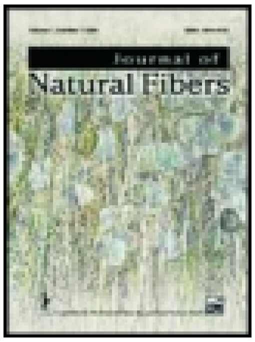

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Use of *Viburnum Opulus* L.*(Caprifoliaceae)* in Dyeing and Antibacterial Finishing of Cotton

Fazlıhan Yılmaz[a], Ö. Faruk Koçak[b], F. Betül Özgeriş[c], Handan Şapçı Selamoğlu[d], Cem Vural[e,g], Hüseyin Benli[f], and M. İbrahim Bahtiyari[a]

[a]Textile Engineering Department, Erciyes University, Kayseri, Turkey; [b]Erzurum Vocational School, Atatürk University, Erzurum, Turkey; [c]Faculty of Health Sciences, Atatürk University, Erzurum, Turkey; [d]Food Processing Department, Vocational School of Yahyalı, Kayseri University, Kayseri, Turkey; [e]Department of Biology, Faculty of Science, Erciyes University, Kayseri, Turkey; [f]Mustafa Çıkrıkçıoğlu Vocational School, Kayseri University, Kayseri, Turkey; [g]Genome and Stem Cell Center, Erciyes University, Kayseri, Turkey

ABSTRACT

It is known that use of chemicals can cause different problems to human health and environment. Therefore, studies to find alternative chemical free finishing processes in textile are now interesting topics. In the light of this, it was tried to minimize chemical uses for antibacterial finishing of cotton by using plant sources “cranberry fruit” and “cranberry fruit branch extract” in dyeing processes. The dyeing experiments were carried out at two different temperatures for different durations. Then, the samples were analyzed in terms of *K/S*, *CIE L*a*b*C** and *h*° values, light fastness, washing fastness, and antibacterial efficiencies. Consequently, it was found that the two natural dye sources could color the cotton-knitted fabrics, and in general, the washing fastness of dyed samples with both natural dye sources was good, but light fastness values were limited and in dyeing processes by using cranberry fruit branch extract it was poor. Moreover, it was seen that among the tested plantal sources, good antibacterial efficiency against the tested bacteria were obtained with the use of cranberry fruit juice in dyeing processes at low temperature and for lower durations.

摘要

众所周知，使用化学品会对人类健康和环境造成不同的问题. 因此，寻找纺织品无化学成分整理的替代工艺成为当前研究的热点. 鉴于此，在染色过程中采用植物源“蔓越莓果”和“蔓越莓果枝提取物”，尽量减少化学成分在棉花抗菌整理中的应用. 在两种不同温度下进行了不同时间的染色实验. 然后对样品进行了*K/S*、*CIE L*a*b*C**和*h*°值、耐光性、耐洗性和抗菌性分析结果表明，这两种天然染料对棉针织物有较好的染色效果，一般情况下，两种天然染料对棉针织物的染色牢度都较好，但耐光值有限，用越橘果枝提取物染色效果较差. 此外，在被测植物源中，用越橘果汁在低温染色和较低时间下对被测细菌具有良好的抗菌效果.

KEYWORDS
Antibacterial finish; cranberry fruit; natural dyes; cotton; textile

关键词
抗菌整理；蔓越莓；天然染料；棉花；纺织品

Introduction

Fibers, especially the natural ones as cotton, provide the conditions bacteria need for growing (Ursache et al. 2011) so antimicrobial effect of natural dyes become popular too. Antimicrobial finishing of textiles has become extremely important recently in the production of protective, decorative and technical textile products (Simoncic and Tomsic 2010). In this study, the dyeability

CONTACT Fazlıhan Yılmaz fazlihan.yilmaz@atauni.edu.tr Textile Engineering Department, Erciyes University, Kayseri, Turkey
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and meanwhile the antimicrobial efficiency of the cotton knit fabrics dyed with a natural dye source *Viburnum opulus* L. fruit and branch have been studied.

Natural dyes are sometimes regarded as positive since "natural" sounds environmentally friendly; sometimes as negative because they many be less reliable than synthetically manufactured dyes (Frose et al. 2019). They are known for their use in coloring of food substrate, leather as well as natural fibers like wool, silk, and cotton since pre-historic times (Samanta and Agarwal 2009). They can be derived from plants, invertebrates, or minerals (Purwar 2016). Moreover, many of the plants traditionally used in dyeing are credited with medicinal properties (Singh et al. 2005) and some of these have recently been shown to possess remarkable antimicrobial activity (İbrahim et al. 2010; Singh et al. 2005).

Cranberry (*Viburnum opulus* L.), called gilaburu in the Middle-Anatolia region, especially Kayseri city, Turkey and European cranberry bush in English (Sagdıc, Aksoy, and Ozkan 2006).*Viburnum opulus* L., belongs to the plant family of *Caprifoliaceae* (Çam and Hışıl 2007). It grows naturally in Anatolia (Kalyoncu et al. 2013). The plant is also called crampbark, guelder rose or snowball bush (Velioglu, Ekici, and Poyrazoglu 2006). European cranberry bush fruits are well known for their biological properties, of which some are due to the presence of anthocyanins in the berries. Gilaburu is a natural mineral source (Kalyoncu et al. 2013). High content of polyphenolic compounds has been identified in *Viburnum opulus* berries, such as hydroxybenzoic acids, tannins, anthocyanins, chlorogenic acid, (+)-catechin, (-)-epicatechin cyanidin-3 glucoside, cyaniding-3-rutinoside, and quercetin (Zaklos-Szyda et al. 2019). Different studies on the antibacterial and antioxidant activities of *Viburnum opulus* L. has been studied before. For example, Karaçelik et al. (2015) investigated the antioxidant activity of the juice and seed and skin extracts of cranberry plant grown in Eastern Black Sea Region in Turkey and they reported the antioxidant activities of the seed extracts were higher than those of skin extracts in general (Karaçelik et al. 2015). Likewise, Andreeva et al. (2004) have studied antioxidant activity of cranberry tree bark extracts. As a result of the studies, they found the presence of antioxidant activity. (Andreeva et al. 2004). Moreover, Sagdıc, Aksoy, and Ozkan (2006) have declared the antibacterial and antioxidant activities of dried fruit extract of cranberry and they were measured the total amount phenolics as 131.99 ± 2.11 mg gallic acid equivalent g^{-1} (Sagdıc, Aksoy, and Ozkan 2006). In our previous study, juice of gilaburu fruits were used in dyeing of wool fabrics and the antibacterial and antifungal properties of the dyed samples were tested with a different method used in this paper and limited reduction in bacterial growth has been detected (Şapcı et al. 2017). Differently in this study extracts obtained from cranberry fruit (*Viburnum opulus* L.) and cranberry fruit branch were used as a natural dye source for the cotton fabrics and the antibacterial efficiencies and dyeabilities of the knitted cotton fabrics were analyzed.

Material and method

Material

In the experiments, 100% cotton knit fabrics with a weight of 265 g/m^2 were used. The fabrics were in pretreated form and ready for dyeing process so no additional process was conducted prior dyeing. Cranberry fruit and cranberry branch were used as dyestuff source (Figure 1). The juices of the cranberry fruit were used directly in the dyeing process. The branches of the cranberry were ground after drying and extraction was carried out from these milled parts. This extract was used directly in the dyeing process.

Method

The dyeing experiments were carried out at two different temperatures (Figure 2). These temperatures are 70°C and 100°C. Liquor ratio was chosen as 1:60 in dyeing experiments. Cranberry fruit juice was directly used as the dye bath without any dilution in dyeing experiments. Cranberry branches were first subjected to an extraction process and this extract was used in the dyeing process.

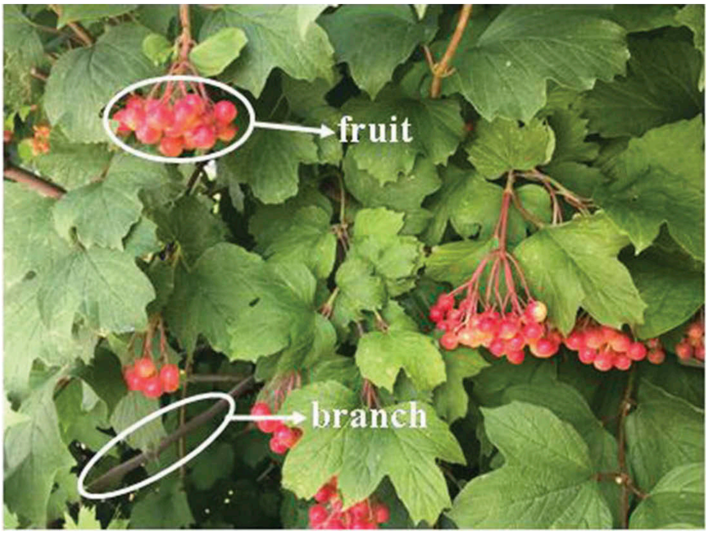

Figure 1. Cranberry bush (fruits and branches) in spring.

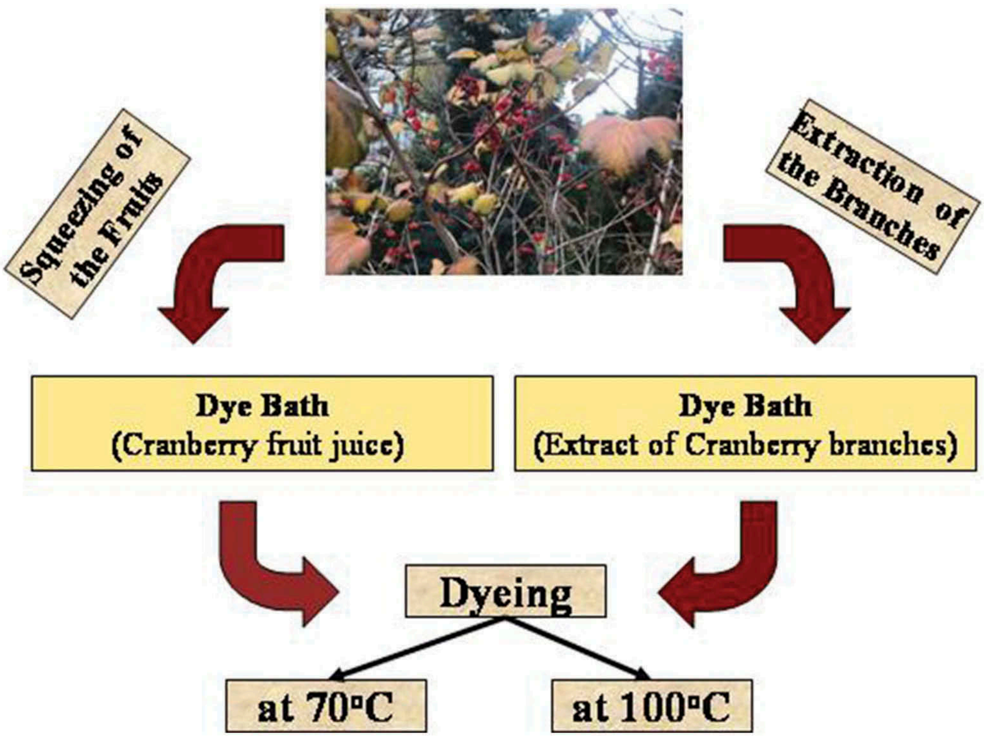

Figure 2. Use of Cranberry in the dyeing of cotton fabrics.

In the extraction process, 1.5 L of distilled water was used for 30 g cranberry branch and extraction was carried out for 3 h.

No mordant material was used in any of the dyeing processes. In other words, all dyeing experiments were carried out without mordant. In the dyeing experiments performed at two different temperatures (at 70°C and 100°C), dyeing processes were completed at three different times. That is, the dyeing process was started at 30 degrees and was expected for 10 min. Then, it reached 70°C in 20 min. After that, dyeing processes were completed in three different periods, 30, 60 and 90 min. Similarly, dyeing processes were carried out at 100 ^{0}C. After dyeing, cotton fabrics were washed with soft water. In the washing process, first 2 min rinsing was performed. After that, warm washing was carried out at 50 ^{0}C for 4 min. Finally, the dyed cotton fabrics were allowed to dry at room temperature after 2 min of final rinse. At the end of these processes, the dyed fabric samples were ready for various measurements.

As a result of the dyeing process, *K/S* and *CIE L*a*b*C** and *h*° color space values of the fabric samples were examined. Also, the fabrics were analyzed in terms of light fastness, washing fastness, and antibacterial efficiencies. Then, the fastness of the samples to washing (ISO 105-C10 2006 in test condition of Test A (1)) and light (ISO 105-B02 1994) were tested, and finally the antibacterial efficiencies of the samples against Gram-negative bacteria (*Escherichia coli* ATCC 25922) and Gram-positive bacteria (*Staphylococcus aureus* ATCC 29213) has been tested. For the testing antibacterial efficiencies of the samples ASTM E2149–01 (2001) standard has been used and the bacteria reduction (%) after 24 h of contact time has been obtained as described in our previous study in detail (Bahtiyari and Yılmaz 2018).

$$\text{Bacterial Reduction}(\%) = 100 \times (\text{B} - \text{A})/\text{B}$$

A: "24 h" contact time CFU/mL of the flask containing dyed sample

B: "0" contact time CFU/mL of the flask

To analyze the chemical content of the extract and juice; 100 µL was taken from the samples placed in screw-cap tubes (15 mL). Four hundred microliter of nitric acid (Merck, Germany) and 100 µL of hydrogen peroxide (supra pure) (Merck, Germany) solutions were added into them. They were burned at 100°C for 120 min in the microwave device (Milestone S.r.l. Italy) then 9.5 mL of deionized water was added to the samples, and test tubes were centrifuged and prepared for analysis. In order to test the performance of the device (Agilent 7700 a series ICP/MS) to make it ready for analysis, the tuning was made with the Y (Yttrium), Li (Lithium), Co (Cobalt), Tl (Thallium), Er (Erbium) tuning solution. With 2% nitric acid solution at increasing concentrations containing elements (Cr, Mn, Co, Ni, Cu, Zn, Se, Se, Ag, and Cd) to be analyzed before measurement standards were prepared (0, 0.625, 1.25, 2.5, 5, 10, 20 ppb), and then they were introduced to the ready-to-analyze device and the calibration curve was drawn. During the analysis, in order to correct the deviations occurring on the calibration line, the internal standard which includes Indium, Scandium, Germanium, Bismuth elements representing the periodic table was given to the device. Then, the results for the desired elements were obtained in ppb.

Results and discussion

When Table 1 is examined, in the dyeing process using cranberry fruit juice and cranberry fruit branch extract, the *K/S* value varies between 0.89 and 0.31. The highest *K/S* value was obtained by using cranberry fruit juice at 100°C and 90 min. The lowest *K/S* value was obtained by using cranberry fruit branch extract at 70°C and 30 min dyeing process. With the increase in dyeing temperature, a continuous increase in *K/S* values occurs. In the same way, *K/S* values increase with the increase in dyeing time.

In the dyeing process using cranberry fruit juice and cranberry fruit branch extract, brown and shades were obtained. For example, in the dyeing process using cranberry fruit juice for 90 min and at 100°C, it was obtained as a^* = 4.48, b^* = 14.48 and $h°$ = 72.8 and the color is perceived to be brown. In the dyeing process, which was conducted for 30 min at 70°C by using cranberry fruit branch extract, a^* was 2.74, b^* was 8.55 and $h°$ was 72.22 and the color is perceived as light brown.

When the L^* values in Table 1 were examined, the highest L^* value was found as 81.97, while the lowest L^* value was 75.17. The highest L^* value was obtained by using cranberry fruit branch extract at 70°C for 30 min in dyeing process. The lowest L^* value was obtained from the dyeing process with cranberry fruit juice for 90 min at 100°C. Increases in dyeing temperature and dyeing time cause a decrease in L^* values. This showed that instead of extract from the branches of the plant, the juice of the fruit gives more color to the sample and higher temperatures and dyeing durations are responsible for the higher *K/S* values and darker colors but in brown shades.

The washing and light fastness values are presented in Table 2. Washing fastness of cotton fabrics dyed with cranberry fruit juice was generally good in terms of color change and staining. The staining on cotton was perfect and the fastness results were 5 and the color changes during the washing fastnesses were generally 4–5. When the light fastness of the fabrics dyed with cranberry fruit juice was examined, it was observed that the limited values were obtained. The change in dyeing temperature and dyeing time did not affect the fastness values of the cranberry fruit juicedyed fabrics.

Likewise, the washing fastness values of the samples dyed with cranberry fruit branch extract have good results in terms of both color change and staining values. However, for light fastness values, it is not possible to say this. Because in all of the dyeing experiments with cranberry fruit branch extract the light fastness was 2. It is a low value and shows insufficient fastness to the light. As with the cranberry fruit juice dyeing experiments, the change in the dyeing temperature or dyeing time in the dyeing experiments with cranberry fruit branch extract did not cause a significant change in the fastness values. So it can be concluded that by this plant good washing fastnesses but limited light fastnesses can be obtained and the dyeing conditions did not change the obtained values but the type of the natural dye source have an effect on the light fastnesses.

As a result of dyeing experiments with cranberry fruit juice and cranberry fruit branch extracts, the antibacterial effects were tested too but no bacterial reduction has been detected from the samples colored with the cranberry fruit branch extracts.

In cotton fabric samples just washed at 70°C or 100°C but not dyed, no antibacterial effect was detected against the tested two bacteria species. In other words, the cottonknitted fabric has no antibacterial effect. However, in the dyeing process using cranberry fruit juice at 70°C, good results were obtained especially against *Staphylococcus aureus* type bacteria. Change in dyeing time did not cause any change in antibacterial effect values for this bacterial species. That is, an antibacterial effect of 99% was obtained at all dyeing times. But it is not possible to say this for *Escherichia coli* type bacteria because in the dyeing process using cranberry fruit juice at 70°C, increased dyeing duration decreased antibacterial effect. Ninety-nine percent of the antibacterial effect was achieved in the 30minute dyeing process, while this value decreased to 50% if the dyeing was conducted for 60 min. And for 90 min, no antibacterial effect had been detected against the *Escherichia coli* bacteria. When the dyeing experiments using cranberry fruit juice at 100°C were examined, it was observed that good antibacterial effect values were obtained against *Staphylococcus aureus* bacteria type as in dyeing at 70°C. The bacterial reductions in that case were nearly 80%. The increase in dyeing time did not cause a significant change for the antibacterial effect. However, an increase in the dyeing temperature caused significant decrease in antibacterial efficiency against the *Staphylococcus aureus* bacteria type. On the other hand, no antibacterial effect was detected against the *Escherichia coli* bacteria in dyeing experiments using cranberry fruit juice at 100°C. In other words, with the increase of dyeing temperature, antibacterial effect was found to be disappeared for *Escherichia coli* bacteria. This is thought to be due to degradation of phenolic compounds in the bath with increased dyeing time and temperature. Interestingly as seen from Table 3, no antibacterial effect was found against both types of bacteria in any of the dyeing experiments performed by using the cranberry fruit branch extract. In other words, the cranberry fruit branch extract has not a feature gaining an antibacterial efficiency to the cotton fabric for these tested parameters. To investigate the possible reasons of this obtained antibacterial features by the tested herbal sources, the ICP/MS analysis of the extract and fruit juice has been managed (Table 4).

According to Table 4, the herbal sources contain different elements but the amount of copper and zinc was significantly observed in cranberry fruit juice. In particular, the zinc ratio is approximately 16 times higher in cranberry fruit juice than the cranberry fruit branch extract. Previous studies have shown that zinc and copper have antibacterial properties (Fiedot-Tobola et al. 2018; Rubin, Neufeld, and Reynolds 2018). So the reason for the high antibacterial effects of the samples dyed with cranberry fruit juice is thought as can be related with the presence of the high amount of metal

Table 1. Colors and Color efficiencies of the dyed samples.

Natural dye source and dyeing temperature	Minute of dyeing	*CIE L*a*b** (D65)						Color
		K/S	*L**	*a**	*b**	*C**	*h°*	
Dyeing with cranberry fruit juice at 70°C	30	0.41	81.33	2.86	10.42	10.81	74.63	
	60	0.44	81.23	2.91	11.12	11.49	75.33	
	90	0.52	80.43	3.16	12.04	12.44	75.28	
Dyeing with cranberry fruit juice at 100°C	30	0.57	78.52	2.34	11.54	11.77	78.56	
	60	0.77	75.86	3.73	13.38	13.89	74.43	
	90	0.89	75.17	4.48	14.48	15.16	72.8	
Dyeing with cranberry fruit branch extract at 70°C	30	0.31	81.97	2.74	8.55	8.98	72.22	
	60	0.34	80.54	2.56	8.25	8.64	72.77	
	90	0.42	79.08	3.37	9.42	10	70.31	
Dyeing with cranberry fruit branch extract at 100°C	30	0.49	77.49	3.54	9.81	10.43	70.15	
	60	0.51	77.19	3.55	10.07	10.67	70.6	
	90	0.55	76.27	3.65	10.16	10.79	70.26	

Table 2. Fastness values of the samples.

Natural dye source and dyeing temperature	Minute of dyeing	Washing fastness		Light fastness
		Sta.	C.C.	
Dyeing with cranberry fruit juice at 70°C	30	5	4–5	3–4
	60	5	4–5	3–4
	90	5	4–5	3–4
Dyeing with cranberry fruit juice at 100°C	30	5	4–5	3–4
	60	5	4	3–4
	90	5	4–5	3–4
Dyeing with cranberry fruit branch extract at 70°C	30	5	3–4	2
	60	5	4	2
	90	5	4–5	2
Dyeing with cranberry fruit branch extract at 100°C	30	5	4–5	2
	60	5	4–5	2
	90	5	4–5	2

Sta.: Staining on cotton; C.C.: Color Change

Table 3. The Antibacterial test results of the samples.

Natural dyeing		Dyeing duration	Bacteria reduction (%)	
			Staphylococcus aureus	*Escherichia coli*
Dyeing without use of any natural source	at 70°C	90 min	-	-
	at 100°C	90 min	-	-
Dyeing with cranberry fruit juice at 70°C		30 min	99	99
		60 min	99	50
		90 min	99	-
Dyeing with cranberry fruit juice at 100°C		30 min	80	-
		60 min	79	-
		90 min	80	-

Table 4. Results from ICP/MS device.

	Al (µg/dL)	Cr (µg/dL)	Mn (µg/dL)	Co (µg/dL)	Ni (µg/dL)	Cu (µg/dL)	Zn (µg/dL)	Se (µg/dL)	Ag (µg/dL)	Cd (µg/dL)
Cranberry fruit juice	12.66	6.17	190.76	6.91	102.34	271.31	1635.35	1.39	0.21	0.41
Cranberry fruit branch extract	5.89	5.43	58.55	0.39	123.89	81.35	108.12	2.62	0.57	0.46

ions especially copper and zinc addition to the phenolic compounds responsible from the antibacterial properties.

Conclusion

Nowadays, it is important for people to choose natural origin substances in terms of health. Therefore, there is often a prejudice against chemical substances. So in this study, the antibacterial properties of natural dye sources were investigated. In the dyeing of cotton fabrics by using cranberry fruit juice or cranberry fruit branch extract, good antibacterial efficiency against the tested bacteria were obtained with the use of cranberry fruit juice. In addition, it was seen that the tested two different parts of the cranberry could color the cottonknitted fabrics. In general, the washing fastness for both natural dye sources was good, but light fastness values were limited and in dyeing processes by using cranberry fruit branch extract it was poor. As a summary by this study, it has been found that cotton knitted fabrics can be colored by using cranberry fruit juice and that the fabric can gain an antibacterial properties if the dyeing temperature and dyeing duration were low. So the fruit juice of the cranberry can be considered as a source of dye and antibacterial agents.

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