results of the stock solution of everlasting flower extract assessed according to the disc

diffusion method against *S. aureus* and *E. coli* bacteria are given in Figure 3. The inhibition zones formed by the extract solution against *S. aureus* and *E. coli* are 11.2 and 8 mm, respectively. Thus, it is possible to say that the prepared stock extract solution had antibacterial activity against both tested bacteria species.

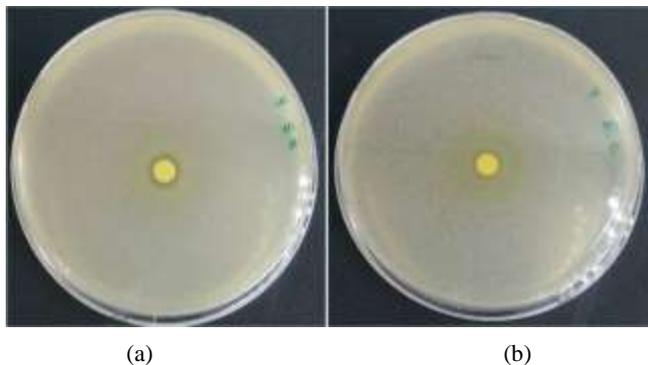


Figure 3. The antibacterial activity results of everlasting extract

The antibacterial activity results of the dyed samples assessed according to the AATCC 100 method against *E. coli* and *S. aureus* bacteria can be found in Table 2. The results show that the fabrics gained antibacterial activity against both *E. coli* and *S. aureus* bacteria. The fabric sample that was not subjected to cationizing and mordanting processes had less activity against *E. coli* than other samples since it had less dye. As *E. coli* is a more resistant bacteria than *S. aureus*, the result was observed more specifically for *E. coli*. As a result, it can be said that it is possible to provide both coloring and antibacterial activity simultaneously to a cotton fabric with a natural dyeing process through applying cationizing and mordanting processes before dyeing.

The bursting strength results of the fabric samples before and after dyeing can be seen in Figure 4. The bursting strength of the untreated raw fabric was 390,8 KPa, as shown in Figure 4, and the strength of the fabrics decreased after the cationizing and mordanting processes. However, the decline was not crucial and occurred at 14%. Generally, the increase in the chitosan concentration had no negative effect on the strength property. In addition, the dyeing process had a negligible effect on the strength properties of the samples, since no harsh chemicals were used in the natural dyeing process.

Table 3 shows the CIEL*a*b* color values as well as the color strength (K/S) values of the dyed samples. It is commonly known that while the L* value indicates the lightness and darkness, the b* value gives information about the yellowness and blueness, and the a* value is about the reddish and greenish tones of the fabric samples in the CIEL*a*b* color system. The L* value changes between 0 and 100, and the color becomes lighter as the L* value increases. The increase in the a* and b* values indicates that the color is becoming more reddish and yellowish, respectively. In addition, the increase in the K/S value means that the fabric is darker. When the results in Table 3 are focused, it can be clearly seen that both the application of the cationizing process and the use of mordants increased the dye uptake. Furthermore, the increase in the mordant concentration also increased the depth of color. In addition, the b* value increased significantly, especially when the mordant was used. This circumstance shows that the use of mordant had an effect on the shade of the fabric samples. The application of the mordant process after the chitosan treatments caused the color to have greater depth and strength. Thus, it is possible to say that in order to have a naturally colored fabric with a darker shade, it is necessary to apply both the cationizing and mordanting processes.

Table 2. % Reduction in bacteria after 24 hours

| Sample number | <i>E. coli</i> | | | <i>S. aureus</i> | | |
|------------------|----------------|--------------|-------------|------------------|--------------|-------------|
| | 0.hour | 24. hours | % Reduction | 0.hour | 24. hours | % Reduction |
| Untreated fabric | 560000 | Reproduction | - | 263000 | Reproduction | - |
| 1 | 560000 | 45000 | 91.96 | 263000 | 100 | 99.96 |
| 2 | 560000 | 610 | 99.89 | 263000 | 50 | 99.98 |
| 3 | 560000 | 3380 | 99.40 | 263000 | 970 | 99.63 |
| 4 | 560000 | 1430 | 99.74 | 263000 | 75 | 99.97 |
| 5 | 560000 | 650 | 99.88 | 263000 | 80 | 99.97 |
| 6 | 560000 | 50 | 99.99 | 263000 | 300 | 99.89 |

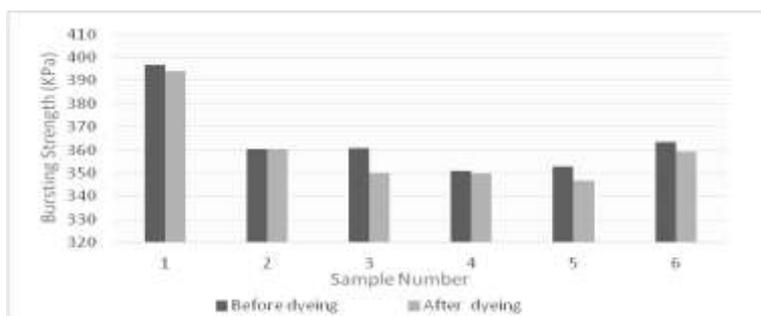


Figure 4. The bursting strength results of the fabric samples before and after dyeing

The results of washing, perspiration (acidic and alkaline), and rubbing (dry and wet) fastness of dyed samples are given in Table 4. Generally, the wash fastness of the fabric samples with dark colors has shown more staining than those with light colors. For that reason, the highest fastness

results were obtained for the samples to which neither the cationizing nor mordanting processes were applied, since they had less dye. Besides, the washing and perspiration fastness results got better when the mordanting process was applied to the fabric samples before the dyeing process.

Table 3. The results of CIEL*a*b* color values and color strength (K/S) values

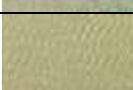
| Sample number | Photos | L* | a* | b* | K/S |
|---------------|---|-------|-------|-------|-------|
| Undyed fabric |  | 90.44 | -0.07 | 1.87 | - |
| 1 |  | 79.02 | -3.91 | 25.95 | 3.19 |
| 2 |  | 71.09 | -3.79 | 27.98 | 6.97 |
| 3 |  | 70.35 | -2.67 | 33.07 | 9.89 |
| 4 |  | 77.43 | -3.95 | 45.67 | 7.96 |
| 5 |  | 71.13 | -4.51 | 40.91 | 11.22 |
| 6 |  | 67.84 | -2.95 | 42.89 | 11.84 |

Table 4. The results of color fastness of dyed samples

| Sample number | Fading | Washing | | | | | | Perspiration | | | | | | | | | | Rubbing | | | | |
|---------------|--------|---------|-----|-----|-----|-----|-----|--------------|-----|-----|-----|-----|----------|-----|-----|-----|-----|---------|-----|-----|-----|-----|
| | | As | Co | PA | PES | PAC | WO | Acidic | | | | | Alkaline | | | | | Wet | Dry | | | |
| | | | | | | | | As | Co | PA | PES | PAC | WO | As | Co | PA | PES | | | PAC | WO | |
| 1 | 5 | 4/5 | 3/4 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 3/4 | 3/4 | 4/5 | 4 | 4 | 4/5 | 2/3 | 3/4 | 4 | 4 | 4 | 4 | 4 | 4/5 |
| 2 | 4 | 4 | 2/3 | 4 | 4 | 4 | 4 | 4 | 3 | 3 | 4/5 | 4 | 4 | 4 | 2/3 | 3 | 4 | 4 | 4 | 3/4 | 3/4 | 4 |
| 3 | 3/4 | 4 | 2/3 | 4 | 4 | 4 | 4 | 4/5 | 3 | 3 | 4 | 4 | 3 | 4 | 1/2 | 2/3 | 4 | 4 | 4 | 3 | 3/4 | 4 |
| 4 | 4 | 4/5 | 3 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 3 | 3 | 4 | 4 | 4 | 4 | 2/3 | 3 | 3/4 | 3/4 | 3/4 | 3/4 | 3/4 | 4 |
| 5 | 4/5 | 4 | 3 | 4 | 4/5 | 4/5 | 4/5 | 4/5 | 3 | 3 | 4/5 | 4/5 | 4 | 4 | 2/3 | 3 | 4 | 4 | 4 | 3/4 | 3 | 4 |
| 6 | 4 | 4/5 | 3 | 4 | 4/5 | 4/5 | 4 | 4/5 | 3 | 3 | 4 | 4 | 3/4 | 4 | 2/3 | 2/3 | 3 | 3 | 3 | 3 | 3 | 4 |

4. CONCLUSION

The subject of applying dyeing process, which has crucial importance among the textile finishing process, with natural dyes and eco-friendly way has been more significant with increasing environmental awareness. For this reason, researchers have studied on coloring of the textile materials with different natural dye source. Furthermore, the vast majority of natural sources have antimicrobial activity, allowing for simultaneous coloring and antibacterial activity in textiles. In the present study, it was aimed to

provide simultaneous coloring and antibacterial activity to 100% cotton knitted fabrics with the everlasting flower extract. For this purpose, firstly, a methanol extract of the everlasting flower was prepared, and the antibacterial activity of the extract was examined. Then, the fabrics were cationized by using chitosan as a natural biopolymer and pre-mordanted with $KAl(SO_4)_2$ as a green mordanting agent. The treated fabrics were dyed with the prepared extract. Following the dyeing process, the fabrics' antibacterial activity, CIEL*a*b* color values, and color fastness to washing, perspiration, and rubbing were



evaluated. In addition, the bursting strength of the dyed and undyed fabrics was also measured. The results can be summarized as given below.

- The methanol extract of everlasting flower has antibacterial activity against both *S. aureus* and *E. coli* bacteria species.
- The bursting strength results are negatively affected by the cationizing and mordanting processes. However, the decline in the results is lower than 15% and not crucial. The increase in chitosan concentration has no crucial effect on the strength property, and the usage of both chitosan and mordant together have the most effect on the results.
- The applications of cationizing and mordanting processes increase the dye uptake, and treated fabrics dye with darker shade. In addition, the mordanting

process have positive effect on the fastness properties of the samples.

- The dyed samples after cationizing and mordanting processes gain high antibacterial activity against both *S. aureus* and *E. coli* bacteria.

As a conclusion, the %100 cotton fabrics can be colored with everlasting flower extract with gaining antibacterial activity. In order to achieve dyeing process with high antibacterial activity, good fastness, and dark color, the cationizing and mordanting processes are recommended before dyeing.

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