

## Application of Different Non-ionic Surfactants on the Conventional Extraction and Dyeing Characteristics of Natural Dyes on the Wool Yarn

M. Sadeghi-Kiakhani<sup>1,2\*</sup>, A. R. Tehrani-Bagha<sup>3</sup>, R. Aladpoosh<sup>1</sup>, E. Hashemi<sup>4</sup>

<sup>1</sup> Department of Organic Colorants, Institute for Color Science and Technology, P.O.Box: 16765-654, Tehran, Iran.

<sup>2</sup> International Scientific Studies and Cooperation Center of the Ministry of Science, Research and Technology, P.O. Box: 15875-7788, Tehran, Iran.

<sup>3</sup> School of Chemical Engineering, Aalto University, Espoo 02150, Finland

<sup>4</sup> Department of Chemistry, Faculty of Sciences, Shahid Rajaei Teacher Training University, P.O. Box: 16785-163, Tehran, Iran.

### ARTICLE INFO

Article history:

Received: 30 Oct 2023

Final Revised: 15 Jan 2023

Accepted: 16 Jan 2023

Available online: 07 Feb 2024

Keywords:

Extraction

Dyeing

Natural dyes

Non-ionic surfactant

Wool yarns

### ABSTRACT

*This study presents the application of three commercial non-ionic surfactants, namely nonylphenol polyethylene glycol ether (Kenon), sorbitan ester polyethylene glycol ether (Tween), and oleic acid polyethylene glycol ester (Keol), to improve the extraction process of weld, madder, and walnut green husk natural dyes. The critical micelle concentration (CMC) values of Keol, Kenon, and Tween were 0.15, 0.19, and 0.25 g/L, respectively. The introduction of non-ionic surfactants into the solution markedly improved colorant extraction from plant sources, leading to a significant increase in the percentage of dye extracted in water. The wool dyeing with extracted dyes was also found to benefit from the presence of Tween, resulting in up to 11, 29 and 28 % increase in the color strength for weld, madder and walnut green husk extracts, respectively. Moreover, non-ionic surfactants improved dyeing and levelling properties of wool samples, and did not negatively impact on the general fastness ratings of dyed samples. Overall, this study suggests that the use of non-ionic surfactants provides a favorable manner for improving the extraction process of natural dyes and enhancing their dyeing properties. Prog. Color Colorants Coat. 17 (2024), 275-288 © Institute for Color Science and Technology.*

### 1. Introduction

The hazardous wastes release in the stage of synthesis of synthetic dyes and their application in the textile industry. They have not only consequence of health matters, but also damage the environment [1]. Recently, there has been an interest exposed to the application of natural dyes due to their higher compatibility with the environment and bio-degradability [2]. Annually, approximately 8500 million tons of colored wastewater is drained all over the world, which 55 % of them go into aquatic sources without the removal of contaminants. The high concentration of colorants

disrupts the aquatic environment due to prevention of the light from entering the deep water. This phenomenon affects the photosynthetic activity of aquatic animals and reduces the quality of water, and consequently it is a threat to agricultural land and marine life [1, 3]. These pollutants contain non-biodegradable and carcinogenic components, and their purification requires a significant amount of cost, time and processes [4, 5].

Industrialists in all fields, including textile, food, pharmaceutical, and cosmetics, are striving to produce green and sustainable products [6]. The progress of living criteria and the increase in ecological awareness

\*Corresponding author: \* sadeghi-mo@icrc.ac.ir

<https://doi.org/10.30509/pccc.2024.167223.1258>

of users have caused natural dyes to be considered as sustainable, safe products, and replacement of synthetic dyes. Natural dyes have low side effects and high safety factor, biodegradable, antifungal, antibacterial, and antioxidant activities and compatible with the environment, [3]. A number of natural dyes have brighter, more attractive, as well as insect repellent properties on the textile [6-8].

The first step to separate the desired chemical compounds from natural sources is extraction. In this process, the desired effective substances are usually obtained by using a medium containing different solvents during a particular time. Usually, water is solvent used in the conventional method, so that a long time and high energy is required to perform this process. The efficiency decreases in many cases due to the heat, energy and time used [9]. Generally, in the extraction process, the solvent penetrates into the matrix of the color source containing the active ingredient, and the effective compounds that must be separated from the color source and transfer the solvent phase. The effective compounds are solubilized inside the solvent phase and finally the extracted compounds are collected. It is noteworthy that the extraction method, the nature of the solvent used in terms of polarity and type, the size of the source particles containing the active substance, the amount of solvent, the extraction temperature and pH are the significant factors that affect the extraction efficiency [10, 11]. It has been mentioned that the extraction process is carried out with higher efficiency solvents using by a polarity close to the polarity of the effective compounds and the smaller particle size of the color sources. However, the very small size of the particles creates a problem in the filtering stage. Despite the fact that high temperature increases the solubility and dispersion of active substances, its excessive increase may cause the decomposition of heat-sensitive components in the active substances, which is undesirable, and it is necessary to use methods that extraction should be performed at a lower temperature [12, 13]. Since natural dyes and their effective compounds have a different chemical structures, the best method and conditions are different for each plant, and it is very important to find methods that can obtain the most effective substances with minimal destruction and impurity. Meanwhile, the ideal method is a method that is simple, low-cost, fast, uses the least number of organic solvents, and has high selectivity and

reproducibility in addition to the more efficiency.

An important group of organic compounds are surfactants. They consist of two chemical parts with different properties in terms of polarity. The head and tail groups in surfactants had affinity to polar and non-polar phases, respectively. Surfactants are usually used as dispersants, detergents, antimicrobial agents, softeners, and auxiliaries in textile industry [14, 15]. Due to the unique structure of surfactants, they are generally utilized to reduce the interfacial tension between two or more phases. Their affinity to stay at self-assembled structures in the solution causes the formation of micelles with variable sizes in the range of nano to micro [16]. The micelle can be formed at a very low concentration of the surfactants, which reduces the free energy of the system. Micelles are also used to increase the solubility of substances that are usually insoluble or weak soluble in the medium [17].

Natural dyes are commonly extracted from plants using water and some organic solvents at varying temperatures. The innovation in this work involves incorporating low concentrations of surfactants into water, improving the extraction efficiency of hydrophobic colorants without the use of organic solvents. This represents a greener, simpler, and more cost-effective approach. In this study, the efficiency of non-ionic surfactants in the extraction of natural colorants from weld, madder and walnut husk has been investigated by conventional method. For this purpose, three types of non-ionic surfactants with the brand names Kenon containing ethoxylated nonylphenol, Keol containing ethoxylated oleic acid and Tween containing ethoxylated polysorbate were selected. CMC (Critical Micelle Concentration) of each surfactant was measured by surface tension method and the extraction efficiency was determined by UV-Visible spectrophotometer at different concentrations of surfactants. Also, colorimetric data and color fastness ratings of dyed samples were studied by reflectance spectrophotometry and standard methods, respectively.

## 2. Experimental

### 2.1. Materials and equipment

Three types of non-ionic surfactants were prepared from Kimyagaran Emrooz Chemical Industries Co. The specifications of them are given in Table 1. The natural dyes of weld, madder and walnut husk were obtained

from the local market of Ardakan Yazd, and were used in the form of powder in the extraction process. Woolen yarn used in hand-woven carpets with specification of 200 Tex (4 fold) was obtained from Azarbarf (Iran). Distilled water was used as solvent in the extraction method. The absorption of colored solutions was measured using a spectrophotometer CECIL 9200 with two beams. Also, the colorimetric data of dyed samples were investigated by Hunter Lab Mini Scan EZ Model Number 4500 L reflective spectrophotometer.

## 2.2. Determination CMC of surfactants

The CMC concentration of the surfactants was measured by surface tension method (platinum ring method). For this purpose, different concentrations of each surfactant were prepared (0.01-1 g/L) and their surface tension was measured using Kruss K100MK2, Germany.

## 2.3. Extraction procedure

The extraction of natural dyes was performed through conventional method in the presence of non-ionic surfactants. First, 0.5 g of dried plant powder was extracted with non-ionic surfactant in the liquid ratio of 100:1 at boiling temperature for 2 h. Then, the colored solutions were filtered to remove biomass and their absorbance was measured with a spectrophotometer. The concentration values of surfactants were 0.15-0.25 g/L. Also, the prepared colorant solution without the presence of surfactants in the extraction bath was considered as a control sample to evaluate better the role of non-ionic surfactants.

## 2.4. Mordanting and dyeing method

Before dyeing, 5 g/L of non-ionic detergent was used to scour the wool yarn at 50 °C with L.R 40:1 for 30 min to remove surface impurities, then rinsed well and dried at ambient temperature.

Due to the low affinity of the natural dyes, the mordanting is essential for many dyeing of fibers. Furthermore, enhancement of the color fastness and colorimetric features is the advantage of mordanting [18]. Mordants are applied to the textile with three varied approaches including pre-mordanting, post-mordanting and on-mordanting. The pre-mordant technique is the most widely used and often gives the best results [19]. In this study, the pre-mordant procedure with alum (5 % o.w.f) was accomplished according to the graph that indicated in Figure 1. Then, the mordanted wool was dyed by using 25 % (o.w.f) of the obtained extracts with L.R 40:1. The dyeing graph is shown in Figure 1. Lastly, wool samples were washed, rinsed thoroughly and dried.

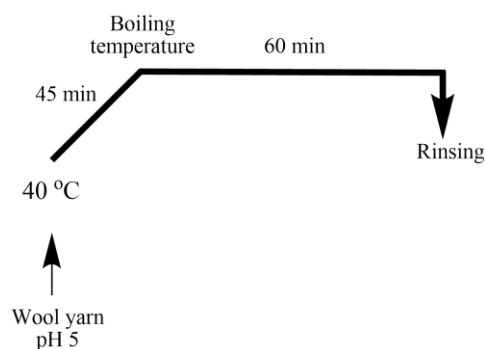


Figure 1: Mordanting and dyeing graph of woolen yarn.

Table 1: Characteristics of different non-ionic surfactants

Chemical name	Trade Name	CAS number	HLB value	Chemical structure
Sorbitan Ester Polyethylene Glycol Ether	Tween 20	9005-64-5	16-17	
Oleic Acid Polyethylene Glycol Ester	Keol 9	9004-96-0	11.6	
Nonylphenol polyethylene Glycol Ether	Kenon 10	9016-45-9	13.3-14	

The reflectance spectrophotometer was used to measure the colorimetric data ( $L^*$ ,  $a^*$ , and  $b^*$ ) of the CIELAB color system of dyed samples through Kubelka-Munk equation (Eq. 1). Where  $K$ ,  $S$  and  $R$  are the coefficient of the light absorption, the scattering coefficient, and the reflectance of the dyed samples, respectively [20, 21].

$$\frac{K}{S} = \frac{(1 - R)^2}{2R} \quad (1)$$

The reflectance data was measured by D65 illuminant and 10° standard observer from 400-700 nm with 10 nm intervals.

## 2.5. Color fastness

The standard of ISO 105-C06:2010 was used to determine the color fastness to wash of the dyed samples. This test was performed at 40 °C for 30 min in a water bath containing a standard detergent with L.R 50:1 and evaluated using the gray scale. Furthermore, the light fastness of the dyed samples was measured by the standard of ISO 105-B02:2013. Partial of the prepared samples were covered to protect them from the light and placed subject to Xenon arc light fastness tester. Then, the difference between two parts of the sample (exposed to light and unexposed to light) was evaluated using the blue scale.

## 3. Results and Discussion

### 3.1. Measuring the CMC of surfactants

Surfactants play a crucial role in enhancing the wetting, dissolution, and dispersion of dyes, and the CMC is a significant parameter that greatly impacts surfactant efficiency. At low concentrations, surfactant molecules and chains exist in a dispersed state, leading to a reduction in surface tension. As surfactant concentration increases, micelle formation occurs, which leads to further decreases in surface tension. When a surfactant

concentration is much less than CMC, the molecular form of monomers exist in however as it increases near to the CMC, small aggregates of monomers ( $n = 2, 3$  and  $4.$ ) begin [22]. Table 2 provides information on the surface tension and CMC of the surfactants. Results showed that Keol, with a CMC value of 0.15 g/L, was more effective at reducing surface tension than Kenon and Tween.

The activities of surfactant above the CMC can be different depending on the surfactant concentration, type, temperature, and pH. In general, the surface tension of the solution tends to remain constant with further increases in concentration. At high concentrations, the number of micelles in the solution reaches a maximum, and additional surfactant molecules may not have a significant effect on the surface tension. The surface tension may also start to increase at very high concentrations, as the surfactant molecules become more tightly packed and can no longer effectively reduce the surface tension.

The higher  $\gamma_{CMC}$  value of Tween compared to Keol suggests that Tween has a stronger tendency to remain as individual molecules in solution, rather than forming micelles. This could have implications for the effectiveness of Tween in certain applications, such as in emulsification or solubilization, where micelle formation is critical. It is important to note that other parameters, for instance the molecular weight, chemical structure, and solubility of the surfactants, can also influence their ability to reduce surface tension [23].

Furthermore, according to the Hydrophilic-Lipophilic Balance (HLB) values reported in Table 1, it can be suggested that Tween has more hydrophilic characteristics in comparison to Keol and Kenon. The HLB is a measure that determines the balance between the hydrophilic and lipophilic properties of surfactants, with higher values signifying greater hydrophilicity [24]. Tween has a higher HLB value than Keol and Kenon, indicating a greater attraction to water.

**Table 2:** The values of CMC and  $\gamma_{CMC}$  of the surfactants by surface tension measurement.

Surfactant	CMC (g/L)	$\gamma_{CMC}$ (mN/m)	Cloud point (°C)
Keol	0.15±0.05	33.16	64-67
Kenon	0.19±0.05	31.82	66-71
Tween	0.25±0.05	37.57	76

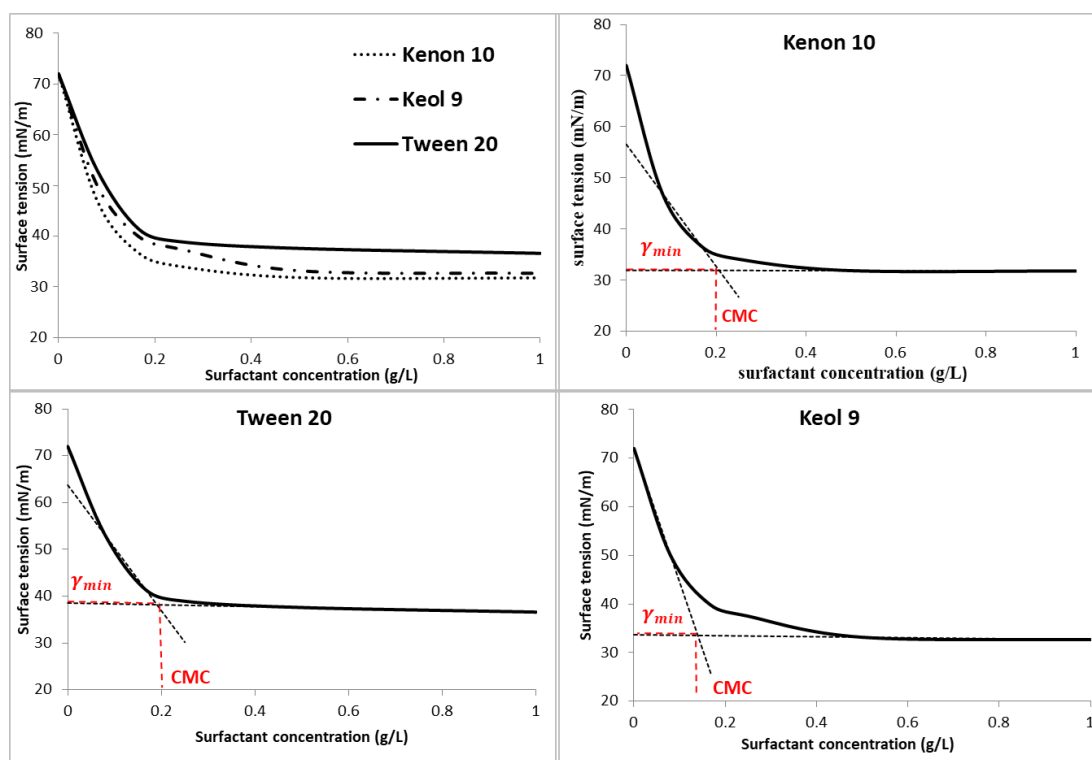


Figure 2: CMC estimation by plotting surface tension versus varied concentration of surfactant solutions.

### 3.2. Extraction of natural dyes

The forming of micelles in surfactants play a crucial role in extraction of colorant compounds in natural dyes and the more solubilization of them by reducing the surface tension of the solution. The formation of micelles provides a favorable environment for the solubilization of hydrophobic dye molecules, allowing them to be more easily extracted from the solid phase into the liquid phase. The presence of surfactant micelles in the extraction solution can increase the efficiency of dye extraction, as more dye molecules are solubilized and extracted from the solid material.

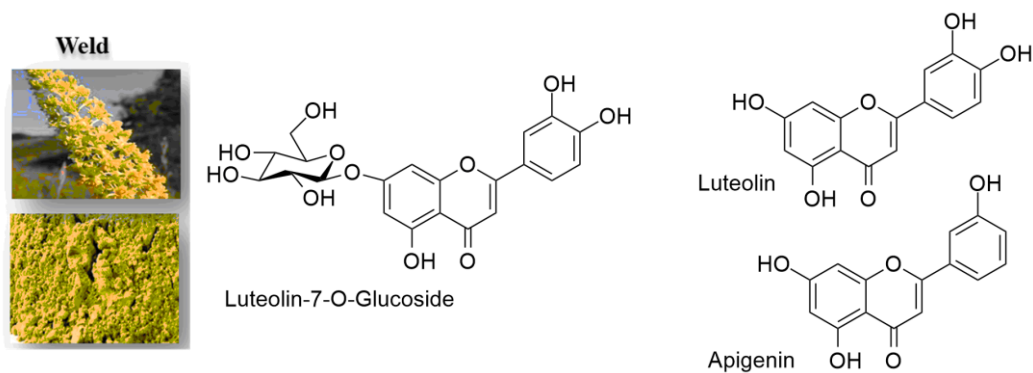
#### 3.2.1. Weld

Weld is a plant with the scientific name *Reseda luteola*. It is a member of the *Resedaceae* family and is native to Europe and Asia. The plant has been historically used for its yellow dye, which is extracted from its leaves and stems. The extracted colorants are introduced in the color index as Natural Yellow 2, C.I. 75590 and 75580 [4]. The main ingredients in the weld natural dye are luteolin and apigenin, which belong to the flavonoid group (Figure 3) [5, 10]. Luteolin-7-O-Glucoside is more soluble in water than Luteolin because of the additional glucoside group which increases its polarity

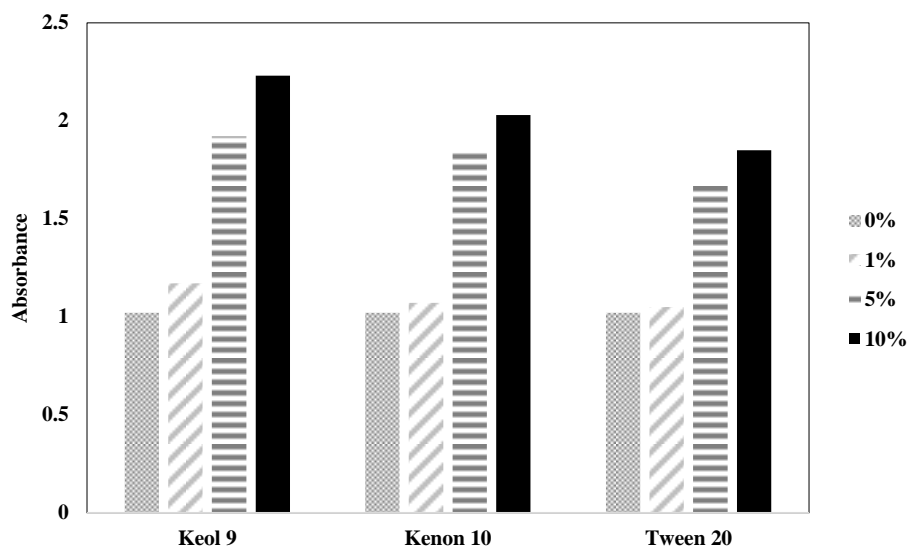
and enhances its interaction with water molecules. The glucoside moiety provides additional hydrophilic (water-loving) functional groups that increase the overall solubility of the molecule in water. As a result, Luteolin-7-O-Glucoside can dissolve more readily in aqueous solutions than Luteolin, which lacks the glucoside moiety.

Two main absorption bands is observed in the UV-Vis spectrum of the most flavonoids, that related to benzoyl and cinnamoyl bands in the ranges of 240-280 and 320-385 nm, respectively [10]. According to the studies, the main flavonoids in the weld have absorption in the wavelength range of 350 nm and before the visible range [4, 9]. The colorants with glycoside moieties are readily extracted in water.

The data presented in Figure 4 demonstrates the absorbance of extracted dye solutions at the wavelength of maximum absorption ( $\lambda_{max}$ ) when using three different concentrations of surfactants, in comparison to conventional dye extraction without any surfactant. The results clearly show that the use of surfactants significantly improves the extraction of dye, with much higher levels achieved than without the presence of surfactants. These surfactants can extract and solubilize hydrophobic dye species in their micelles and increase the overall dye extraction efficiency.



**Figure 3:** Chemical structure of the weld main flavonoids, luteolin and apigenin.



**Figure 4:** Absorbance spectra of extracted weld with different surfactants at various concentrations (0-10 %) over weight of dye powder by conventional method at  $\lambda_{\max}$  390 nm (the extracted solution was diluted one-tenth).

The use of Keol resulted in noticeably higher dye extraction than the other two surfactants, Kenon and Tween. This suggests that Keol, with its lower critical micelle concentration (CMC),  $\gamma_{\text{CMC}}$ , and HLB values, is able to extract dye molecules more efficiently. Keol is also more hydrophobic than the other surfactants, and consequently has the more potential to decrease the surface tension and dissolving of hydrophobic dye molecules.

Absorbance spectra of extracted weld with different surfactants at various concentrations is shown in Figure 4. Results indicated that surfactants can improve the absorbance value of extracted solution. Also, the dye extraction in the presence of Keol was noticeably higher than those by Kenon and Tween. It seems Keol with lower CMC,  $\gamma_{\text{CMC}}$  and HLB values, can extract the dye molecules with the more efficiently. Keol with higher

hydrophobicity can probably accommodate hydrophobic dye species more efficiently in its micelles than the other two surfactants.

### 3.2.2. Madder

Madder, with the scientific name *Rubia tinctorum*, has been utilized for textile dyeing since ancient times. Its roots contain a natural red dye called alizarin, as well as other colorants like purpurin and munjistin, which are complex anthraquinone hydroxyl derivatives (Figure 5) [25]. The color of the dye extracted from madder can vary from pale pink to deep red depending on the extraction process and the type of madder plant used. Additionally, madder has been used for medicinal purposes and has been found to have anti-inflammatory and anti-cancer properties [11]. The madder root contains 2 to 3.5 % di-trihydroxy-

anthraquinone glycosides, also the alizarin content in madder root can vary between 6.1 to 11.8 mg/g [6]. The most important anthraquinone structure that is present in madder root and causes the red color is alizarin and purpurin [8]. Alizarin is considered as the foremost ingredient of madder dye, and madder extract has been used for dyeing cotton, linen, wool and silk in the past [26].

The absorption spectrum of madder dye indicates the keto or quinone group in the anthraquinone structure in the range of 380-390 nm. Furthermore, distinct peaks and in some instances, multiple peaks in the visible range indicate the several color component in the extract solution [13].

The graph in Figure 6 depicts the absorbance of extracted madder solutions at the wavelength where the absorption is maximum ( $\lambda_{max}$ ) when using three different surfactants. In general, the presence of surfactants leads to a higher amount of dye extraction. Among the three surfactants used, Keol resulted in

slightly higher dye extraction compared to Kenon and Tween.

### 3.2.3. Walnut husk

Walnut husk, scientifically known as *Juglans regia*, is a species of walnut tree that grows worldwide with over 15 different types. The husk of the walnut fruit contains numerous bioactive polyphenolic compounds (Figure 7), including gallic acid, caffeic acid, quercetin, myrcetin, and juglone, which is the main colorant in walnut husk. The natural colorants extracted from Walnut husk are commonly used for dyeing textiles, paper, and leather. The dye compounds are extracted from the husks through boiling in water or soaking in alcohol, and its color ranges from light to dark brown, depending on the concentration and extraction method used. Walnut husk dye has been used for centuries and is still popular today as an eco-friendly alternative to synthetic dyes [27, 28].

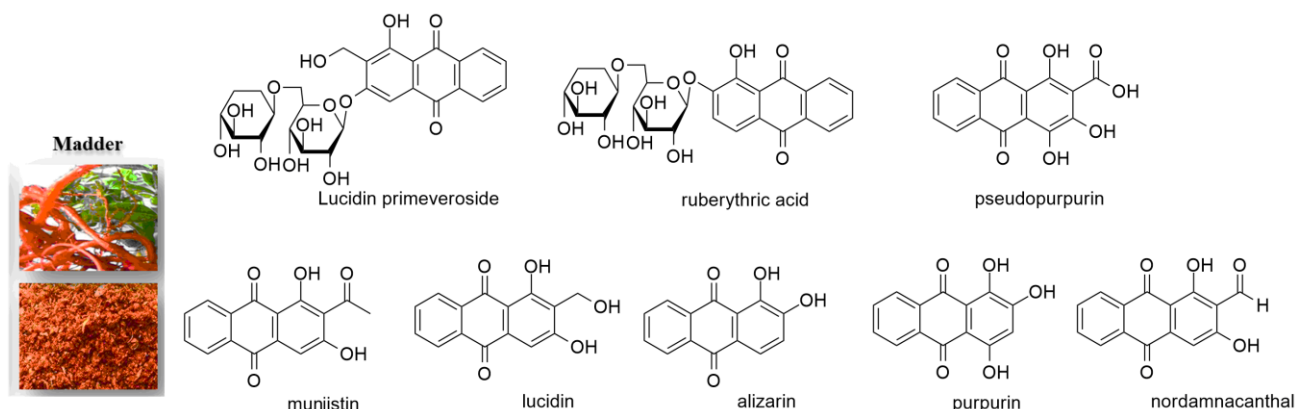


Figure 5: Chemical structures of compounds extracted from Madder.

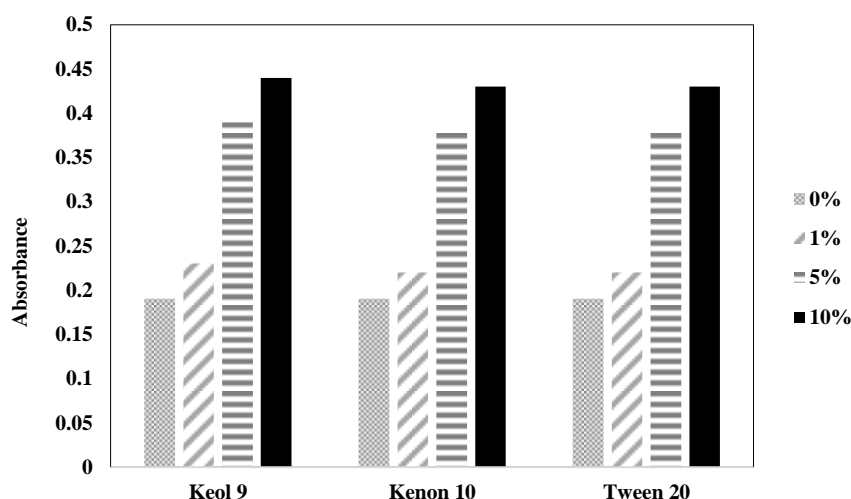
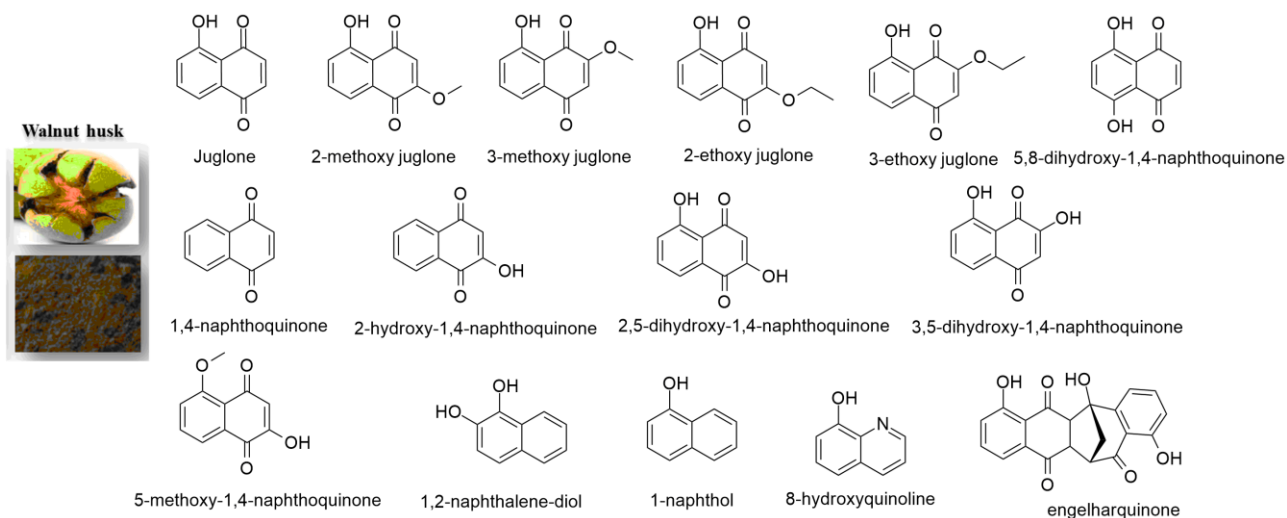


Figure 6: Absorbance spectra of extracted madder (at  $\lambda_{max}$  = 420 nm) with different surfactants at various concentrations (0-10 %) over weight of dye powder by conventional method (the extracted solution was diluted one-tenth).



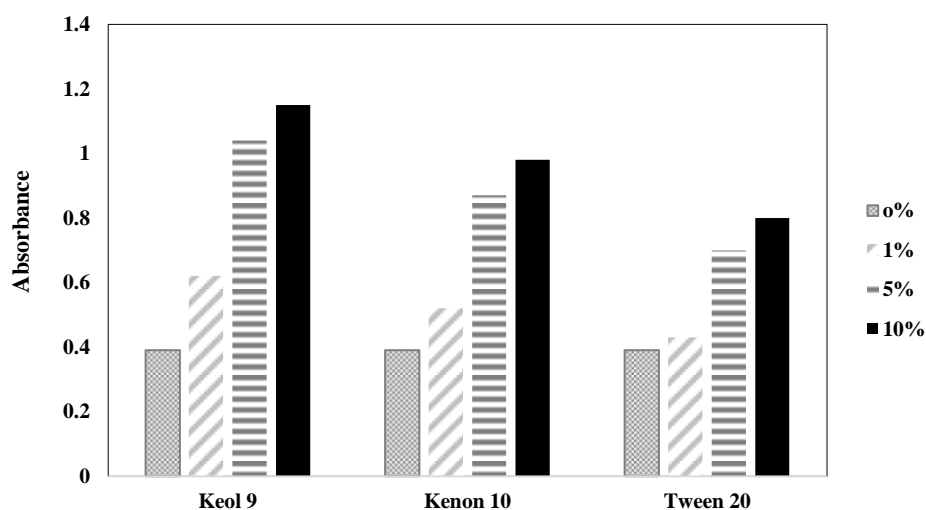


**Figure 7:** Chemical structures of the compounds extracted from walnut husk.

Figure 8 displays a graph illustrating the absorbance of dye solutions extracted from walnut husks at the wavelength of maximum absorption ( $\lambda_{\max}$ ) using three different surfactants. Generally, the presence of surfactants increases the amount of dye extraction. The order of dye extraction among the surfactants used is as follows: Keol > Kenon > Tween. This indicates that Keol, a more hydrophobic surfactant with lower CMC and HLB values, is more efficient in extracting dyes from walnut husks than the other two surfactants.

We summarized the results of Figures 4, 6, and 8 in Table 3. The chemical structure of the surfactants is the significant factor in the extraction efficiency of natural dyes. The ability to dissolve insoluble dyes in water

increases with higher concentrations of micelle formation, which in turn is influenced by the length of the hydrophobic chain and the hydrophilic oxyethylene chain (Table 1). Interestingly, the noticeable shift in the  $\lambda_{\max}$  of extracted weld by using different surfactant was seen. It could be due to the more varied hydrophobic components (flavonoids) in the chemical structure of the weld. Therefore, it was more affected by the presence of surfactants and variation of surface tension. Surfactants do not show any visible peaks (Absorbance: 0.0), therefore, they have no interaction with natural colorants. Also, the presence of non-ionic surfactants does not change the  $\lambda_{\max}$  of the colored solutions.



**Figure 8:** Absorbance spectra of extracted walnut husks with different surfactants at various concentrations (0-10 %) over weight of dye powder by conventional method  $\lambda_{\max} = 370$  nm (the extracted solution was diluted one-tenth).



Table 3: Summary of UV-Vis measurements.

Natural dye	Surfactant	$\lambda_{\max}$ (nm)	Absorbance at surfactant concentrations			
			0 %	1 %	5 %	10 %
Weld	Keol	400	1.02	1.17	1.92	2.23
	Kenon	395	1.02	1.07	1.85	2.03
	Tween	388	1.02	1.05	1.68	1.85
Madder	Keol	420	0.19	0.23	0.39	0.44
	Kenon	419	0.19	0.22	0.38	0.43
	Tween	417	0.19	0.22	0.38	0.43
Walnut	Keol	370	0.39	0.62	1.04	1.15
	Kenon	370	0.39	0.52	0.87	0.98
	Tween	370	0.39	0.43	0.70	0.80

### 3.3. Wool dyeing with natural dye extracts

Table 4 presents colorimetric data for wool samples that were dyed using different natural dyes extracted through conventional methods, both with and without the addition of various surfactants. The presence of surfactants led to a substantial increase in K/S values of samples up to 40 %. The ascending order of K/S values for dyed wool was as follows: no-surfactant, Keol, Kenon, and Tween.

The reason for the higher absorption of extracted dyes on the wool in the presence of surfactants can be attributed to several factors. Firstly, the surfactant micelles increase the dye molecules by extracting more dye species. Secondly, the surfactants create a more favorable environment for the dye molecules to absorb and diffuse into the fibers. The surfactants can also attach to the surface of wool fiber and decrease the interfacial surface tension between dye molecules and fiber chains, which facilitates the dyeing process. Lastly, the surfactants can break down and disperse impurities, such as lipids and proteins that may hinder the dye extraction process.

Interestingly, this finding contradicts the expected trend based on the extraction percentage of dye molecules in the presence of nonionic surfactants. It was found that Keol, with its high extraction capacity, hindered the dye adsorption on wool fibers by trapping the dye molecules in its micelles. Therefore, surfactants with very high dye solubilization capacity can slow down the dye adsorption process. Tween micelles can release the solubilized/dispersed dye molecules more readily during the dyeing process which enhances the

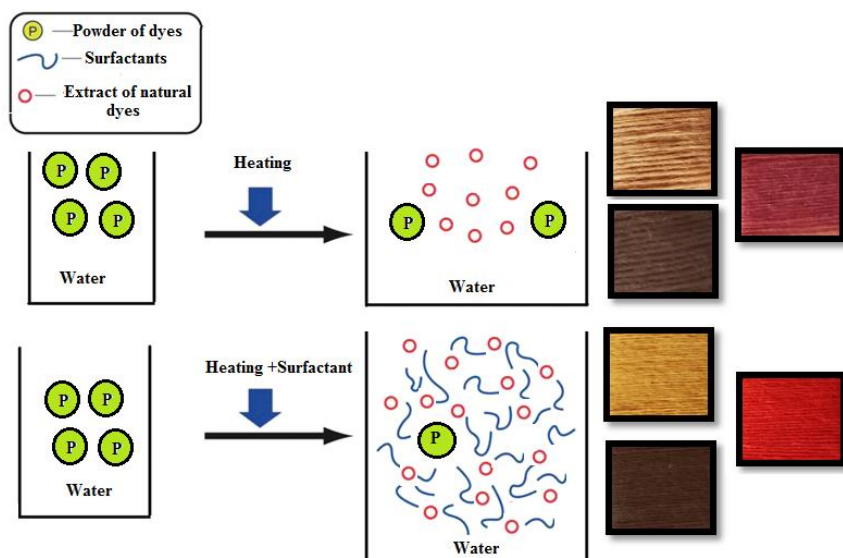
dye adsorption on wool fibers.

The cloud point of Tween 20 occurs at approximately 75 °C, and surpassing this temperature induces the formation of larger surfactant micelles and aggregates. This temperature-induced increase in micelle size significantly enhances their capacity for solubilizing hydrophobic ingredients in colorants. By expanding the size of micelles at elevated temperatures, there is a substantial improvement in their ability to dissolve and accommodate hydrophobic ingredients in colorants [17, 30].

Overall, the extraction efficiency and dyeing process of natural dyes on wool fibers can enhance in the presence of surfactants. It is important to note that the optimal type and concentration of surfactants may vary depending on the specific dye and fiber types being used, and further research is needed to determine the most effective surfactant for different natural dyes on the wool samples. Schematically, the performance of the surfactants on the extraction of natural dyes and dyeing of wool yarn with them is shown in Figure 9.

All three types of non-ionic surfactants can improve the extraction of natural colorants from weld, madder and walnut husk. As a result, dyeing of wool yarns at the higher concentration of colorant has increased the color strength with more uniformity.

After conducting experiments on wool yarns dyed with various dye extracts in the presence of different nonionic surfactants, it was observed that the dye absorption of the wool yarns in presence of Tween was better than the other two nonionic surfactants tested. Therefore, we have decided to focus on using Tween in our future experiments.



**Figure 9:** The schematic representation of the performance of the surfactants on the extraction and dyeing of wool yarn with natural dyes.

**Table 4:** The colorimetric data of dyed samples with extracted weld, madder and walnut husks in the presence of surfactants (10 %)

Natural dye	Surfactant	L*	a*	b*	$\Delta E$	C*	K/S	K/S Enhancement (%)
Weld	No	70.18	-0.1	34.32	34.33	90.17	17.61	-
	Keol	69.17	-0.22	34.56	34.56	90.37	18.83	6.47
	Kenon	69.2	-0.09	34.04	34.04	90.15	19.13	7.94
	Tween	68.98	0.26	34.12	34.12	89.57	19.99	11.90
Madder	No	45.46	27.72	14.57	31.31	27.73	4.15	-
	Keol	45.56	27.72	15.69	31.85	29.5	5.10	18.62
	Kenon	44.02	28.54	15.75	32.59	28.9	5.72	27.44
	Tween	43.48	28.51	16.07	32.72	29.41	5.91	29.78
Walnut	No	49.53	8.23	15.39	17.46	61.87	3.86	-
	Keol	49.05	8.29	15.85	17.89	62.4	5.06	23.71
	Kenon	48.84	8.77	16.55	18.73	62.07	5.40	28.51
	Tween	48.22	8.47	16.21	18.29	62.4	5.41	28.65

Table 5 shows the colorimetric data of wool yarns that were per-mordanted and dyed with alum and extracted natural dyes in the absence and presence of Tween. It was observed that the addition of Tween during the extraction of dyes increased the chroma and absorption of dyes on the wool yarns.

Alum is a common mordant used for wool, which helps in fixing the dye onto the fiber and improving color fastness. When wool is treated with alum, the

alum molecules bind with the fiber and create a base for the dye molecules to attach. This results in a more even and durable dyeing process. Mordanting wool with alum leads to a shift in the color coordinates, especially the L\* value. The L\* value of mordanted wool is generally lower than that of untreated wool, meaning that it appears darker. This is because the alum can cause the wool fibers to absorb more dye, leading to a darker color. a\* and b\* coordinates are


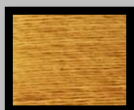
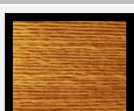
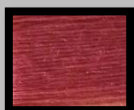
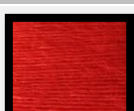
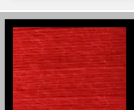
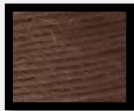
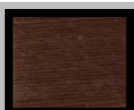
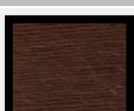
also change by mordanting, related to the type of dye used (Table 5). For example, when wool is mordanted with alum and then dyed with Weld, the b\* coordinate increase, indicating a more yellow hue.

### 3.3.1. Color fastness properties

The durability of dyed textiles is important because these fabrics are often subjected to repeated washing and

exposure to light, which can cause them to fade or lose their color over time. Evaluating the fastness features of these textiles is crucial to ensuring that they maintain their appearance and quality over time. Over time, repeated washing can cause the dyes to fade or bleed, leading to a loss of color and pattern intensity. This can be particularly problematic in textiles that are frequently washed, such as clothing, towels, and bed linens.

**Table 5:** Colorimetric data of pre-mordanted wool yarns dyed with extracted dyes (Al 5 % o.w.f and Tween 10 %).

Dye	Sample	Surfactant	L*	a*	b*	C*	h°	K/S	Picture
Weld	Non mordanted wool	No	70.18	-0.1	34.32	90.17	89.83	17.61	
	Mordanted wool	No	69.89	0.61	61.6	61.6	89.43	20.86	
	Mordanted wool	Tween 20	69.09	0.97	63.11	63.12	89.12	24.79	
Madder	Non mordanted wool -Madder	No	45.46	27.72	14.57	27.73	27.72	5.15	
	Mordanted wool-Madder	No	42.09	38.66	26.98	47.14	34.91	6.02	
	Mordanted wool-Madder	Tween 20	45.24	36.23	25.5	44.31	35.13	6.92	
Walnut husk	Non mordanted wool	No	49.53	8.23	15.39	61.87	-	4.86	
	Mordanted wool	No	42.58	9.40	17.7	20.04	62.04	5.42	
	Mordanted wool	Tween 20	42.19	10.03	18.91	21.41	62.06	6.81	

**Table 6:** Washing and light fastness of pre-mordanted wool yarns dyed with extracted dyes (AI 5 % o.w.f and Tween 10 %).

Dye	Sample	Washing fastness			Light fastness
		Staining on cotton	Staining on wool	Color change	
Weld	Non mordanted wool	4-5	4	3	4-5
	Mordanted wool	5	4-5	3-4	5
	Mordanted wool+Tween	5	4-5	3-4	5
Madder	Non mordanted wool	4-5	3-4	3-4	5-6
	Mordanted wool	5	4-5	4	6
	Mordanted wool+Tween	5	4-5	4	6
Walnut husk	Non mordanted wool	4-5	4	4	6
	Mordanted wool	5	5	4	6-7
	Mordanted wool+Tween	5	5	5	6-7

In addition to washing, exposure to light can also have an impact on the durability of dyed textiles. Ultraviolet (UV) light, for example, can cause some dyes to break down and fade over time. Therefore, many textiles that are used outdoors or in sunny areas, such as upholstery fabrics or outdoor clothing, need to be treated with UV-resistant coatings to help preserve their color [29].

The color fastness to light and wash of dyed samples with extracted natural dyes in presence of Tween is given in Table 6. The use of surfactant can improve the dye absorption on the wool fibers, but did not have a negative effect on the fastness data. The improved dye absorption resulted in better overall fastness, and there was no evidence of agglomeration of dye molecules on the surface of fibers that would have decreased the fastness ratings of samples after washing.

#### 4. Conclusion

This study provides valuable insights into the potential application of non-ionic surfactants for the extraction and dyeing of wool yarn with natural dyes through conventional method. The results demonstrate that the use of non-ionic surfactants can improve the absorbance intensity, which is directly linked to higher dye extraction percentages. The higher dye absorption in the

solution containing of surfactants can be related to the ability of surfactants to reduce surface tension of the extraction bath. The surfactants increase the solubility of natural dyes in water, allowing for more colorant to be dissolved from the solid phase into the liquid phase. This phenomenon leads to an increase in absorbance intensity and ultimately results in higher dye extraction efficiency and color strength on the wool yarn. The fastness properties of dyed samples showed that non-ionic surfactants have no adverse effect on the color fastness of the dyed wool yarn. The findings of this study open avenues for future research and practical applications in the field of natural dyeing of wool yarn. The successful use of non-ionic surfactants in the conventional extraction and dyeing process highlights a promising direction for enhancing color extraction efficiency and color strength. Further exploration could focus on optimizing the surfactant concentrations and exploring their compatibility with various natural dyes to maximize the benefits.

#### Acknowledgments

This work has been supported by the Center for International Scientific Studies and Collaborations (CISSC), Ministry of Science, Research and Technology of Iran.

## 5. References

- Adeel S, Rehman FU, Rafi S, Zia KM, Zuber M. Environmentally Friendly Plant-Based Natural Dyes: Extraction Methodology and Applications. In: Ozturk, M., Hakeem, K. (eds) Plant and Human Health, 2019, Volume 2. Springer, Cham. <https://doi.org/10.1007/978-3-030-03344-6-17>.
- Bairabathina V, Shanmugam KSK, Chilukoti GR, Ponnamm V, Raju G, Chidambaram P, A review on reverse micellar approach for natural fiber dyeing. *Color. Technol.* 2022; 138: 329-341, <https://doi.org/10.1111/cote.12605>.
- Salaudiddin MSK, Rony MIA, Haque MA, Shamim AM, Review on extraction and application of natural dyes. *Text Leather Rev.* 2021; 4: 218-233, doi: 10.31881/TLR.2021.09.
- Ozan D, Recep K. A review on the flavonoids-a dye source. *Int J Adv Eng Pure Sci.* 2019; 3: 188-120, <https://doi.org/10.7240/jeps.476514>.
- Clark M, editor. Handbook of textile and industrial dyeing: principles, processes and types of dyes. Elsevier; 2011 Oct 25.
- Yusuf M, Shabbir M, Mohammad F, Natural colourants: historical, processing and sustainable prospects. *Nat. Prod. Bioprospect.* 2017; 7: 123-145, <https://doi.org/10.1007/s13659-017-0119-9>.
- Mehrparvar L, Safapour S, Sadeghi-Kiakhani M, Gharanjig K. A cleaner and eco-benign process for wool dyeing with madder. *Rubia tinctorum L.*, root natural dye. *Int J Environ Sci Technol.* 2016; 13: 2569-2578, <https://doi.org/10.1007/s13762-016-1060-x>.
- Haji A. Dyeing of cotton fabric with natural dyes improved by mordants and plasma treatment. *Prog Color Colorant Coatings.* 2019; 12: 191-201, <https://doi.org/10.30509/PCCC.2019.81586>.
- Thomas B, Mussak R. Handbook of natural colorants. Wiley, 2009 Chichester, doi:10.1002/9780470744970.
- Vankar PS, Shukla D. Spectrum of colors from reseda luteola and other natural yellow dyes. *J Text Eng Fashion Technol.* 2018; 4: 106-119, <https://doi.org/10.15406/jteft.2018.04.00127>.
- Jiang HY, Hu XD, Zhu JJ, Wan J, Yao JB. Studies on the photofading of alizarin, the main component of madder. *Dyes Pigm.* 2021; 185: 108940, <https://doi.org/10.1016/j.dyepig.2020.108940>.
- Schmidt-Przewozna K. Green Sustainable Textile Supercritical Dyeing Process Using CO<sub>2</sub> Madder (*Rubia tinctorum L.*) Extract. *J Nat Fibers.* 2023; 20:2,<https://doi.org/10.1080/15440478.2023.2277836>.
- Pizzicato B, Pacifico S, Cayuela D, Mijas G, Ribamolinier M. Advancements in sustainable natural dyes for textile applications: A Review. *Molecules.* 2023; 28: 5954. <https://doi.org/10.3390/molecules28165954>.
- Shaban SM, Kang J, Kim DH. Surfactants: Recent advances and their applications. *Compos Commun* 2020; 22: 100537, <https://doi.org/10.1016/j.coco.2020.100537>.
- Febriana ID, Kusuma HS, Gala S, Mahfud M. The effect of temperature on extraction of Swietenia mahagoni by ultrasound-assisted extraction (UAE) method. *ASEAN J Chem Eng.* 2016; 16: 44-49, <https://doi.org/10.22146/ajche.49673>.
- Sadeghi-Kiakhani M., Gharanjig K. Study of the influence of gemini cationic surfactants on the dyeing and fastness properties of polyester fabrics using naphthalimide dyes. *J Surfactants Deterg.* 2015; 18: 47-54, <https://doi.org/10.1007/s11743-014-1622-1>.
- Tehrani-Bagha AR, Holmberg K. Solubilization of hydrophobic dyes in surfactant solutions. *Mater.* 2013; 6(2): 580-608; <https://doi.org/10.3390/ma6020580>.
- Hosseinnezhad M, Gharanjig K, Jafari R, Imani H, Razani N. Cleaner colorant extraction and environmentally wool dyeing using oak as eco-friendly mordant, *Environ Sci Pollut Res.* 2021; 28: 7249-7260,<https://doi.org/10.1007/s11356-020-11041-2>.
- Ghaheh FS, Moghaddam MK, Tehrani M. Comparison of the effect of metal mordants and biomordants on the colorimetric and antibacterial properties of natural dyes on cotton fabric. *Color Technol.* 2021; 137: 689-698, <https://doi.org/10.1111/cote.12569>.
- Sadeghi-Kiakhani M, Tehrani-Bagha AR, Safapour S, Eshaghloo-Galugahi S, Etezad SM. Ultrasound-assisted extraction of natural dyes from Hawthorn fruits for dyeing polyamide fabric and study its fastness, antimicrobial, and antioxidant properties. *Environ Dev Sustain.* 2021; 23: 9163-9180, <https://doi.org/10.1007/s10668-020-01017-0>.
- Sadeghi-Kiakhani M, Gharanjig K, Arami M. Study on dyeing and fastness properties of wool-polyester blend fabrics using novel mono azo-naphthalimide dyes. *J Text Inst.* 2014; 105: 52-58, <https://doi.org/10.1080/00405000.2013.810020>.
- Ghosh DC, Sen PK, Pal B. Dye-surfactant interaction in aqueous premicellar and micellar environments in the alkaline fading of di-positive methyl green carbocation. *Colloids Surfaces A Physicochem. Eng Asp.* 2023; 666: 131300, <https://doi.org/10.1016/J.COLSURFA.2023.131300>.
- Holmberg K, Jonsson B, Kronberg B, Lindman B. Surfactants and Polymers in Aqueous Solution. 2003; 2<sup>nd</sup> Edition, Wiley, New York.
- Dave N, Joshi T. A Concise Review on Surfactants and Its Significance. *Int J Appl Chem.* 2017; 13: 663-672, <https://doi.org/10.37622/ijac/13.3.2017.663-672>.
- Willemsen H, Meijdenberg GJP, Beek TA, Derksen GCH. Comparison of madder (*Rubia tinctorum L.*) and weld (*Reseda luteola L.*) total extracts and their individual dye compounds with regard to their dyeing behaviour, colour, and stability towards light. *Color*

- Technol. 2019; 135: 40-47, <https://doi.org/10.1111/cote.12384>.
26. Abdel-Hamied M, Hassan RRA, Salem MZM. et al. Potential effects of nano-cellulose and nano-silica/polyvinyl alcohol nanocomposites in the strengthening of dyed paper manuscripts with madder: an experimental study. *Sci Rep.* 2022; 12: 19617. <https://doi.org/10.1038/s41598-022-23907-1>.
27. Arifeen W, Rehman FU, Adeel S, Zuber M, Ahmad MN, Ahmad T. Environmental friendly extraction of walnut bark-based juglone natural colorant for dyeing studies of wool fabric. *Environ Sci Pollut Res.* 2021; 28: 49958-49966, <https://doi.org/10.1007/s11356-021-14277-8>.
28. Ramezani N, Raji F, Rezakazemi M, Younas M. Juglone extraction from walnut (*Juglans regia L.*) green husk by supercritical CO<sub>2</sub>: Process optimization using Taguchi method. *J Environ Chem Eng.* 2020; 8: 103776, <https://doi.org/10.1016/j.jece.2020.103776>.
29. Taghizadeh borujeni R, Akbari A, Gharehbaii A, Yunessnia A. Extraction and preparation of dye powders from *Reseda luteola L.* using membrane processes and its dyeing properties. *Environ Technol Innov.* 2021; 21: 101249, <https://doi.org/10.1016/j.eti.2020.101249>.
30. Tehrani-Bagha AR, Singh RG, Holmberg K. Solubilization of two organic dyes by cationic ester-containing gemini surfactants. *J Colloid Int Sci.* 2012; 376: 112-118, <https://doi.org/10.1016/j.jcis.2012.02.016>.

How to cite this article:

Sadeghi-Kiakhani M, Tehrani-Bagha AR, Aladpoosh R, Hashemi E. Application of Different Non-ionic Surfactants on the Conventional Extraction and Dyeing Characteristics of Natural Dyes on the Wool Yarn. *Prog Color Colorants Coat.* 2024;17(3):275-288. <https://doi.org/10.30509/pccc.2024.167223.1258>.

