INVESTIGATION OF COLOR AND ANTIMICROBIAL PROPERTIES OF WOOL FABRICS DYED WITH *POLYGONUM COGNATUM* NATURAL DYE EXTRACTS

POLYGONUM COGNATUM DOĞAL BOYA EKSTRAKTLARI İLE BOYANMIŞ YÜN KUMAŞLARIN RENK VE ANTİMİKROBİYAL ÖZELLİKLERİNİN ARAŞTIRILMASI

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ABSTRACT

In the textile industry, natural dyestuffs are used to color the fabric and give antibacterial properties. In this study, wool fabrics were dyed with natural dyes extracted from *Polygonum cognatum* plant collected from Şebinkarahisar district of Giresun. Before dyeing, the mordanting processes were performed with 3 different mordant materials. After the mordanting process, dyeing was carried out with 2 different concentrations. At the end of the dyeing processes, color and antibacterial properties of dyed fabrics were examined. Increased concentration caused the improvement of color strength values (K/S). As a result of the antibacterial test performed according to AATCC 100 method, it was observed that all samples showed antibacterial properties against *Escherichia coli* ATCC 25922 at 24 contact time. In addition, sample, mordanted with copper sulfate ($CuSO_4.5H_2O$) and dyed with 25% dyestuff concentration, have the best antibacterial property against *Staphylococcus aureus* ATCC 25923 with 99.65%.

Keywords: *Polygonum cognatum*, Natural Dye Extract, Wool, Natural Dyeing

ÖZET

Tekstil endüstrisinde, kumaşı renklendirmek ve antibakteriyel özellikler vermek için doğal boyar maddeler kullanılır. Bu çalışmada, yünlü kumaşlar Giresun'un Şebinkarahisar

ilçesinden toplanan *Polygonum cognatum* bitkisinden ekstrakte edilen doğal boyalarla boyanmıştır. Boyama işleminden önce, mordanlama işlemleri 3 farklı mordan malzeme ile gerçekleştirilmiştir. Mordanlaştırma işleminden sonra boyama 2 farklı konsantrasyonda gerçekleştirilmiştir. Boyama işlemlerinin sonunda, boyalı kumaşların renk ve antibakteriyel özellikleri incelenmiştir. Konsantrasyon değerlerinin artışı renk mukavemeti değerlerinin (K / S) artmasına neden olmuştur. AATCC 100 yöntemine göre yapılan antibakteriyel test sonucunda tüm numunelerin *Escherichia coli* ATCC 25922'ye karşı antibakteriyel özellikler sergilediği gözlemlenmiştir. Bunlara ek olarak, bakır sülfat (CuSO4.5H2O) ile mordanlanmış ve % 25 boyar madde konsantrasyonu ile boyanmış numune,% 99.65 ile *Staphylococcus aureus* ATCC 25923'e karşı en iyi antibakteriyel özelliğe sahiptir.

Anahtar Kelimeler: *Polygonum cognatum,* Doğal Boya Ekstraktları, Yün, Doğal Boyama

1. INTRODUCTION

The textile industry is among the industries that pollute the environment. Especially in terms of pollution of water resources, the textile industry ranks second after the agricultural industry. According to the World Bank data, one of the main causes of industrial water pollution is the use of synthetic dyes. In addition, it is known that some of the synthetic dyes used in the industry contain carcinogenic substances (Srivastava and Singh, 2019). For these reasons, synthetic dyes cause environmental problems (El-Nagar et al., 2005). Many parts of plants are used in many fields such as food, health, textile and cosmetics especially as biotechnologically (Ercisli et al., 2008; Cüce et al., 2017b) In the last quarter century, the interest in natural dyestuffs has increased in terms of eliminating all these problems and in terms of sustainability (Merdan et al., 2014; Dayioğlu et al., 2016). Some scientists have stated that different natural dyestuffs have antibacterial, ultra-violet protection, flame retardancy and insect repellent properties (Kilinc et al., 2015). Natural dyes are preferred in the textile industry not only because of their coloring properties but also because of these properties. To date, various natural dyestuffs have been studied to provide antibacterial properties to textile surfaces, such as, *Acacia catechu* (L. f.) Willd., *Quercus infectoria* G. Oliver, *Rubia cordifolia* L., *Rumex maritimus* L., *Chamaecyparis lawsoniana* (A. Murr.) Parl., *Viburnum opulus* L. and *Allium cepa* L. etc (Doğan et al., 2007; Kilinc et al., 2015; Şapcı et al., 2017).

Polygonum cognatum Meissn., commonly known as "*Madımak"* in Turkey, is an medicinal and aromatic herbaceous perennial plant belonging to the *Polygonaceae* family. *P. cognatum*

is a wild plant species found in especially Central Anatolia of Turkey. This species, is also distributed in Şebinkarahisar district of Giresun province, can be seen at heights of 720-3000 m in the countryside, road and field edges (Davis, 1967; Tatlı, 1988; Baytop, 1999; Yıldırım et al., 2003; Demir, 2006). *P. cognatum* is considered as a natural antioxidant thanks to its high vitamin C, carotenoids, polar and apolar antioxidants. Because of these active phytochemicals, it has been used in alternative medicine as a diuretic and to treat diabetes mellitus (Tatlı, 1988; Baytop, 1999; Dereli et al., 2019).

In this study, wool fabrics were dyed with natural dyes extracted from *P. cognatum* plant collected from Şebinkarahisar district of Giresun. Mordanting process was carried out with potassium aluminum sulfate, copper sulfate and iron sulfate chemicals before dyeing of wool fabrics. Dyeing process was carried out in two different dye concentrations (25% and 50%). Antibacterial activity and color strength properties of fabrics were examined after dyeing process.

2. MATERIALS AND METHODS

2.1 Materials

2.1.1 Fabric

In this study, 100% woven wool fabric was used. The fabric specimens were 1:1 plain fabrics (37 ends cm^{-1}, 44 Nm; 31 picks cm^{-1}, 44 Nm; 150 g/m^2).

2.1.2 Plant Material

The fresh aerial parts of the *P. cognatum* (combined above-ground parts including stem, leaves and flowers) were collected from the indigenous natural populations of Şebinkarahisar-Toplukonak village (40° 22′ 38.17″ N, 38° 34′ 43.58″ E; 1771 m), Giresun, Turkey, during the April-May 2018 and 2019 early in the morning without too much sunlight. The initially, fresh weights of the plantlets transferred to the laboratory were calculated to determine the yield amount. The plant materials were allowed to dry in a well-ventilated condition without direct sunlight. The dry weights were then determined and stored for extraction studies.

2.2. Method

2.2.1. Extraction

Initially, the weight of the glass container to which the solvent was introduced was determined. 10 g air-dried and the thoroughly ground *P. cognatum* samples were placed in the cellulosic cartridge and extracted with 250 ml of methanol (MeOH) for 6 hrs in soxhlet

apparatus. Finally, the MeOH was wholly evaporated in a rotary evaporator at less than 80 °C under reduced pressure. Finally, the final weight of the glass container was weighed to determine how much natural fabric dye was obtained from 10 grams of the sample. The samples were then stored at + 4 ºC until analyses and apply fabrics.

2.2.2. Mordanting Processes

Mordanting process was done before dyeing. Potassium aluminum sulfate ($KAl(SO_4)_2.12H_2O$), copper sulfate ($CuSO_4.5H_2O$) and iron sulfate ($FeSO_4.7H_2O$) chemicals were used for mordanting process. Mordant concentrations were determined based on fabric weight. The information about the mordant process is given in the Table1.

Table 1. Mordanting recipe with three different mordants on wool fabric dye

	Mordants		
Mordanting	**$KAl(SO_4)_2.12H_2O$**	**$FeSO_4.7H_2O$**	**$CuSO_4.5H_2O$**
% Mordant	20	3	5
Temperature (°C)	100	100	100
Time (min)	60	60	60

$KAl(SO4)_2.12H_2O$ = Potassium aluminum sulfate, $FeSO_4.7H_2O$ = Iron sulfate, $CuSO_4.5H_2O$ = Copper sulfate

2.2.3. Dyeing Process

P. cognatum dye pigments obtained by soxhlet extraction method were dissolved in 1 ml Dimethyl Sulfoxide (DMSO) which was added to 10 ml test tube before staining for each dyeing processes. Vortex mixer was used to accelerate the dissolution process. In dyeing process, two different concentrations of dyestuffs, 25% and 50%, were used based on fabric weight. Wool fabrics were dyed in liquor ratio of 1:50 at the boiling temperature for 30 minute. After dyeing, samples were subjected to cold, hot (at boiling point) and cold washings respectively. As a final treatment, the washed samples were squeezed and dried at room temperature.

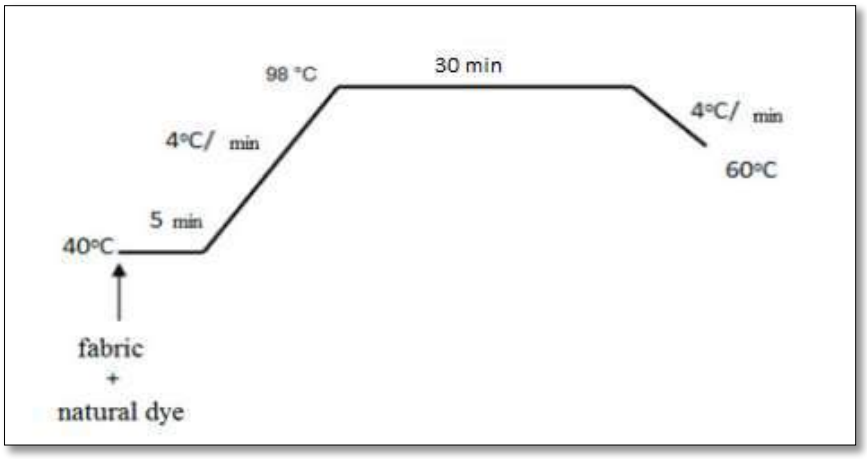

Figure 1. Dyeing Process

2.2.4. Color Measurement

Color strength (K / S) values of dyed fabric samples were measured on Konica Minolta CM-3600d spectrophotometer. The calculations were performed with respect to D65 light source and 10^{o} standard observer. % reflectance values in the visible range (400-700nm) were measured. Obtained % reflectance values (R) were converted to K / S values by using Kubelka-Munk equation (Eqn 1) in the instrument software.

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

In the study, color coordinate (L*: lightness (0 = black, 100 = white), L = lightness; a* ± = red/green; b* ± = yellow/blue) values were also measured.

2.2.5 Antibacterial Activity

For the AATCC 100 method, a square sample of dyed wool fabric (4.8 × 4.8 cm) was used. In the AATCC 100 method, an inoculum with 100 ml of NB and incubated for 24 h at 37 ± 1°C was used. Its bacteria concentration was adjusted with NB 105 cells ml^{-1}. Then, 1 ± 0.1 ml of the diluted inoculum was placed in each sample. 50 ml sterile distilled water was added and the sample 1. This was vigorously shaken for 5 min. 100 µL of the liquid sample was placed on NA agar plate. This represents "zero contact time". The other samples were incubated for 24 h at 37 ± 1 °C. After the incubation period, 50 ml sterile distilled water was added and was vigorously shaken for 5 min. 100 µL of the liquid sample was placed on NB agar plate. This represents "24 contact time". All plates were incubated 37 ± 1 °C for 24 hours. After incubation, colonies of recovered were counted used to determine percent reductions. In this

study, *Staphylococcus aureus* ATCC 25923 (Gram positive) and *Escherichia coli* ATCC 25922 (Gram negative) were used as test organisms. The following equation (Eqn 2) was used to determine the percent reduction in the number of bacteria.

$$\% R = 100 (B - A) / B \quad (2)$$

In equation 2, R is % reduction, A is the number of bacteria recovered from the inoculated treated test specimen swatches in the jar incubated over desired contact period ("24" hours) and B is the number of bacteria recovered from the inoculated treated test specimen swatches in the jar immediately after inoculation (at "0" contact time).

3. RESULTS AND DISCUSSION

3.1. Colorimetric Measurements

P. cognatum has a yield ratio of 24.69% in obtaining dry weight from fresh weight. In addition to this, the amount of extraction obtained from dry weight was calculated as 24.16%. The wool samples mordanted with different mordant materials and dyed with dyestuff from *P. cognatum* are shown in Figure 1. When Figure 1 is examined subjectively, it is seen that the samples which were mordanted with $KAl(SO_4)_2.12H_2O$ have more yellow nuances, the samples which were mordanted with $FeSO_4.7H_2O$ have more red and the samples which were mordanted with $CuSO_4.5H_2O$ have more green nuances.

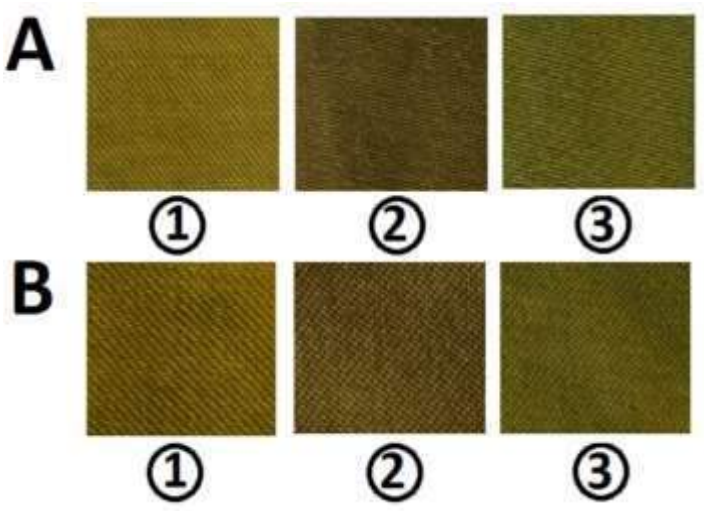

Figure 2. Natural dyed wool fabric with *P. cognatum*, **(A)** 25% natural dye concentration, **(B)** 50% natural dye concentration. Mordanting processes **(1)** 20% $KAl(SO4)_2.12H_2O$, **(2)** 3% $FeSO_4.7H_2O$ **(3)** 5% $CuSO_4.5H_2O$

In color measurements after dyeing process, it is seen that increase of dye concentration causes increase in sample darkness. Samples, mordanted with $KAl(SO4)_2.12H_2O$, appear to be closer to the color of the dye obtained from *P. cognatum*. This result is due to the fact that $KAl(SO4)_2.12H_2O$ imparts a white color to the wool fabric prior to dyeing. Woolen fabrics, mordanted with $CuSO_4.5H_2O$, have a blue nuance before dyeing, so it is observed that green and blue nuances are increased when compared to samples, mordanted with $KAl(SO4)_2.12H_2O$. Compared to other processes, it is seen from the color coordinates that the increase in red and blue nuances is highest in the samples treated with iron sulfate. The color coordinates are given in the Table 2.

The natural dyes obtained from plants go back to ancient times. People have used different plant species to obtain rope and fabric in different colors and tones (Adeel et al., 2019a and b). Ozturk et al. (2013) in their studies of the extracts obtained from the plant *P. cognatum* have stated that the yellow color. In the present study, textile surfaces painted in yellow, brown and green tones were obtained with the effect of mordant materials. These results support each other.

Table 2. Colour coordinates of dyed samples

Sample	Colour Coordinates		
	L*	a*	b*
Mordanted with $KAl(SO4)_2.12H_2O$ and dyed with 25% dyestuff	78.3132	0.6454	27.2102
Mordanted with $KAl(SO4)_2.12H_2O$ and dyed with 50% dyestuff	55.3734	1.0462	34.2186
Mordanted with $FeSO_4.7H_2O$ and dyed with 25% dyestuff	54.3074	1.5013	19.3644
Mordanted with $FeSO_4.7H_2O$ and dyed with 50% dyestuff	42.9498	2.1246	18.5738
Mordanted with $CuSO_4.5H_2O$ and dyed with 25% dyestuff	55.4599	-5.9493	23.4540
Mordanted with $CuSO_4.5H_2O$ and dyed with 50% dyestuff	49.5777	-3.6279	26.0468

$KAl(SO4)_2.12H_2O$ = Potassium aluminum sulfate, $FeSO_4.7H_2O$ = Iron sulfate, $CuSO_4.5H_2O$ = Copper sulfate, L*: lightness (0 = black, 100 = white), L = lightness; a* ± = red/green; b* ± = yellow/blue

After dyeing at two different concentrations, it is seen from the following figures that the increase in concentration increases the color strength (K / S) value.

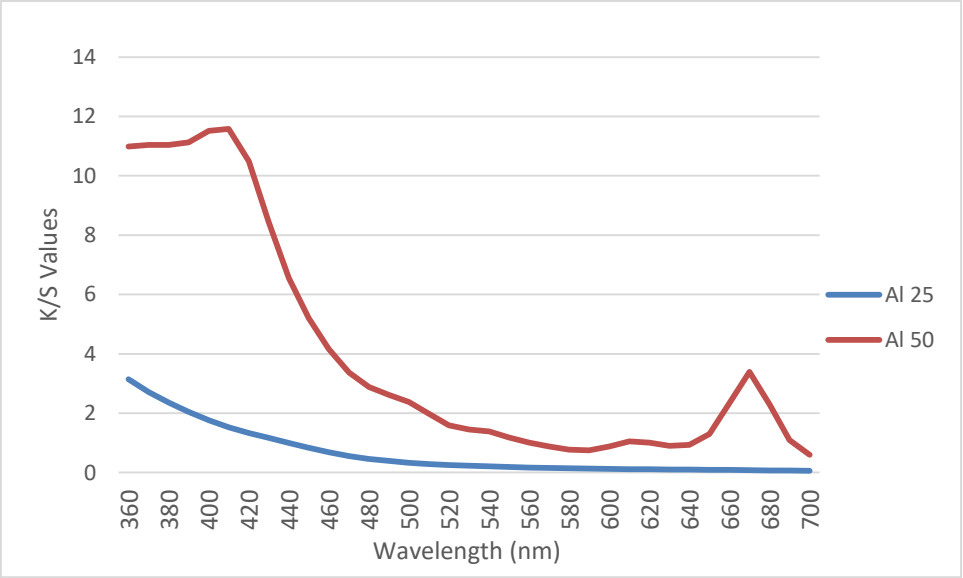

Figure 3. Color strength of samples mordanted with 20% $KAl(SO4)_2.12H_2O$ and dyed at 25% and 50% dyestuff concentration.

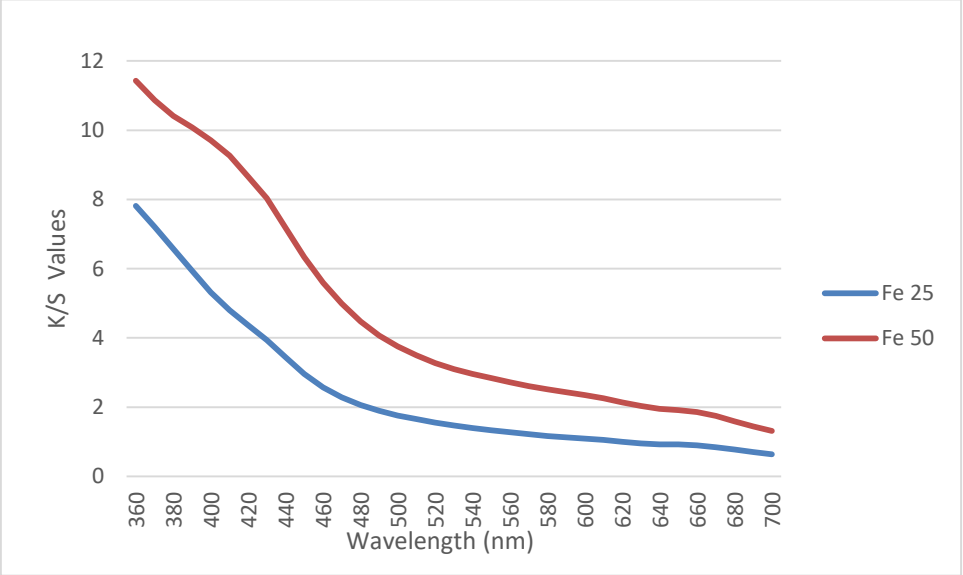

Figure 4. Color strength of samples mordanted with 3% $FeSO_4.7H_2O$ and dyed at 25% and 50% dyestuff concentration.

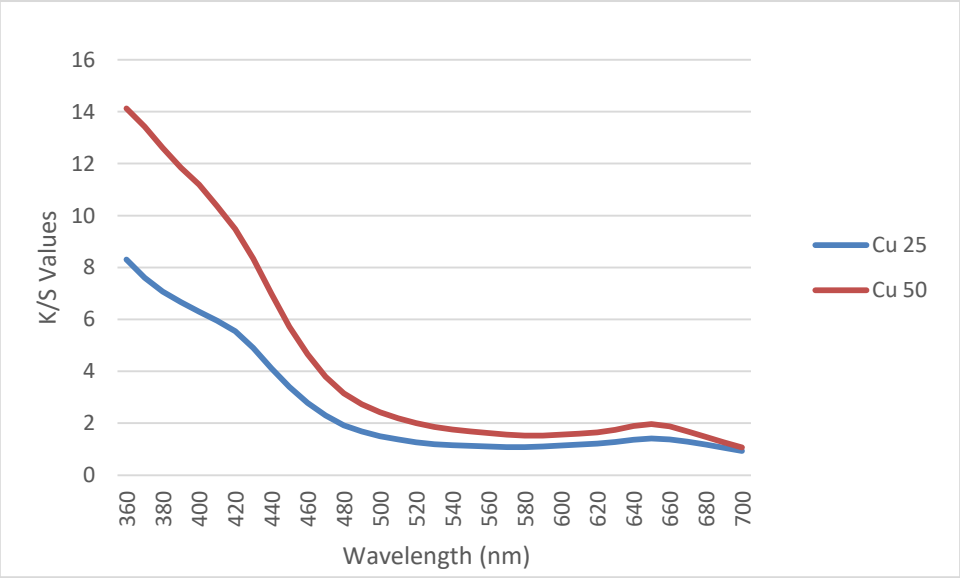

Figure 5. Color strength of samples mordanted with 5% $CuSO_4.5H_2O$ and dyed at 25% and 50% dyestuff concentrations.

3.2. Antibacterial Activity

Antibacterial activity was observed in all tested samples. When the data given in Table 3 are examined, $KAl(SO4)_2.12H_2O$ and $CuSO_4.5H_2O$ are similar in terms of antibacterial activity. However, it is observed that the samples, mordanted with iron sulfate, have less antibacterial activity than the others. The antibacterial activity of wool fabrics dyed with *P. cognatum* extract is thought to result from the phenolic components of the plant [13]. Antibacterial activity-related values and visuals are given in the Table 3, Figure 6 and figure 7. In recent years, the production of antibacterial fabrics has recently gained momentum all over the world (Ammayappa and Moses, 2009; Prabhu et al., 2011; Jafari et al., 2014). Many researchers have preferred a chemical compound as an antibacterial agent in their studies (Shahidi and Moazzenchi, 2019; Noorian et al., 2019; Gao et al., 2019) However, in order to prevent the harmful effects of chemicals in the long term, naturally obtained dyestuffs are now preferred (Gupta et al., 2001; Chan et al., 2009; Feiz and Norouzi, 2014). Han and Yang (2005) used preferred curcumin as an antibacterial agent against *S. aureus* and *E. coli*. These researchers have obtained very effective antibacterial results similar to our results on wool fabric dyeing with natural dye. In another study, Prabhu et al. (2014) observed the antibacterial activity of turmeric and pomegranate dyed wool, cotton and silk fabric. As a result of their work they have observed 88.36-100% reduction in *E. coli* and *S. aureus* bacteria. In our study, bacterial

colonies were decreased between 75.2% and 100% with comparison to Prabhu's study. The data obtained from these studies support the data in our study.

Table 3. Antibacterial activities of *P. cognatum* dyed wool fabrics with AATCC 100.

Sample	Bacteria	Antibacterial Activity (% R)
Mordanted with 20% $KAl(SO4)_2.12H_2O$ and dyed with 25% dyestuff	*E. coli*	100
	S. aureus	98.19
Mordanted with 20% $KAl(SO4)_2.12H_2O$ and dyed with 50% dyestuff	*E. coli*	100
	S. aureus	99.56
Mordanted with 3% $FeSO_4.7H_2O$ and dyed with 25% dyestuff	*E. coli*	99.93
	S. aureus	82.55
Mordanted with 3% $FeSO_4.7H_2O$ and dyed with 50% dyestuff	*E. coli*	100
	S. aureus	75.22
Mordanted with 5% $CuSO_4.5H_2O$ and dyed with 25% dyestuff	*E. coli*	100
	S. aureus	99.65
Mordanted with 5% $CuSO_4.5H_2O$ and dyed with 50% dyestuff	*E. coli*	100
	S. aureus	99.42

$KAl(SO4)_2.12H_2O$ = Potassium aluminum sulfate, $FeSO_4.7H_2O$ = Iron sulfate, $CuSO_4.5H_2O$ = Copper sulfate, % R = % Reduction

The antibacterial test results show that the bacteria colonies counted after 24 hours are less than the bacteria colonies counted after 0 hours. This result is observed in Figure 6 and 7.

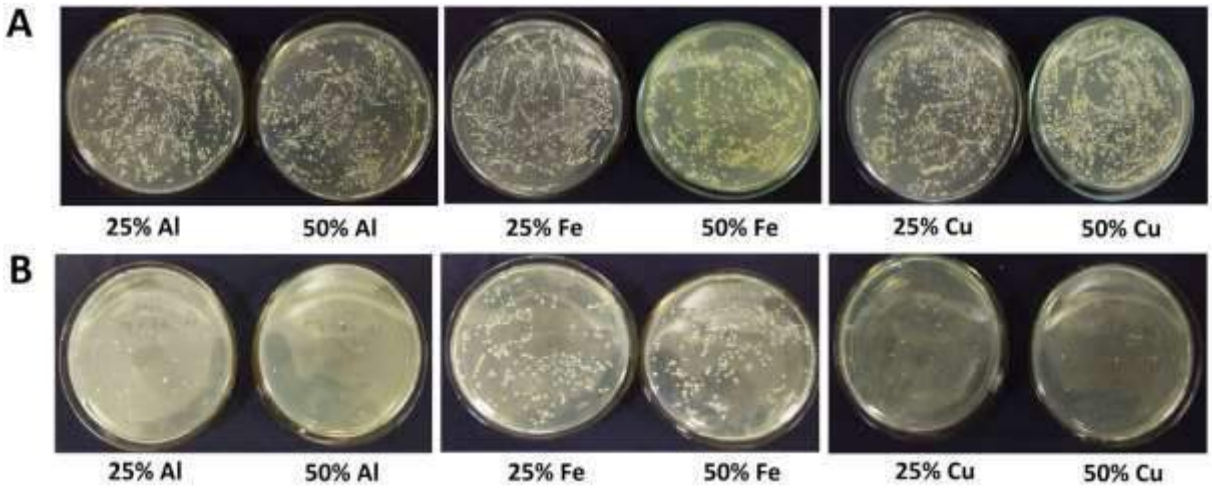

Figure 6. Antibacterial effect of 25% and 50% concentrations of *P. cognatum* natural dye extract applied to wool fabrics with AATCC 100 after pre-mordanting with $KAl(SO4)_2.12H_2O$, $FeSO_4.7H_2O$ and $CuSO_4.5H_2O$ on *S. aureus* ATCC 25923, **(A)** 0 h. treatments **(B)** 24 h. treatments

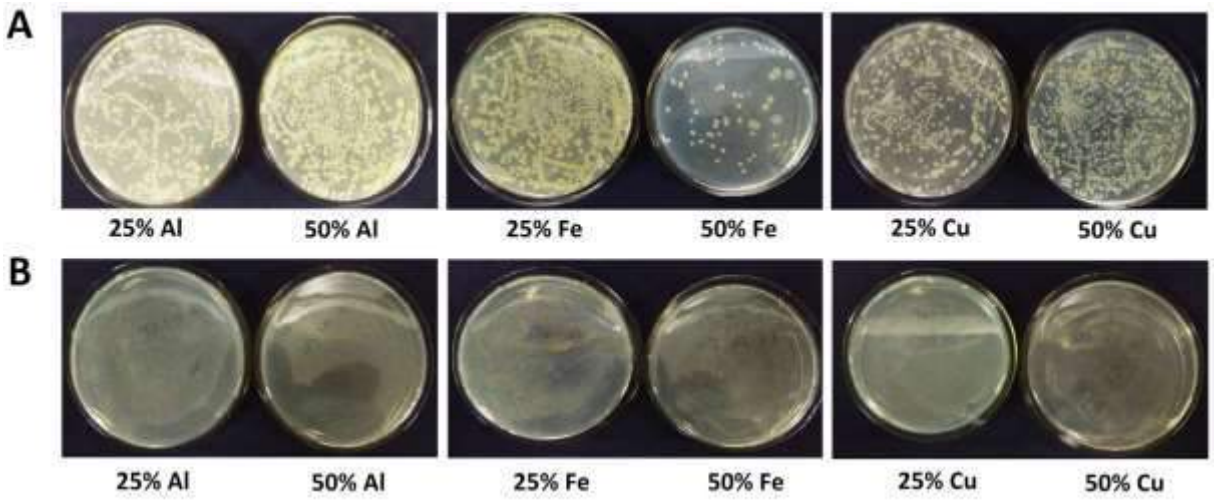

Figure 7. Antibacterial effect of 25% and 50% concentrations of *P. cognatum* natural dye extract applied to wool fabrics with AATCC 100 after pre-mordanting with $KAl(SO4)_2.12H_2O$, $FeSO_4.7H_2O$ and $CuSO_4.5H_2O$ on *E. coli* ATCC 25922, **(A)** zero contact time treatments **(B)** 24 contact time treatments

4. Conclusion

The mordanting process made with three different mordants ($KAl(SO4)_2.12H_2O$, $FeSO_4.7H_2O$ and $CuSO_4.5H_2O$) caused the fabric surfaces to have different colors after dyeing. This result shows that wool fabric can be dyed with the extract of *P. cognatum* plant in different colors and tones. In addition, dyed materials can be used in the field of medical textiles because of the antibacterial properties. The fastness of the samples and antibacterial properties of the samples after washing will be examined in future studies. As a result of this study, the obtained data show that *P. cognatum* plant can be used as antibacterial natural dyestuff in textile industry.

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