



# Investigation of Color, Fastness, and Antimicrobial Properties of Wool Fabrics Dyed with *Rosa Canina* Leaf Extract

Mustafa Cuce

Department of Food Technology, Şebinkarahisar School of Applied Sciences, Giresun University, Giresun, Turkey

## ABSTRACT

Natural dyestuffs are innovative natural resources alternative to chemicals in the textile industry due to their antibacterial properties. The objective of this study was to reveal the color, fastness, and antibacterial efficiency of *Rosa canina* leaf extract in wool fabric dyeing. Aluminum potassium sulfate (KAl(SO<sub>4</sub>)<sub>2</sub>·12H<sub>2</sub>O), copper sulfate (CuSO<sub>4</sub>·5H<sub>2</sub>O), and iron sulfate (FeSO<sub>4</sub>·7H<sub>2</sub>O) metal salts were preferred in the pre-mordanting process. Two different natural dye concentrations (25% and 50%) were tested in mordant and non-mordant wool fabrics. The fastness values were ranked between medium and good grades with 2–6. One of the remarkable results was that the fastness values of the un-mordanted dyed samples were acceptable with 5. In disc diffusion analysis, ultrasonic water bath CH<sub>3</sub>OH extracts were found to be more effective against *Staphylococcus aureus* ATCC 25923 and *Escherichia coli* ATCC 25922 with 16.4 mm and 7.51 mm, respectively. Also, the dyed wool fabrics showed remarkable antimicrobial activity (reduction rates: w46e100) against two different bacteria according to AATCC 100 method.

## 摘要

天然染料由于其抗菌性能,是纺织工业中替代化学品的创新自然资源。本研究旨在揭示犬蔷薇叶提取物在羊毛织物染色中的颜色、牢度和抗菌效果。在预媒染工艺中,首选硫酸铝钾(KAl(SO<sub>4</sub>)<sub>2</sub>·12H<sub>2</sub>O)、硫酸铜(CuSO<sub>4</sub>·5H<sub>2</sub>O)和硫酸铁(FeSO<sub>4</sub>·7H<sub>2</sub>O)金属盐。在媒染和非媒染羊毛织物中测试了两种不同的天然染料浓度(25%和50%)。牢度值在中等和良好等级之间,为2-6。其中一个显著的结果是,未经媒染剂染色的样品的牢度值为5。在圆盘扩散分析中,发现超声波水浴CH<sub>3</sub>OH提取物对金黄色葡萄球菌ATCC 25923和大肠杆菌ATCC 25922更有效,分别为16.4mm和7.51mm。此外,根据AATCC 100方法,染色羊毛织物对两种不同的细菌具有显著的抗菌活性(还原率:w46e100)。

## KEYWORDS

*Rosa canina*; AATCC 100; wool fabric; natural dyeing; antibacterial activity; secondary metabolites

## 关键词

关键词; 犬蔷薇; 羊毛织物; 天然染色; 抗菌活性; 次生代谢物

## Introduction

Natural dyes, derived from plants (seed, root, stem, barks, leaves, and flowers), insects (lac and cochineal), and minerals (mineral ores, red clay, and ball clay) have been used to color products in many areas such as food, textiles, etc., from yesterday to today (Dayioglu et al. 2016; Kilinc et al. 2015a; Kilinc et al. 2015b; Shukla and Vankar 2013). Especially in recent years, the use of natural dyes has been increasing day by day, instead of synthetic dyestuffs that can show toxic and allergic properties in order to protect nature and human health (Aggarwal 2021; Geelani et al. 2017; Gupta 2019; Kamel, Abdelghaffar, and El-Zawahry 2011). In addition, the use of natural dyes is also important in terms of sustainability. Although natural dyes have environmentally friendly and renewable properties, most of these dyes are weakly bound to textile fiber, and consequently, low fastness values are obtained (Gupta 2019). Due to this situation, mordant substances, some of which are not environmentally friendly, are

used during applications. In addition to this disadvantage, the fact that natural dyes cannot be made with a standard recipe and the application of mostly natural dyes to natural fibers are among the problems that need to be solved in textile dyeing made with natural dyes (Gupta 2019). Despite these negative aspects, they can give antibacterial, antifungal, UV protective, deodorizing, and flame retardant properties to textile products (Kilinc et al. 2015a; Mishra and Gautam 2020). People come into contact with microorganisms by the contact of the clothes they wear during the day with living and non-living beings (Sanders, Grunden, and Dunn 2021). Some of these microorganisms are beneficial, while others are harmful to human health. Bacteria divided into two groups as Gram negative (*Escherichia coli*, *Klebsiella pneumonia* and etc.) and Gram positive (*Staphylococcus aureus*, *Streptococcus epidermidis* and etc.) according to the chemical and physical properties of their cell walls and these are one of the most important microorganism groups. Bacteria on the fabric surface can have a negative effect on both human health and the structural properties of the clothing worn (Pargai, Jahan, and Gahlot 2020). From this perspective, giving antibacterial properties to textile surfaces creates an advantage in terms of both human health and protection of the properties of textile products. It has been stated in the relevant literature that some natural dyes can provide this desired feature. Examples of these plants are *Punica granatum* L., *Lawsonia inermis* L., *Melia azedarach* L., *Rubia tinctorum* L., *Rumex maritimus* L., *Quercus infectoria* Olivier, *Paeonia officinalis* L., and *Syzygium aromaticum* L. (Kamboj, Jose, and Singh 2021; Samanta 2020). Rosehip is one of the important sources of natural dyes, especially the leaves of which are not economically evaluated today.

*Rosa canina* L. belonging to the genus of the *Rosa* in the Asteraceae family, called “dog rose”, is an highly medicinal, ornamental, deciduous shrub perennial plant species globally distributed in Europe, West Asia, Middle East, North Africa and North America. The *Rosa* genus, all parts are valuable, is represented by 200 species all around the world (Wissemann 2003a). It has been reported that this number increases up to 250 species based on different taxonomic evaluations (Kalkman 2004; Simulders et al. 2011). In Turkey, 25 different species were reported of this genus (Kultur 2004; Nilsson 1972). This species, culturally and economically existing in human life, are covered with pharmacologically valuable phytochemicals, such as tocotrienols, tannins, catechins, anthocyanins, carotenoids, organic acids, ascorbic acid, fatty acids, and polyphenolics (Ercisli 2007; Ouerghemmi et al. 2016; Rein, Kharazmi, and Winther 2004). All these bioactive components have given the species antiulcerogenic, antimutagenic, antioxidant, neuroprotective, diuretic, antiallergic, and antiinflammatory as well as especially antibacterial and natural dyestuff properties for dyeing of different fabrics such as cotton, polyester, wool, and silk (Cuce et al. 2020; Mármol et al. 2017; Milenkovic-Andjelkovic et al. 2016; Turan et al. 2018). Because of all these pharmacological effects, the fruit parts of this plant are preferred, while the leaf parts cannot be evaluated much economically. Studies in the literature show that there is no data on the evaluation of the leaves of this valuable plant, which has antibacterial properties, as a natural dye. This study is the first report on revealing the natural dye properties of the leaves of this plant, which is not economically evaluated.

## Materials and methods

### Materials

#### Fabric

Raw wool (100%) fabrics obtained from Yünsa (Turkey) company were used in the study. Fabric analysis results made in accordance with ASTM-20, ASTM 3776, ASTM 3775, and ASTM 1095 standards are given in Table 1.

#### Plant material

The leaf segments of the *R. canina* were taken from the natural wild populations of Şebinkarahisar (40° 17' 00" N, 38° 23' 21" E; 1424 m), Giresun, Turkey, between May and July 2019 and 2020. Fresh leaf samples were transported to laboratory conditions and weighed to determine the wet and dry weight

**Table 1.** The properties of the wool fabric.

Properties	100% Cotton	Standard
Fabric construction	Plain weave 1/1	ASTM 20
GSM (g/m <sup>2</sup> )	150	ASTM 3776
Ends per inch	37	ASTM 3775
Picks per inch	31	ASTM 3775
Warp count (Nm)	44	ASTM 1095
Weft count (Nm)	44	ASTM 1095

ratio with an analytical balance (Radwag AS 220, Poland). The leaf parts were put in a well-ventilated place without direct sunlight to dry. This drying technique avoids active phytochemical components from being negatively affected during the drying phase. The amounts of dry weight obtained per fresh weight of samples were calculated when samples completely dry and kept at 4°C until extraction processes were performed.

## Method

### Extraction

Air-dried leaf segments of the *R. canina* were shered into a fine powder at room temperature with the aid of a grinder (Arçelik K 8540). Since factors such as temperature and solvent affect the biological properties of phytochemicals in plants, two different methods and three different solvents were preferred in extraction studies. A 30-gram wholly ground leaf sample and cellulosic soxhlet extraction cartridges were preferred for the soxhlet extraction (FilterLab). After the mouth of the cartridge was covered with glass cotton, it was carefully placed in the device. Depending on the size of the soxhlet device, 150 mL of methanol (CH<sub>3</sub>OH)-water mixture (80:20, v/v) was added in apparatus, and it was extracted for 6 h at 60°C. Again, the same amounts of ground leaf samples were individually placed directly into two different volumetric flasks. In the above mentioned volume and ratio, CH<sub>3</sub>OH and distilled water individually were added to these samples and extracted in the ultrasonic water bath at 35°C for 12 h. Two different ultrasonic water bath extractions were filtered with Whatman No. 2, and the final stage CH<sub>3</sub>OH was thoroughly evaporated in a rotary evaporator at less than 35°C under reduced pressure (Heidolph Instruments GmbH & Co. KG, Germany). The lyophilization procedure was performed for the aqueous extracts using a freeze-dryer lyophilizator (Christ, Alpha 1-2LD plus, Germany). After determining the efficiency of extraction methods, all extracts were preserved in the refrigerator at +4°C until being analyzed and treatment to wool fabrics.

### Mordanting processes

Aluminum potassium sulfate (KAl (SO<sub>4</sub>)<sub>2</sub>.12H<sub>2</sub>O), copper sulfate (CuSO<sub>4</sub>.5H<sub>2</sub>O), and iron sulfate (FeSO<sub>4</sub>.7H<sub>2</sub>O) metal salts (Sigma-Aldrich) were used in the mordant process before the dyeing process. Since it is stated in the relevant literature that the dyeing process after pre-mordanting gives better results, pre-mordanting application was preferred in this study. The amount of mordant used during the application was adjusted according to the quantities used in traditional Turkish textile natural dyeing. Besides, the quantities are determined according to the fabric weight. Data on the mordanting are given in Table 2.

### Dyeing process

The dyestuffs obtained from soxhlet extraction were dissolved in 10 ml test tubes containing sufficient amount of DMSO. Vortex (Heidolph) process was applied to test tubes to accelerate the dissolution process. 25% and 50% dyestuff concentrations, adjusted according to the fabric weight, were used in the dyeing process. Specimens were dyed in liquor ratio of 1:50 at the boiling temperature for 30 min. The dyeing process was performed according to the study of Cuce et al. (2020). After the dyeing process, the washing process, with cold water, hot (boiling point) water, and finally cold water was carried out to remove the non-bonding dyestuffs on the fabric surface.

**Table 2.** Mordanting recipe with three different mordants on wool fabric dye.

Mordanting	Mordants		
	KAl(SO <sub>4</sub> ) <sub>2</sub> ·12H <sub>2</sub> O	FeSO <sub>4</sub> ·7H <sub>2</sub> O	CuSO <sub>4</sub> ·5H <sub>2</sub> O
% Mordant	20	3	5
Temperature (°C)	100	100	100
Time (min)	60	60	60

KAl(SO<sub>4</sub>)<sub>2</sub>·12H<sub>2</sub>O = Potassium aluminum sulfate, FeSO<sub>4</sub>·7H<sub>2</sub>O = Iron sulfate, CuSO<sub>4</sub>·5H<sub>2</sub>O = Copper sulfate

### Color measurement

Color measurements were made with a Konica Minolta CM-3600d spectrophotometer. CIELAB (L\* a\* b\*) values have been taken in accordance with D65 daylight and 10° standard. Color strength values of the samples were calculated by using the reflectance value at the maximum absorbance (λ<sub>max</sub>) at visible wavelength (360–700 nm) with Kubelka–Munk equation (Equation 1) for each dyeing.

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

where:

K- absorption coefficient of the sample, S- scattering coefficient of the sample, and R- reflectance value of the sample at maximum absorbance

In addition to color strength measurement, color coordinate values (L\*: lightness (0 = black, 100 = white), L = lightness; a\* ± = red/green; b\* ± = yellow/blue, C\* to vividness-dulness (100 = vivid, 0 = dull)) were gauged.

### Fastness measurement

Light, washing, and rubbing fastness tests were carried out on the dyed samples. Fastness tests were performed and evaluated according to ISO 105-C06 (A1S), ISO 105-B02 and ISO 105-X12 standards.

### Activity test

#### Microbial strains

Generally preferred in natural dyeing study gram-positive *Staphylococcus aureus* ATCC 25923 and gram-negative *Escherichia coli* ATCC 25922 strains were used for the determination of the antimicrobial activities of the three different extracts obtained from two different methods.

#### Disc diffusion

The disc diffusion test was conducted by following the protocols of The Clinical and Laboratory Standards Institute (2013) and The European Committee on Antimicrobial Susceptibility Testing (2014). Initially, the well-dried plant extract was dissolved in 20% (v/v) dimethyl sulfoxide (DMSO) to a final concentration of 100 mg/mL and sterilized with 0.45 μm Millipore filter (Merck, Germany). Antimicrobial tests were then carried out by the disc diffusion method using 100 μL of suspension containing 1 × 10<sup>8</sup> CFU/mL bacteria, spread on Muller Hinton Agar (MHA) medium. 0.5 mg/L extract (500 μg per disc) was impregnated on discs with a diameter of 6 mm and placed on the inoculated MHA medium. DMSO impregnated discs were preferred as a negative control. Ofloxacin (10 μg per disc), sulbactam (30 μg) + cefoperazone (75 μg); (105 μg per disc) and/or netilmicin (30 μg per disc) were tested as positive reference standards to determine the sensitivity of each strain tested. The inoculated Petri dishes were first kept in sterile microbiological cabinet nearly 1 h for the diffusion of the extract in agar and then incubated at 37°C for 16 h. The inhibition zone was measured for antibacterial activity against the test organisms at the end of the incubation period. Each experiment was repeated triplicates.

### Antibacterial activity of wool fabric

Antibacterial tests of the raw, dyed, and mordanted samples were carried out in accordance with the AATCC 100–2004 standard. As stated above, *S. aureus* and *E. coli* strains were used as test microorganisms. Bacterial colonies formed on the MHA medium were counted with the help of the microscope. The reduction in the number of bacteria was calculated using the following equation (Eqn 2);

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

Where:

R- % reduction, B- the number of bacteria colonies at the beginning of the test (0 hours), A- the number of bacteria colonies after 24 h contact of dyed wool fabrics.

## Results and discussion

### Colorimetric measurements





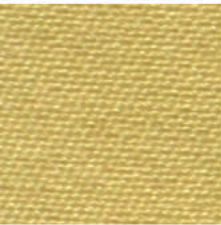
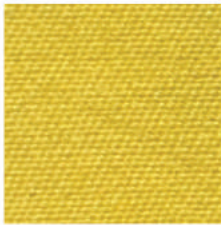
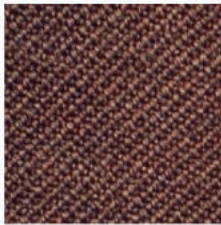
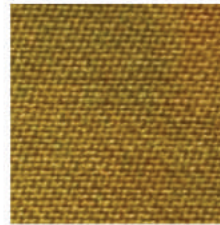
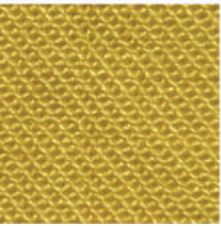
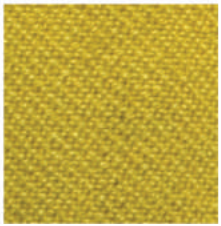

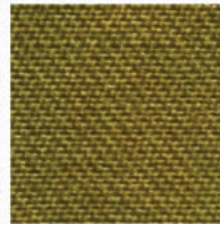
In this part of the study, the colorimetric data obtained after dyeing wool fabric with the leaf of *R. canina* plant was examined. Aluminum potassium sulfate ( $KAl(SO_4)_2 \cdot 12H_2O$ ), copper sulfate ( $CuSO_4 \cdot 5H_2O$ ), and iron sulfate ( $FeSO_4 \cdot 7H_2O$ ) metal salts were used in the study, as they are mostly preferred mordant types in traditional Turkish textile natural dyeing. Different colors and colorimetric data were obtained as a result of the dyeing process with and without mordant with two different concentrations of the same extract (25% and 50%). It can be seen from the color data that the most vivid colors are in samples that have been treated with aluminum potassium sulfate before dyeing. According to the  $a^*$  ( $\pm$  = red/green) value, the fabric that is mordanted with iron sulfate and dyed with 50% concentration has the reddest nuance, while the fabric mordanted with copper sulfate and dyed with 50% concentration has the greenest color. When Table 3 is viewed in terms of yellow and blue nuance ( $b^*$ ), it is seen that the sample mordant with aluminum potassium sulfate has the highest yellow nuance. Related studies in the literature stated that iron sulfate gives darker and redder color nuances and aluminum sulfate gives more yellow nuances compared to other samples (Janani and Lukyambuzi 2013; Mulec and Gorjanc 2015). This supports the results obtained from the study. In addition, samples treated with iron sulfate have the highest blue nuance (Figure 1).

When the samples were examined according to the obtained K/S values, it was observed that the K/S value increased with the increase in the dye concentration. This situation was also stated in previous studies with natural dyes. Bukhari et al. (2017) stated that in natural dyeing studies with walnut, the increase in dye concentration causes an increase in dye transfer and a higher color depth, thus an increase in color strength. In addition, it is seen that the value of the sample pre-mordant with iron

**Table 3.** Colorimetric data of the dyed wool fabrics.

Samples	L*	a*	b*	C*	h°	$\lambda$	K/S
Standard	86.8711	−0.7351	10.5759	10.6014	93.9762	360	0.9339
20% $KAl(SO_4)_2 \cdot 12H_2O$	88.4353	−0.9201	11.8238	11.8595	94.4496	360	0.9169
3% $FeSO_4 \cdot 7H_2O$	69.6711	3.5612	18.6975	19.0336	79.2164	360	3.7134
5% $CuSO_4 \cdot 5H_2O$	67.5278	−5.8023	17.6197	18.5505	108.2270	360	4.1821
25% natural dyestuff	78.5700	1.0600	14.7200	14.7500	85.8900	360	1.0969
50% natural dyestuff	74.2100	1.2600	18.1700	18.2100	86.0400	360	2.1526
20% $KAl(SO_4)_2 \cdot 12H_2O$ + 25% natural dyestuff	72.6702	−1.7134	42.6094	42.6438	92.3027	360	9.2285
20% $KAl(SO_4)_2 \cdot 12H_2O$ + 50% natural dyestuff	67.0814	−3.1137	41.5756	41.6921	94.2830	360	13.6369
3% $FeSO_4 \cdot 7H_2O$ + 25% natural dyestuff	38.2579	3.3848	9.1872	9.7909	69.7748	360	15.0927
3% $FeSO_4 \cdot 7H_2O$ + 50% natural dyestuff	34.3337	5.4918	9.7624	11.2011	60.6400	360	23.2821
5% $CuSO_4 \cdot 5H_2O$ + 25% natural dyestuff	51.2483	−2.0131	27.1081	27.1827	94.2471	360	14.7887
5% $CuSO_4 \cdot 5H_2O$ + 50% natural dyestuff	49.0256	−3.8555	25.9509	26.2357	98.4505	360	17.4637

$KAl(SO_4)_2 \cdot 12H_2O$  = Aluminum potassium sulfate,  $FeSO_4 \cdot 7H_2O$  = Iron sulfate,  $CuSO_4 \cdot 5H_2O$  = Copper sulfate, L\*: lightness (0 = black, 100 = white), L = lightness;  $a^* \pm$  = red/green;  $b^* \pm$  = yellow/blue,  $C^*$  = to vividness-dullness (100 = vivid, 0 = dull)

Raw, Mordanted and Dyed Samples:				
		Mordanted Type		
Concent.	Without Mordanted	KAl (SO <sub>4</sub> ) <sub>2</sub> ·12H <sub>2</sub> O (20%)	FeSO <sub>4</sub> ·7H <sub>2</sub> O (3%)	CuSO <sub>4</sub> ·5H <sub>2</sub> O (5%)
Raw Wool Fabric				
25% Dyed Wool Fabric				
50% Dyed Wool Fabric				

**Figure 1.** Mordanted and unmordanted raw and dyed wool fabric samples with methanol extracts of *R. canina* leaf. (KAl (SO<sub>4</sub>)<sub>2</sub>·12H<sub>2</sub>O = Aluminum potassium sulfate, FeSO<sub>4</sub>·7H<sub>2</sub>O = Iron sulfate, CuSO<sub>4</sub>·5H<sub>2</sub>O = Copper sulfate).

sulfate and dyed with 50% dye concentration is higher than the others. It is known that this occurs after the strong interaction of iron sulfate and wool fibers (Bhattacharya and Shah 2000). When the K/S values of the mordanted and un-mordanted samples were compared, the mordanting process increased the interaction between the functional groups in the fabric and the functional groups in the dye, thereby increasing the dye uptake and therefore the color strength of the fixed samples increased (Rather et al. 2016).

### **Antibacterial activity**

#### **Disc diffusion**

In studies examining biological activities, the yield obtained from plants is a crucial parameter. In this context, a dry weight of the *R. canina* leaf per fresh weight was calculated with 46.67% yielding after the drying process. In the extraction method, the maximum efficiency was obtained from the extraction with the soxhlet apparatus with 29.35%. This value is higher than the result obtained by Ouerghemmi et al. (2016). A similar result was obtained from the decoction extraction of *R. canina* leaves with a yield of 24.81% (Deliorman Orhan et al. 2012). Yielding performance may differ according to the extraction method, extraction time, the habitat, the time of collecting the specimens,

the drying method, and the grinding amount, etc. These ratios were 18.59% and 16.45% for CH<sub>3</sub>OH and aqueous extracts, in the ultrasonic water bath, respectively. Similar results were reported in a study on *H. arenarium* subsp. *aucheri* (Cuce et al. 2020). These researchers have stated that soxhlet extraction had higher yielding than ultrasonic water bath extraction. Despite these results, it is known that the extraction method affects the disk diffusion results. Both of the obtained CH<sub>3</sub>OH extracts were found to be effective against *S. aureus*. CH<sub>3</sub>OH extracts from the ultrasonic water bath gave the higher disc diffusion result with 16.4 mm. This value was measured as 13.7 mm in CH<sub>3</sub>OH extracts from soxhlet apparatus. It was determined that among these two extracts only extracts obtained from in water bath were effective on *E. coli* with 7.51 mm (Figure 2).

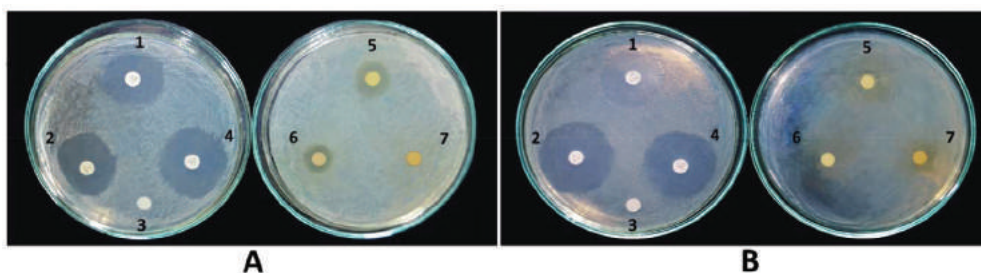
The antibacterial properties of *R. canina* leaf extracts obtained by different extraction techniques on these two bacterial strains have been studied by many researchers (Deliorman Orhan et al. 2012; Milenkovic-Andjelkovic et al. 2016; Polumackanycz et al. 2020). While it was determined that gram negative bacteria are more resistant than gram positives, an effective result could not be obtained in *E. coli* according to the extraction method and solvent properties in some of them of these studies. All these results are consistent with our results. Also aqueous extracts were not found to be effective in both of these bacterial strains. These results are an indication of why methanol extracts obtained from ultrasonic water bath extractions were used in dyeing studies. The disk diffusion results obtained by the above-mentioned researchers on methanol and water extracts and similar bacteria support the results of our study. Any activity of DMSO (maximum 1% v/v), preferred as negative control, could not be determined against these two bacterial strains. All of the disc diffusion results are given in Table 4.

#### Antibacterial properties of wool fabric

Generally, dyestuffs obtained from traditional plants are used to color textile products. However, if these plants contain anthraquinone, flavonoid, tannin, and naphthoquinone compounds in their ingredients, they can add antibacterial properties to textile products (Cuce et al. 2020). Dyestuffs obtained from plants with antibacterial properties prevent the growth of microorganisms and thus give antibacterial properties to the textile surfaces (Yusuf, Shabbir, and Mohammad 2017).

Antibacterial test results of the samples are shown in Table 5. When the undyed mordanted samples are examined, it is seen that aluminum potassium sulfate and copper sulfate are 100% effective against the relevant bacteria. However, samples mordanted with ferrous sulfate showed less potency against *E. coli* and *S. aureus* than others. These are 46% and 53.07%, respectively (Figures 3 and 4).

Ghaheh et al. (2014) had stated that samples mordanted with iron sulfate provide less antibacterial effect than aluminum potassium sulfate and copper sulfate. This result supports the data obtained. 100% effect against *E. coli* was observed in samples dyed without mordanting. Concentration increase in the same samples increased the effect against *S. aureus* from 80% to 83.72%. It has been stated in the studies in the literature that the antibacterial activity will increase with the increase in concentration (Cuce et al. 2020; Mirjalili and Karimi 2013). Having antibacterial properties of the samples as a result



**Figure 2.** Disc diffusion assay of *R. canina*, (A) *Staphylococcus aureus* ATCC 25923, (B) *Escherichia coli* ATCC 25922, (1) Netilmicin, (2) Ofloxacin, (3) DMSO, (4) Sulbactam + Cefeprozone, (5) Methanol extract in ultrasonic water bath, (6) Methanol extract in soxhlet, (7) Aqueous extract in ultrasonic water bath.

**Table 4.** Disc diffusion activity of *Rosa canina* leaf extracts.

Bacteria	Plant Extracts			Negative Control	Positive Controls		
	MeOH (Ultrasonic Bath)	MeOH (Soxhlet)	Aqueous (Ultrasonic Bath)	DMSO	Netilmicin	Ofloxacin	Sulbactam + Cefoperazone
<i>Staphylococcus aureus</i>	16.4 ± 0.50*	13.7 ± 0.22	-	-	23.64 ± 0.3	27.02 ± 0.29	28.45 ± 0.47
<i>Escherichia coli</i>	7.51 ± 0.31	-	-	-	23.87 ± 0.45	27.44 ± 0.34	28.94 ± 0.57

MeOH = Methanol, DMSO = Dimethylsulfoxide

-: not detected

\* Values expressed are mean ± standard deviation of three experiments

**Table 5.** Antibacterial activities of *Rosa canina* dyed wool fabrics with AATCC 100.

Sample	Bacteria	Antibacterial Activity (% R)
Standard	<i>E. coli</i>	0
	<i>S. aureus</i>	0
20% KAl(SO <sub>4</sub> ) <sub>2</sub> .12H <sub>2</sub> O	<i>E. coli</i>	100
	<i>S. aureus</i>	100
3% FeSO <sub>4</sub> .7H <sub>2</sub> O	<i>E. coli</i>	46
	<i>S. aureus</i>	53.07
5% CuSO <sub>4</sub> .5H <sub>2</sub> O	<i>E. coli</i>	100
	<i>S. aureus</i>	100
25% natural dyestuff	<i>E. coli</i>	100
	<i>S. aureus</i>	80
50% natural dyestuff	<i>E. coli</i>	100
	<i>S. aureus</i>	83.72
20% KAl(SO <sub>4</sub> ) <sub>2</sub> .12H <sub>2</sub> O + 25% natural dyestuff	<i>E. coli</i>	100
	<i>S. aureus</i>	100
20% KAl(SO <sub>4</sub> ) <sub>2</sub> .12H <sub>2</sub> O + 50% natural dyestuff	<i>E. coli</i>	100
	<i>S. aureus</i>	100
3% FeSO <sub>4</sub> .7H <sub>2</sub> O + 25% natural dyestuff	<i>E. coli</i>	100
	<i>S. aureus</i>	99.15
3% FeSO <sub>4</sub> .7H <sub>2</sub> O + 50% natural dyestuff	<i>E. coli</i>	100
	<i>S. aureus</i>	100
5% CuSO <sub>4</sub> .5H <sub>2</sub> O + 25% natural dyestuff	<i>E. coli</i>	100
	<i>S. aureus</i>	100
5% CuSO <sub>4</sub> .5H <sub>2</sub> O + 50% natural dyestuff	<i>E. coli</i>	100
	<i>S. aureus</i>	100

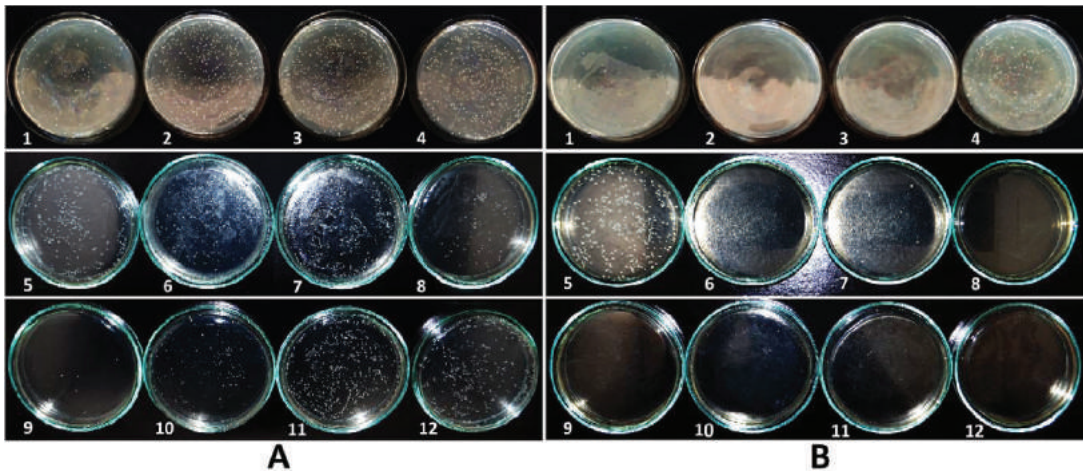
KAl(SO<sub>4</sub>)<sub>2</sub>.12H<sub>2</sub>O = Aluminum potassium sulfate, FeSO<sub>4</sub>.7H<sub>2</sub>O = Iron sulfate, CuSO<sub>4</sub>.5H<sub>2</sub>O = Copper sulfate,  
% R = % Reduction

of the dyeing process performed without mordant is an ecologically important result. It is clearly seen from the samples, mordanted with iron sulfate, that the dyestuff and mordant materials increase the antibacterial effect in harmony. While the effect of undyed samples, mordanted with iron sulfate, against bacteria was around 50%, it was observed that this effect took a value close to 100% after the same samples were dyed. Apart from this information, while no antibacterial effect was observed in the standard fabric, nearly 100% antibacterial effect was observed in the other samples (mordanted and dyed samples).

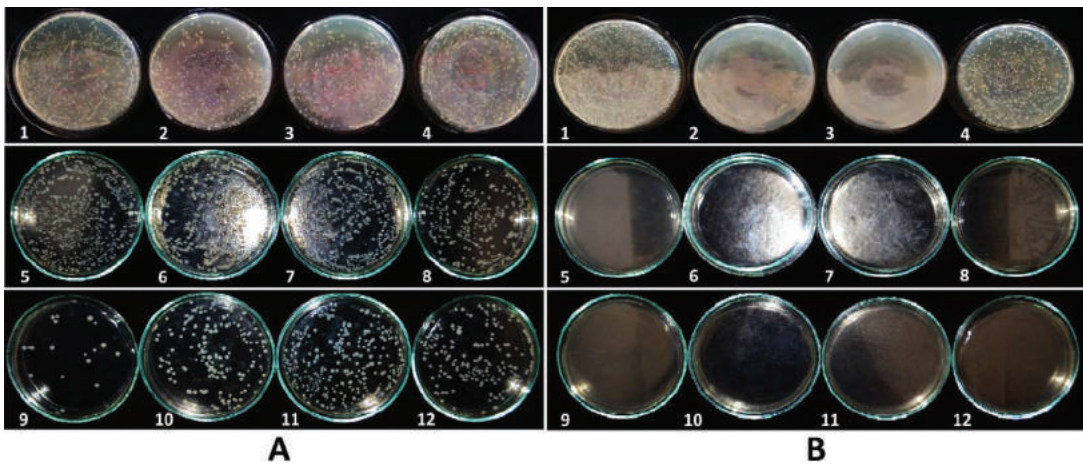
### Fastness properties

The data obtained as a result of the fastness tests are presented in Table 6. According to the results, it is seen that the color changes occurring after washing in the samples are between moderate and good levels. It was determined that the color changes of the samples dyed without mordanting were less than the other samples. In addition to these, the increase in dyeing concentration improved the color change values by 1 degree. Among the samples, the lowest light fastness was observed in samples that were mordanted with aluminum potassium sulfate. Also, the samples with the highest value are the





**Figure 3.** AATCC100 assay of wool fabric natural dyeing with *R. canina* on *Staphylococcus aureus* ATCC 25923, (A) 0. Contact time (B) 24. Contact time, (1) Standard, (2)  $\text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ , (3)  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ , (4)  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ , (5) 25% natural dyestuff, (6)  $\text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$  + 25% natural dyestuff, (7)  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  + 25% natural dyestuff, (8)  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$  + 25% natural dyestuff, (9) 50% natural dyestuff, (10)  $\text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$  + 50% natural dyestuff, (11)  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  + 50% natural dyestuff, (12)  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$  + 50% natural dyestuff.



**Figure 4.** AATCC100 assay of wool fabric natural dyeing with *R. canina* on *Escherichia coli* ATCC 25922, (A) 0. Contact time (B) 24. Contact time, (1) Standard, (2)  $\text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ , (3)  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ , (4)  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ , (5) 25% natural dyestuff, (6)  $\text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$  + 25% natural dyestuff, (7)  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  + 25% natural dyestuff, (8)  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$  + 25% natural dyestuff, (9) 50% natural dyestuff, (10)  $\text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$  + 50% natural dyestuff, (11)  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  + 50% natural dyestuff, (12)  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$  + 50% natural dyestuff.

samples mordant with ferrous sulfate. When the light fastness values were compared with the data in the literature, it was observed that the results were parallel to each other (Avinash, Paul, and Bechtold 2016; Cuce et al. 2020). In addition to this, in related studies, it has been stated that iron sulfate can bind more dyestuff molecules to the fiber due to its higher affinity for dye and fiber compared to alum. Correspondingly, specimens treated with ferrous sulfate have deeper shades and may appear less faded after exposure to light. Considering the samples treated with alum in the same frame, it can be said that they may be bound with less dyestuff, and in connection with this, they fade more when exposed to light (Loum, Byamukama, and Wanyama 2021; Priyanti et al. 2021; Uddin 2014). The change in dye concentration did not cause any change in light fastness values. While the rubbing fastness values of the samples dyed without mordant process are excellent, this value differs between poor and good in samples with mordant. Especially, poor wet rubbing fastness results were obtained from mordanted

**Table 6.** Fastness properties of the dyed wool fabrics.

Samples	Fastness to light	Rubbing fastness		Color fastness to washing						
		Dry	Wet	Color change	Staining					
					CA	Co	PE	PAC	Wo	PA
25% natural dyestuff	5	5	5	4	5	4-5	5	5	4-5	5
50% natural dyestuff	5	5	5	4-5	5	4-5	5	5	4-5	5
20% KAl(SO <sub>4</sub> ) <sub>2</sub> .12H <sub>2</sub> O + 25% natural dyestuff	2-3	4	3-4	3	5	4-5	5	5	4-5	5
20% KAl(SO <sub>4</sub> ) <sub>2</sub> .12H <sub>2</sub> O + 50% natural dyestuff	2-3	3-4	3	3-4	5	4-5	5	5	4-5	5
3% FeSO <sub>4</sub> .7H <sub>2</sub> O + 25% natural dyestuff	6	3-4	3	3-4	5	5	5	5	5	5
3% FeSO <sub>4</sub> .7H <sub>2</sub> O + 50% natural dyestuff	6	3	2-3	4	5	5	5	5	5	5
5% CuSO <sub>4</sub> .5H <sub>2</sub> O + 25% natural dyestuff	4-5	4-5	3	3-4	5	5	5	5	5	5
5% CuSO <sub>4</sub> .5H <sub>2</sub> O + 50% natural dyestuff	4-5	4	2-3	4	5	5	5	5	5	5

KAl(SO<sub>4</sub>)<sub>2</sub>.12H<sub>2</sub>O = Aluminum potassium sulfate, FeSO<sub>4</sub>.7H<sub>2</sub>O = Iron sulfate, CuSO<sub>4</sub>.5H<sub>2</sub>O = Copper sulfate, CA = Cellulose diacetate, Co = Cotton, PE = Polyester, PAC = Polyacrylic, Wo = Wool, PA = Polyamide.

samples. When the multi-fiber fabric is examined after the washing fastness process, it is seen that the staining is on the cotton and wool parts; however, the staining seen in these parts is very small. In general, excellent values have been obtained after washing fastness.

## Conclusion

At the end of the experiments, methanol extracts of *R. canina* leaves have been found a crucial dyeing and antibacterial potentials. Mordanted and unmordanted sample results were acceptable levels. Depending on the antibacterial properties of the used herbal sample, the antibacterial levels of the fabrics after dyeing are also at a significant level. The highest antibacterial reduction rate on wool fabrics were generally observed at mordanted samples with aluminum potassium sulfate and copper sulfate. The color strength and fastness values of the wool fabrics dyed with natural dyestuff without being exposed to mordant pre-treatment were remarkably better than other applications. All these impressive results are important evidence that rosehip leaves, which are not economically evaluated, can be utilized in the textile industry.

## Disclosure statement

No potential conflict of interest was reported by the author(s).

## References

- Aggarwal, S. 2021. Indian dye yielding plants: Efforts and opportunities. *Natural Resources Forum* 45 (1):63–86. doi:10.1111/1477-8947.12214.
- Avinash, M. P., R. Paul, and T. Bechtold. 2016. Metal mordanting in dyeing with natural colourants. *Coloration Technology* 132 (2):107–13. doi:10.1111/cote.12199.
- Bhattacharya, S. D., and A. K. Shah. 2000. Metal ion effect on dyeing of wool fabric with catechu. *Coloration Technology* 116 (1):10–12. doi:10.1111/j.1478-4408.2000.tb00002.x.
- Bukhari, M. N., M. Shabbir, L. J. Rather, M. Shahid, U. Singh, M. A. Khan, and F. Mohammad. 2017. Dyeing studies and fastness properties of brown naphthoquinone colorant extracted from *Juglans regia* L. on natural protein fiber using different metal salt mordants. *Textiles and Clothing Sustainability* 3 (1):1–9. doi:10.1186/s40689-016-0025-2.
- Clinical and Laboratory Standards Institute. 2013. *Performance standards for antimicrobial susceptibility testing; twenty-third information supplement*. CLSI document M100-S23. Wayne PA: Clinical and Laboratory Standards Institute.
- Cuce, M., M. Kilinc, N. Kilinc, A. Tekin, and D. Kut. 2020. Color, fastness, and antimicrobial Properties of wool fabrics dyed with *Helichr ysum arenarium* subsp. *aucheri* extract. *Journal of Natural Fibers* 1–13. doi:10.1080/15440478.2020.1848706.
- Dayioglu, H., N. Merdan, S. Eyupoglu, M. Kilinc, and D. Kut. 2016. The effect of plasma treatment on the dyeability of silk fabric by using *Phytolacca decandra* L. natural dye extract. *Textile and Apparel* 26 (3):262–69.

- Deliorman Orhan, D., B. Özçelik, S. Hoşbaş, and M. Vural. 2012. Assessment of antioxidant, antibacterial, antimycobacterial, and antifungal activities of some plants used as folk remedies in Turkey against dermatophytes and yeast-like fungi. *Turkish Journal of Biology* 36 (6):672–86. doi:10.3906/biy-1203-33.
- Ercisli, S. 2007. Chemical composition of fruits in some rose (*Rosa* spp.) species. *Food Chemistry* 104 (4):1379–84. doi:10.1016/j.foodchem.2007.01.053.
- European Committee on Antimicrobial Susceptibility Testing. 2014. EUCAST disk diffusion test manual, version 4.0. Retrieved 23 September 2020 from [http://www.eucast.org/ast\\_of\\_bacteria/disk\\_diffusion\\_methodology/](http://www.eucast.org/ast_of_bacteria/disk_diffusion_methodology/).
- Geelani, S. M., S. Ara, N. A. Mir, S. J. A. Bhat, and P. K. Mishra. 2017. Dyeing and fastness properties of *quercus robur* with natural mordants on natural fibre. *Textiles and Clothing Sustainability* 2 (1):1–10. doi:10.1186/s40689-016-0019-0.
- Ghaheh, F. S., S. M. Mortazavi, F. Alihosseini, A. Fasshi, A. S. Nateri, and D. Abedi. 2014. Assessment of antibacterial activity of wool fabrics dyed with natural dyes. *Journal of Cleaner Production* 72:139–45. doi:10.1016/j.jclepro.2014.02.050.
- Gupta, V. K. 2019. Fundamentals of natural dyes and its application on textile substrates. In *Chemistry and technology of natural and synthetic dyes and pigments*, eds. A. K. Samanta and N. Awwad, 43–74. London: IntechOpen. doi:10.5772/intechopen.89964.
- Janani, L., and H. Lukyambuzi. 2013. Effects of mordanting methods of dye from *Vernonia amygdalina* on cotton fabrics coloration. *Journal of Language, Technology & Entrepreneurship in Africa* 4 (2):17–27.
- Kalkman, C. 2004. Rosaceae. In *The families and genera of vascular plants. VI flowering plants-dicotyledons*, ed. K. Kubitzki, 386–443. Berlin, Heidelberg: Springer. doi:10.1007/978-3-662-07257-8\_39.
- Kamboj, A., S. Jose, and A. Singh. 2021. Antimicrobial activity of natural dyes—a comprehensive review. *Journal of Natural Fibers* 1–15. doi:10.1080/15440478.2021.1875378.
- Kamel, M. M., F. Abdelghaffar, and M. M. El-Zawahry. 2011. Eco-friendly dyeing of wool with a mixture of natural dyes. *Journal of Natural Fibers* 8 (4):289–307. doi:10.1080/15440478.2011.627114.
- Kilinc, M., S. Canbolat, N. Merdan, H. Dayioglu, and F. Akin. 2015a. Investigation of the color, fastness and antimicrobial properties of wool fabrics dyed with the natural dye extracted from the cone of *Chamaecyparis lawsoniana*. *Procedia-Social and Behavioral Sciences* 195:2152–59. doi:10.1016/j.sbspro.2015.06.281.
- Kilinc, M., D. Koçak, S. Canbolat, H. Dayioglu, N. Merdan, and F. Akin. 2015b. Investigation of the effect of the dyeing method on the dyeing properties of wool fabrics dyed with natural dyes extracted from *Vaccinium corymbosum* L. *Marmara Journal of Pure and Applied Sciences* 27 (3):78–82. doi:10.7240/mufbed.37837.
- Kultur, S. 2004. A new *Rosa* species for European flora. *Journal of Faculty of Pharmacy Istanbul* 37:75–83.
- Loum, J., R. Byamukama, and P. A. G. Wanyama. 2021. Application of natural dyes from selected indigenous plants on cotton and silk fabrics. *Journal of Textile Engineering & Fashion Technology* 7 (2):71–77. doi:10.15406/jteft.2021.07.00270.
- Mármol, I., C. Sánchez-de-diego, N. Jiménez-Moreno, C. Ancín-Azpilicueta, and M. J. Rodríguez-Yoldi. 2017. Therapeutic applications of rose hips from different *Rosa* species. *International Journal of Molecular Sciences* 18 (6):1137. doi:10.3390/ijms18061137.
- Milenkovic-Andjelkovic, A. S., M. Z. Andjelkovic, A. N. Radovanovic, B. C. Radovanovic, and V. Randjelovic. 2016. Phenol composition, radical scavenging activity and antimicrobial activity of berry leaf extracts. *Bulgarian Chemical Communications* 48 (1):27–32.
- Mirjalili, M., and L. Karimi. 2013. Extraction and characterization of natural dye from green walnut shells and its use in dyeing polyamide: Focus on antibacterial properties. *Journal of Chemistry* 2013:9. doi:10.1155/2013/375352.
- Mishra, A., and S. Gautam. 2020. Application of natural dyes for herbal textiles. In *Chemistry and technology of natural and synthetic dyes and pigments*, eds. A. K. Samanta and N. Awwad, 123–50. London: IntechOpen. doi:10.5772/intechopen.93183.
- Mulec, I., and M. Gorjanc. 2015. The influence of mordanting on the dyeability of cotton dyed with turmeric extract. *Tekstilec* 58 (3):199–208. doi:10.14502/Tekstilec2015.58.199-208.
- Nilsson, O. 1972. *Rosa* L. In *Flora of Turkey and the East Aegean Islands*, ed. P. H. Davis, 106–28. Edinburgh: Edinburgh University Press.
- Ouergemmi, S., H. Sebei, L. Siracusa, G. Ruberto, A. Saija, F. Cimino, and M. Cristani. 2016. Comparative study of phenolic composition and antioxidant activity of leaf extracts from three wild *rosa* species grown in different tunisia regions: *Rosa canina* L., *rosa moschata* herrm. and *Rosa sempervirens* L. *Industrial Crops and Products* 94:167–77. doi:10.1016/j.indcrop.2016.08.019.
- Pargai, D., S. Jahan, and M. Gahlot. 2020. Functional properties of natural dyed textiles. In *Chemistry and technology of natural and synthetic dyes and pigments*, eds. A. K. Samanta and N. Awwad, 207–24. London: IntechOpen. doi:10.5772/intechopen.88933.
- Polumackanycz, M., M. Kaszuba, A. Konopacka, U. Marzec-Wróblewska, M. Wesolowski, K. Waleron, A. Buciński, and A. Viapiana. 2020. Phenolic composition and biological properties of wild and commercial dog rose fruits and leaves. *Molecules* 25 (22):5272. doi:10.3390/molecules25225272.
- Priyanti, R. A., A. Khairiah, M. Hilal, A. M. Putra, N. A. Andriansyah, A. D. Fransiska, M. M. Assyaidin, Z. Chairullisan, and R. Fauziah. 2021. The cotton fabric coloring with *leucaena leucocephala* peel at room temperature. *KOBI-2019 (EPIC Series in Biological Sciences* 1: 106–11.

- Rather, L. J., M. Azam, M. Shabbir, M. N. Bukhari, M. Shahid, M. A. Khan, and F. Mohammad. 2016. Antimicrobial and fluorescence finishing of woolen yarn with *Terminalia arjuna* natural dye as an ecofriendly substitute to synthetic antibacterial agents. *RSC Advances* 6 (45):39080–94. doi:10.1039/c6ra02717b.
- Rein, E., A. Kharazmi, and K. Winther. 2004. A herbal remedy, hymen vital (stand. powder of a subspecies of *rosa canina* fruits) reduces pain and improves general wellbeing in patients with osteoarthritis –a double-blind, placebo-controlled, randomised trial. *Phytomedicine* 11 (5):383–91. doi:10.1016/j.phymed.2004.01.001.
- Samanta, P. 2020. A review on application of natural dyes on textile fabrics and its revival strategy. In *Chemistry and technology of natural and synthetic dyes and pigments*, eds. A. K. Samanta and N. Awwad, 75–98. London: IntechOpen. doi:10.5772/intechopen.90038.
- Sanders, D., A. Grunden, and R. R. Dunn. 2021. A review of clothing microbiology: The history of clothing and the role of microbes in textiles. *Biology Letters* 17 (1):20200700. doi:10.1098/rsbl.2020.0700.
- Shukla, D., and P. S. Vankar. 2013. Natural dyeing with black carrot: New source for newer shades on silk. *Journal of Natural Fibers* 10 (3):207–18. doi:10.1080/15440478.2012.757031.
- Simulders, M. J. M., P. Arens, C. F. S. Koning-Boucoiran, V. W. Gitonga, F. A. Krens, A. Atanassov, I. Atanassov, K. E. Rusanov, M. Bendahmane, A. Dubois, et al. 2011. Rosa. In *Wild crop relatives: Genomic and breeding resources*, ed. C. Kole, 243–75. Berlin, Heidelberg: Springer. doi:10.1007/978-3-642-21201-7\_12.
- Turan, I., S. Demir, K. Kilinc, S. O. Yaman, S. Misir, H. Kara, B. Genc, A. Mentese, Y. Aliyazicioglu, and O. Deger. 2018. Cytotoxic effect of *Rosa canina* extract on human colon cancer cells through repression of telomerase expression. *Journal of Pharmaceutical Analysis* 8 (6):394–99. doi:10.1016/j.jppha.2017.12.005.
- Uddin, M. G. 2014. Effects of different mordants on silk fabric dyed with onion outer skin extracts. *Journal of Textiles* 2014:1–8. doi:10.1155/2014/405626.
- Wissemann, V. 2003a. Conventional taxonomy of wild roses. In *Encyclopedia of rose sciences*, ed. A. Roberts, T. Debener, and S. Gudin, 111–17. London: Elsevier Science: Oxford.
- Yusuf, M., M. Shabbir, and F. Mohammad. 2017. Natural colorants: Historical, processing and sustainable prospects. *Natural Products and Bioprospecting* 7 (1):123–45. doi:10.1007/s13659-017-0119-9.