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Investigation of the Possibilities of Using *Rhaponticoides Iconiensis* in Wool Dyeing

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Article

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ABSTRACT

*Recently, many scientists have been struggling to identify natural dyestuff sources from different plants, especially for dyeing of textile materials such as wool and cotton. One of these struggles was carried out in this paper. The aim of this study was made to reveal R. iconiensis (hub.-mor.) M.V.Agab. & Greuter plant used as a source of natural dyes. In this study, both plant's flowers and stems have been used separately for the dyeing of wool fabrics. These parts of plants were collected in the summer and separately dried, then milled and powdered and then used as a dyeing material in the dyeing of woollen fabrics via five (Fe, Cu, Al, Cr, Sn) different metal salts. All the dyeing processes were carried out for one hour at boiling temperature. Subsequently, CIE L*a*b* and K/S values of dyed wool fabrics were examined for colour strength. Various colour fastness tests, such as rubbing, washing and light fastness of dyed fabrics, have also been made. In the light of the obtained data, it was seen that R. iconiensis plant could be a natural dyestuff for wool materials.*

KEYWORDS

Rhaponticoides iconiensis, natural dyes, wool, dyeing, mordant

INTRODUCTION

Textile manufacturing is one of the oldest industrial sectors and it consumes water and energy in almost every stage of the textile manufacturing chain [1]. In addition to this, effluent from textile finishing processes contains high concentrations of biologically difficult-to-degrade or even inert auxiliaries and chemicals [2]. There is pressure on dye manufacturers to develop dyes that can be successfully applied using fewer auxiliary chemicals, especially salt, to reduce environmental problems associated with textile industry effluent [3]. Studies on natural dyes have become popular again, owing to the increasing demand for natural-based materials [4-7]. Natural dyes are less toxic, less polluting, less health hazardous, non-carcinogenic and non-poisonous [8] and it is known that many of the plants from which natural dyes are obtained have medicinal properties [9]. Today it becomes important to investigate different plants both for colouration and functionalization of textiles and also different studies available to apply novel methods in natural dyeing. For example, in one study, isolation of tannin from neem bark and its application onto silk fabric via the help of ultrasound has been investigated [10]. In another study, a new eco-friendly source of natural brown colourant from arjun bark has been tested and both bark powder and fabrics were microwave irradiated and then tested for dyeing performance [11]. Differently, in another study, the extraction of lutein dye from marigold flowers and the role of gamma radiation in improving colour strength and fastness characteristics of the extracted dye has been investigated [12].

In this study, the *R. iconiensis* plant, an endemic plant [13], has been studied in order to contribute to the world of natural dyes. *Rhaponticoides* is a steppe related genus that has widespread taxa, ranging from the Tain Shan to the Iberian Peninsula [14]. *R. iconiensis*, 1 to 2 meters in length, with numerous leafy yellow flowers interspersed with foliage, is an endemic plant in the Central Anatolian region in Turkey [15]. Demirpolat et al. [16] reported that flower extracts of *R. iconiensis* have showed considerable cytotoxic activity against various cancer cell lines. On the other and, Paşayeva et al. [17] reported that the α-amilaz and α-glucosidase inhibition activity, antioxidant, antimicrobial properties and phytochemical composition of the extract and sub-extracts obtained from the leafy stem of *R. iconiensis* were evaluated. And also, they reported that the favonoids, such as naringin, apigenin and patuletin derivatives, were determined as the main compounds of the active TSE sub-extract.

In this context, it has been tested whether *R. iconiensis* can be a natural dyestuff source in the colouring of woolen fabrics.

EXPERIMENTAL

Materials and Methods

In the experiments, both the flowers and stems of the *R. iconiensis* plant (Figure 1) were used separately for dyeing woolen fabrics. Washed and ready-to-dye 100% wool fabric with a weight of 200 g/m^2 was used in the experiments. Natural dyes are mostly non-substantive and must be applied to textile materials with auxiliaries called mordants, which is usually a metallic salt [18].

a) b) c)

Figure 1. *R. iconiensis* plant (a), dried and ground flower part of the plant, (b), stem part of the plant (c)

Because of this, five different mordanting agents (MA), MA1 ($KAl(SO_4)_2.12H_2O$), MA2 ($CuSO_4.5H_2O$), MA3 ($FeSO_4.7H_2O$), MA4 ($SnCl_2.2H_2O$) and MA5 ($K_2Cr_2O_7$), were used at a concentration of 3%, except the MA1 mordant (alum). Alum (MA1) was used at a concentration of 20%. Although it is known that the metal salt-based mordanting agents, such as copper or chromium-based ones, were not suitable for the environmentally friendly productions, they were tested just to shape the effect on the obtained colours. The dried and ground plant-based natural dyestuff source was taken equal to the weight of the material to be dyed (1:1) and added directly to the dyebath (Figure 2).

Dyeing Procedures

In this study, dyeing and mordanting processes were carried out simultaneously, and at the beginning of dyeing, the mordant agent, dyestuff source and woollen fabric were placed in water with pH 6.5-7, and then this mixture was heated to reach 100 °C for 1 hour. At this temperature, the process was continued for 1 hour and then the washing process was started. The studies were carried out in the Termal branded HT sample dyeing laboratory machine. The mordanting and dyeing methods are shown in Figure 2. As seen from the figure the dyeing was started at room temperature. After 5 minutes at room temperature the bath was heated to the boiling temperature and the dyeing was continued for 1 hour at this temperature. After the dyeing process, dyed wool fabrics were subjected to warm rinsing, soapy washing (1 ml/L non-ionic surfactants, at boiling temperature) and cold rinsing, respectively. Finally, the samples were dried at room temperature.

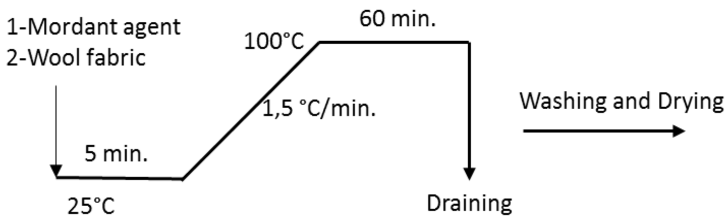


Figure 2. Dyeing procedure

The all-experimental plan applied in this study is presented in detail in Figure 3.

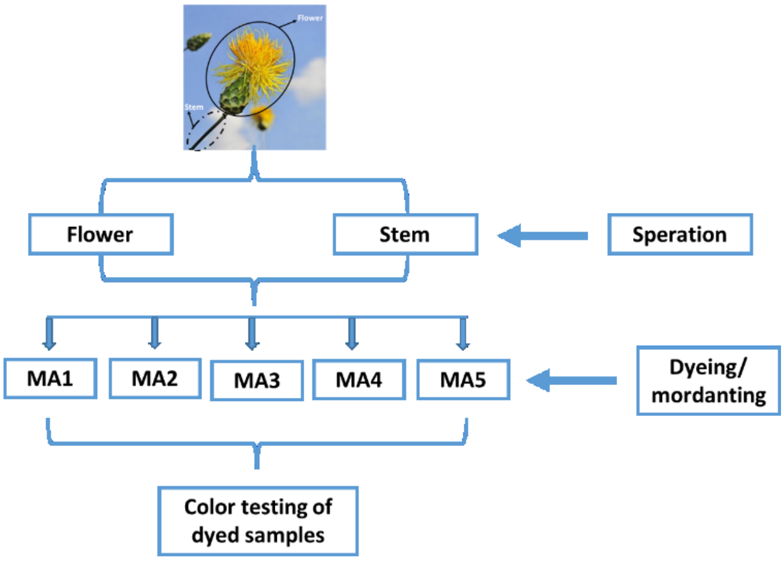


Figure 3. Schematic representation for all experiments performed

Colour measurements

The dye adsorbed by the wool fabrics and the obtained colour properties were measured using a spectrophotometer (Minolta 3600d) under illuminant D65, with a 10° standard observer. For this aim the colour yields (*K/S*) and colour values (CIE $L^*a^*b^*$) of the dyed wool fabrics were collected according to Kubelka-Munk equation [19]. Moreover, the scanned photos of the samples were added to the paper to show the obtained colours easily.

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

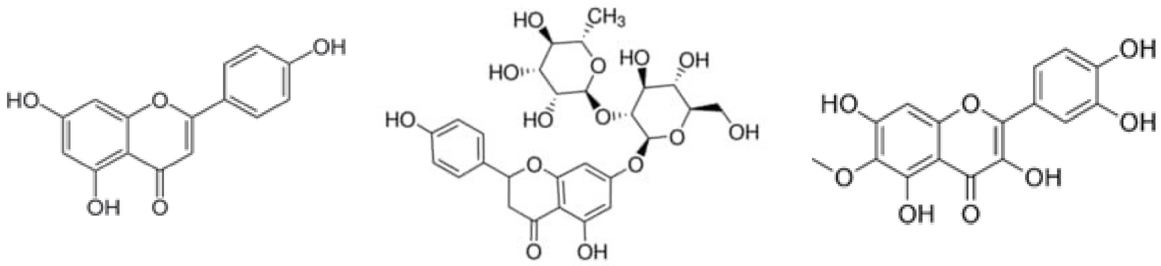
where K was the absorption coefficient, S was the scattering coefficient, R was the reflectance.

Evaluation of colour fastness

Rubbing, washing and light fastness properties have been tested. Wet and dry rubbing fastness of dyed fabrics is according to ISO 105 X12 [20]; wash fastness was measured according to ISO 105 C010-A [21] and light fastness according to ISO 105 B02 [22].

RESULTS AND DISCUSSION

Today, many different natural dyestuff sources are used in the colouring of textile materials. In our previous studies, we reported that red cabbage [17], red beetroot [18], and black carrot [19] can be considered as a source of dyestuff. In this study, the *R. iconiensis* plant, which is a different natural material, was investigated as a source of dyestuff. Paşayeva et al. [20] reported that the chemical compounds in the structure of the plant-based dyestuffs we use in dyeing processes are the chemical compounds such as patuletin, apigenin and naringin shown in Figure 4.



a) Apigenin b) Naringin c) Patuletin

Figure 4. Important chemical compounds in the structure of the plant

The structure of the wool contains amino (-NH_2) and carboxylic (-COOH) groups held together by salt linkages that connect the main peptide cahins. Proteins and carboxylic groups that form the structure of amino acid form the dyeing process by forming hydrogen bridges with dyestuffs over metal ions. The metal salt forms an ionic bond between the fiber and the dye [26] (Figure 5).

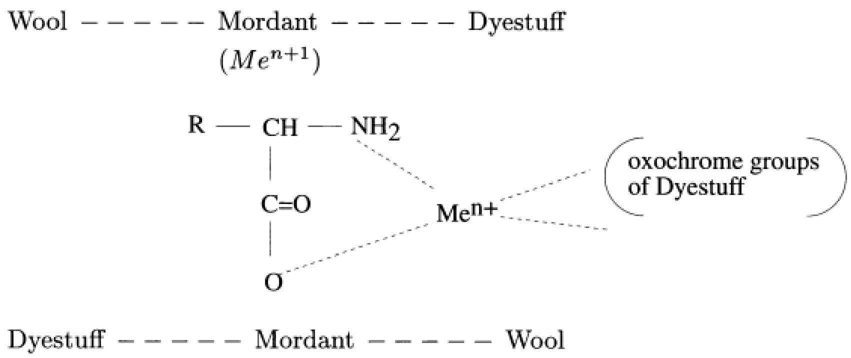


Figure 5. The mechanism of simultaneous mordanting and dyeing for wool fabric

Synthetic commercial dyestuffs, such as acid, chromium, metal-complex and reactive dyes, can be used to colour wool fabrics [27]. But here in this study an endemic plant *R. iconiensis* has been tested for the colouration of the wool fabrics. In order to evaluate the dyeing efficiency of the plant on wool fabric, the flowers and stems of the plant were used separately as natural dyestuff sources. CIE *L*a*b** colour values and *K/S* colour yields of dyed wool fabrics given in Table 1 were collected. As known, in CIE *L*a*b** space, *L** indicates the lightness; the perfect reflecting diffuser has *L**= 100 and the perfect black has *L**= 0. The colours with *a**>0 represent redness and with *a**<0 greenness; *b**>0 means yellowness and *b**<0 blueness [28].

As seen from the Table 1 and Figure 6, the obtained colours from *R. iconiensis* plant were changing depending on the part of the plant used and the mordanting agent type. The flavonoids are a remarkable group of plant metabolites [17]. It is thought that obtaining particular colours is related to the flavonoid content of the plant. The flavonoids that give yellow colours can be classified as flavones, isoflavones, aurones and chalcones.

Table 1. The CIE $L^*a^*b^*$ and *K/S* values of dyed samples

	Flower					Stem				
	MA1	MA2	MA3	MA4	MA5	MA1	MA2	MA3	MA4	MA5
K/S	1.64	10.73	8.75	9.78	11.61	9.07	11.94	7.66	20.02	15.74
L^*	75.34	50.66	44.50	54.24	64.33	61.99	49.08	48.21	47.96	65.34
a^*	4.52	5.61	4.92	13.07	24.05	13.40	3.06	4.17	17.18	6.53
b^*	26.86	28.75	18.17	38.08	64.34	46.59	26.77	18.11	42.08	53.64
C^*	27.24	29.29	18.82	40.26	68.69	48.48	26.95	18.59	45.45	54.04
$h°$	80.46	78.96	74.84	71.06	69.51	73.96	83.49	77.04	67.79	83.06

Most of the naturally obtained yellow colours are derivatives of flavones and isoflavones containing hydroxyl and methoxy groups [21]. The difference of the colours from different parts of the plant can also be related with the type of the flavonoids and their derivatives as well. From Table 1, it can be easily said that colour yields and colour values (CIE $L^*a^*b^*$) may vary depending on the type of mordant in dyed fabrics. For instance, with the MA3 mordant, different shades of brown were obtained depending on the part of the plant used. When the flower of the plant was used for dyeing the fabric, the CIE $L^*a^*b^*$ values of the dyed fabrics were determined as 44.5, 4.92 and 18.17, respectively. With MA4, the lightness of the dyed sample was 54.24 and the colour was redder (a^*:13.07) and yellower (b^*:38.08) when compared with the sample dyed with the mordant 3 and the flower of the plant. The highest yellowness (b^*) has been observed from the dyeing managed with both the flower and stem of the plant in the presence of the MA5 mordant (b^*:64.34 when dyeing with the flower; b^*:53.64 when dyeing with the stem). Therefore, depending on the mordanting agent and the plant part, different colours have been found; however, when the hues were taken into account it could be said that they were all in red-yellow region in CIE colour space.

Deciding on the plant-based natural dyestuff source used is one way of evaluating colour yields (K/S). In the light of the data obtained from the table, it can be said that sufficient colour yield can be obtained from this natural dye source. As a result of dyeing using the MA4 mordant, it was observed that the highest colour yield could be obtained when the stem of the plant was used. The colour yield in this case was 20.02. The lowest colour yield was obtained from the fabric samples were dyed in combination with the flower of the plant and the MA1 mordant.

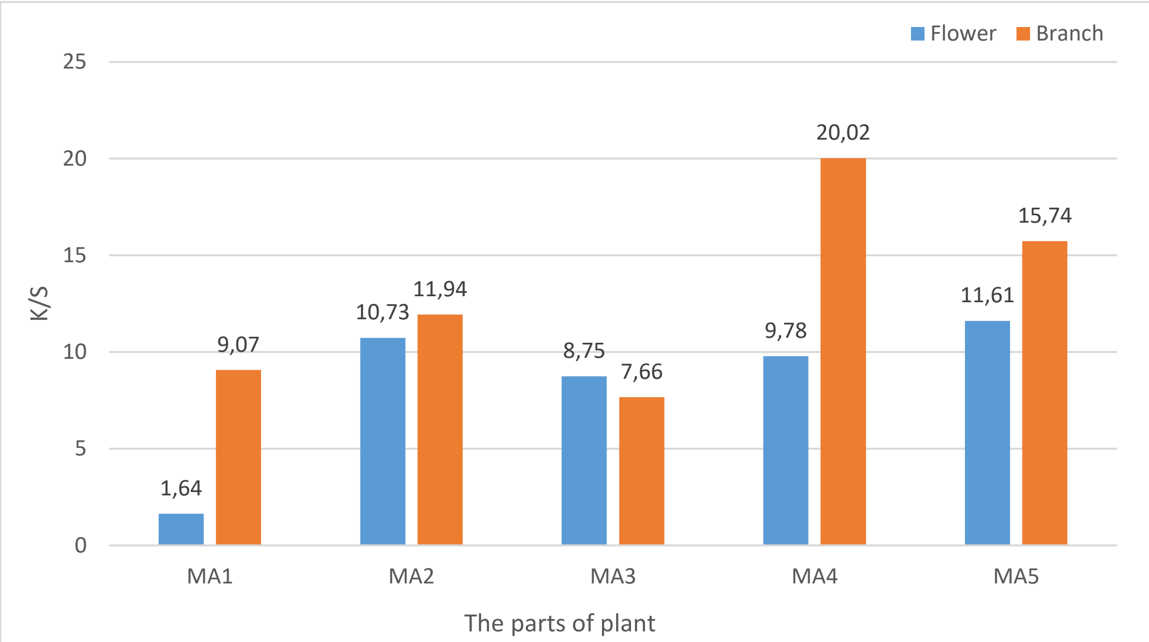


Figure 6. The K/S values of dyed wool fabrics with *R. iconiensis*

To show the obtained colours clearly, the dyed samples' scanned photos were collected as well (Table 2). As can be seen from Table 2, it can be said that the colours of the fabric samples dyed using the stem and the flower of the plant are different in general and the colour differences may be more dominant and variable depending on the mordant agents. These results were also confirming the results in Table 1. For example, in dyeings with MA1 the colours obtained from the flower were significantly different from those obtained from the stem. When beige colours were obtained from the flower of the plant, the stem of the plant produced mustard colours. Likewise, with the MA5 mordant, the colours obtained from the samples dyed with the stem were also significantly different from those obtained from the flower of the plant. In the case of the flower with the mordant 5, the colour was in orange shades; however, light mustard colours were obtained with the use of the stem. On the other hand, if the mordant 2 had been used during dyeing with *R. iconiensis,* the khaki colours would have been in different shades both with the flower and the stem, but especially with the stem.

Table 2. Wool fabrics dyed with natural dye source *R. iconiensis*

	Type of the mordant agent				
	MA1	MA2	MA3	MA4	MA5
Flower					
Stem					

Actually, the data in Table 1 supports this. As seen from Table 1, the lowest a^* value had been obtained from the sample coloured with the stem of *R. iconiensis* in the presence of the MA2 mordant. Otherwise, with the use of MA4 in dyeing with the stem part the brick red (Table 2) had been obtained and the L^*= 47.96, a^*=17.18, and b^*= 42.08 values were measured (Table 1). Consequently, as can be seen from Tables 1 and 2, it was found that the flower and stem parts of the plant can produce different colours and tones in woolen fabrics.

Another important parameter in the evaluation of the natural dye source in terms of usability in dyeing textile materials is the fastness values of the dyed fabric samples, and the fastness of the dyed samples obtained at the end of the trials was also tested (Table 3). It has been determined that excellent results (4/5-5 values) can be obtained for both colour change and staining in terms of washing fastness properties. In general, the same trend holds for rubbing fastnesses, except for the stem in the presence of the MA3 mordant. In this case, the wet rubbing fastness is 3/4.

Table 3. Fastnesses of the dyed samples

		Rubbing		Washing		
		Wet	Dry	Alteration	Alteration	Light
MA1	flower	5	5	5	5	2
	stem	5	5	5	5	2/3
MA2	flower	4/5	5	4/5	5	4
	stem	4/5	5	5	5	4
MA3	flower	4/5	5	4/5	5	3/4
	stem	3/4	4	4/5	5	4/5

		Rubbing		Washing		
		Wet	Dry	Alteration	Alteration	Light
MA4	flower	5	5	5	5	3
	stem	5	5	5	5	4/5
MA5	flower	4/5	5	4/5	5	2
	stem	4/5	5	4/5	5	2

Another important fastness is light fastness. When Table 3 is examined, it is seen that different light fastness results can be obtained depending on the mordant type. In other words, the mordant type is of great importance in terms of light fastness. Poor light fastness results were obtained in the dyeings with the MA1 and MA5 mordants. The MA2, MA3 and MA4 mordants have moderate fastness values depending on the part of the plant used. This may be due to the obtained colours. From Table 1, it can be said that the lightest colours (colours have the highest *L** values) were obtained by using the MA1 and MA5 mordant agents, and light colours gave lower light fastness than dark colours. This may be the reason for these low light fastnesses.

CONCLUSION

Nature has presented many alternatives to human beings for colouration of their goods and their life since ancient times. Nature has given us inspiration to create civilization and art. In this respect, people have been using different herbal sources for the colouration of textile materials and today it has started to become popular again due to different advantages of using these natural resources.

This study focused on the introduction of a plant as a new natural dye source. *R. iconiensis* has been tested for this aim. The flowers and stem of the plant were dried and milled separately and used for colouration of the wool fabrics after the pre-mordanting process. It has been noticed that different colours and tones of surfaces can be obtained by using different mordanting agents together with the pre-mordanting process. It can be stated that when different parts of the plant, such as the flower and the stem, are used as a dyestuff source, different colour tones can be obtained, although there are more differences in the tones obtained by different mordant agents. In addition, the fastness tests of the dyed wool fabric samples were also carried out. In general, the results of the washing and rubbing fastnesses of the dyed samples were between good and excellent, and it was determined that these results differed little, depending on the mordant agents used and the part of the plant to be used as the dyestuff. In addition, the light fastnesses of the dyed samples showed varying degrees, from 2 to 4/5, depending on the mordanting agent used and the region where the plant was used.

The results clearly show that the tested *R. iconiensis* plant can be a natural source of dyestuff and can be used for colouring woolen fabrics.

Author Contributions

Conceptualization - Benli H and Bahtiyari Mİ; methodology - Benli H and Bahtiyari Mİ; formal analysis - Benli H and Bahtiyari Mİ; investigation - Benli H and Bahtiyari Mİ; resources - Benli H and Bahtiyari Mİ; writing - original draft preparation - Benli H, Bahtiyari Mİ, Paşayev N and Tugay O; writing - review and editing - Benli H and Bahtiyari Mİ; visualization - Benli H, Bahtiyari Mİ and Tugay O; supervision - Benli H and Bahtiyari Mİ, All authors have read and agreed to the published version of the manuscript.

Conflicts of Interest

The authors declare no conflict of interest.

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