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INVESTIGATION OF THE EFFECT OF THE DYEING METHOD ON THE DYEING PROPERTIES OF SILK FABRIC DYED WITH NATURAL DYE

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Abstract

In this study, silk fabric was dyed with the natural dye extracted from blueberry (Vaccinium corymbosum L.) via ultrasonic, microwave and conventional methods. One of the samples dyed via conventional method was treated oxygen plasma treatment whereas the other was mordanted with aluminium potassium sulphate prior to the dyeing process. The colour and fastness properties of the dyed fabrics were investigated and compared with each other in terms of dyeing method. The physical structure of samples was analysed with scanning electron microscope (SEM). The chemical structure of samples was characterized by Fourier transform infrared spectroscopy (FTIR ATR). According to the results, the samples dyed via microwave method had the best results in terms of colour properties. Furthermore, when compared with one another in terms of dyeing methods, there were no significant differences between fastness properties of dyed samples.

Key words: Vaccinium Corymbosum L., Ultrasonic Method, Microwave Method, Conventional Method, Plasma Treatment, Fastness Properties.

1. INTRODUCTION

In textile dyeing industry, in comparison to natural dyes, synthetic dyes have been prevalently used due to their lower prices, a wide range of colour and better fastness properties [1,2]. However, one should have regard to the significant though often overlooked issue which synthetic dyes are toxic, carcinogenic, non-biodegradable [3]. In addition, synthetic dyes cause environmental pollution and waste water problem[3]. Recently, the dye industry has been endeavouring to reduce toxic effluents and to stop the production of potentially hazardous dyes or pigments so as to prevent global warming. As a result, the interest in the use of natural dyes in textile colouration revives as natural dyes are biodegradable, non-toxic and eco-friendly, and that they do not cause health problems, environmental pollution and waste water problem [4-7]. The pigments which have potential use in textile dyeing industries are produced from plants such as Hibiscus mutabilis rose, Lawsonina Inermis L., Indigofera tinctoria, Rubia tinctorum, Rubia cordifolia, Anchusa tinctoria, Salix negra etc.[8,9]. In the textile dyeing process, it is necessary that the auxiliaries, chemicals and the dyes used to dye textile materials should be eco-friendly in nature, and the dyeing methods adopted should lead to conservation of water, energy, and chemicals.

The textile industries have been researching for new and alternative technologies to meet both the quality and eco-friendly production [10]. Plasma technology, using ultrasonic and microwave energy, enzymatic process are the new and alternative technologies used in the textile industry. Plasma technology, innovatively applied to textile material as a pre-treatment, causes a variety of surface modifications of textile materials. It also improves a wide range of textile properties such as hydrophobicity, dye exhaustion, and adhesion [11]. In addition, plasma technology, as a clean, ecologic and dry technique, is characterized by low consumption of water, energy and chemicals [12-14]. Ultrasonic energy results in occurrence of the dyeing process at low temperatures, which, in turn, optimizes the use of heat energy. However, the materials dyed via conventional method gave better results than those dyed via ultrasonic method in terms of the fastness and colour retention [15,16]. Another technology for conserving energy is microwave technique using in the textile wet process. In this process, microwave energy is used to heat the liquor and microwave radiation provides more uniform heating in the liquor as opposed to the heating via conventional method. Whilst the outer wall heats up first in conventional method, the center of the liquor heats up first in microwave method. Subsequently, each point of solvent equally warms up. As such, the temperature of the liquor increases faster compared to conventional method. Thus, as an alternative method, microwave energy decreases energy consumption, and saves time in comparison with the conventional method [17-19].

In this study, silk fabric was dyed with the natural dye extracted from blueberry via ultrasonic, microwave, and conventional methods. One of the samples dyed via conventional method was also treated oxygen plasma application whereas the other was mordanted with aluminium potassium sulphate prior to the dyeing process. Then the colour and fastness properties were investigated with regard to dyeing process.

2. MATERIAL AND METHODS

2.1. Materials

In this study, plain weaved silk fabric with the weight of 80 g/m^2 was dyed with the natural dye which is of anthocyanins chemical structure (Figure 1) extracted from *Vaccinium Corymbosum.* The compounds of anthocyanins are given in the Table 1.

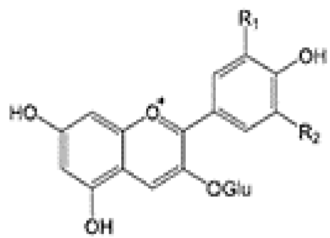


Figure 1. Chemical structure of anthocyanins [20]

Blueberry fruits were picked from the region of Black Sea. After, the fruits were cleaned and 1000 grams fruit and 20 l soft water were blended. This blend was boiled for an hour. Lastly, after the blend was refrigerated, the blend was filtered and the dyes fluid was extracted.

Table 1. The compounds of anthocyanins [20]

Compound name	Substituents		Ingredients (%)
	R_1	R_2	
Meleidin-3-glucoside	OMe	OMe	49
Peonidin-3- glucoside	OMe	H	20
Petunidin-3- glucoside	OMe	OH	15
Delphinidin-3- glucoside	OH	OH	13
Cyanidin-3- glucoside	OH	H	3

2.2. Plasma Treatment

The plasma treatment of silk fabric was carried out with oxygen gas in Diener vacuum plasma. The sample was placed onto the anode with the pressure of the chamber at 0.3 mbar and the plasma treatment was performed with low frequency, at 40 kHz, at a power of 100 W, for 5 minutes.

2.3. The Mordanting Process

The mordant solution was prepared by 10 grams of aluminium potassium sulphate was dissolved in 1000 ml distilled water. Then the fabrics were treated at the liquor ratio of 1:50 with this solution for one hour at the boiling temperature about 98o C.

2.4. Dyeing Process

In this study, the samples were dyed with the blueberry extracts via conventional, ultrasonic and microwave dyeing method at the liquor ratio of 1: 50. In conventional method, the samples were dyed at the boiling temperature for an hour. In ultrasonic method, the dyeing process was carried out with Maxwell Ultrasound Dyeing device at 70 ºC for 15 minutes and the samples were exposed to 20 kHz ultrasonic power. After many pre-treatments were performed in the ultrasonic device the optimum dyeing conditions were determined. Microwave energy method was applied by using Kenwood MW 467 Model Oven at the frequency of 2.45 GHz. The microwave oven had a maximum power of 800 W with six discrete settings. The fabric specimens were immersed to cooled dyestuff solution, and were dyed in microwave oven at the middle-high level for 3 minutes. After the all dyeing process, the samples were firstly rinsed with cold water, secondly with hot water, and finally with cold water. The properties of dyeing process and samples code were given in the Table 2.

2.5. Fastness Properties

The washing fastness of samples was tested by using Gyrowash/James H.HealCo.Ltd test instrument according to ISO105:C06 [24]. Then the results were evaluated with the gray scale. The light fastness of samples was analyzed by using Atlas Alfa 150 S test instrument according to EN ISO 105 – B02. The rubbing fastness of samples was determined by using James H. Heal 255 crock meter according to ISO 105 X12 [21-23].

Table 2. The properties of dyeing process

Sample Code	Dyeing Properties
Conventional without Mordant	The conventional dyeing without mordant
Conventional with Mordant	The conventional dyeing with mordant
Ultrasonic	The dyeing with ultrasonic energy
Microwave	The dyeing with microwave energy
Plasma Pre-treatment	The conventional dyeing after the plasma pre-treatment

2.6. Colour Measurements

The reflectance values of the dyed fabrics were measured by using Gretaq Macbeth – Colour Eye 2180UV spectrophotometer and the CIELab values were calculated using illuminant D65 and 10o standard observer values. The colour strength (K/S) values of samples were calculated with the Kubelka-Munk equation24 and the reflectance values (R) at the maximum absorption wavelength (λmax). The calculation of K/S values was achieved in accordance with the maximum absorption in 520 nm.

2.7. Analysis of Surface

The surface of samples was analyzed with scanning electron microscope (SEM) by using ZEISS/EVO 40 electron microscope. Furthermore, the chemical groups of samples were analyzed with PERKIN ELMER Spectrum 100 ATR-FTIR Model Spectrophotometers.

3. RESULTS AND DISCUSSION

3.1. Colour Measurements

The samples were categorized into five groups. The first group of samples dyed with the blueberry extracts without mordant via conventional method was accepted as standard for colour measurement. The second group of samples dyed with blueberry extracts after the mordant process. The third group of samples treated with oxygen plasma treatment was dyed with the blueberry extracts via conventional method. The fourth and fifth group of samples were dyed with the blueberry extracts via ultrasonic and microwave method, respectively. CIELab values and colour differences of dyed samples are given in Table 3. The samples dyed with blueberry extracts without mordant was accepted as standard for colour measurements.

The results indicated that the ΔL* values of samples were lighter than the standard sample. The closest value to the standard sample as the lightness-darkness (ΔL*) value was obtained with the sample dyed via microwave method. Furthermore, green nuance was dominant in all of the samples. The least green nuance was observed in the sample dyed after the plasma treatment. In the blue-yellow axis, the closest value to the standard sample was obtained with the sample dyed via conventional method with mordant. In addition to this result, the other samples were bluer than the sample dyed via conventional method with mordant. Moreover, the results indicated that the colour differences of all the samples were inadmissible (ΔE* > 1). Here, the greatest ΔE* value was obtained with the sample dyed after the plasma treatment whereas the lowest value was obtained dyed via conventional method with mordant. Finally, the investigation of the chroma values of samples demonstrated that the chroma value of the sample dyed via microwave method was higher than those of the other samples.

The results of colour strength (K/S) values of the samples dyed via different methods are given in Figure 2.

Table 3. CIELab values of dyed samples

Method	L*	a*	b*	C*	h°	X	Y	Z
Conventional with Mordant	45.37	7.15	1.80	7.37	14.16	15.21	14.81	15.09
Ultrasonic	46.48	8.44	-2.30	8.75	344.77	16.25	15.63	17.87
Microwave	43.99	8.72	-4.58	9.85	332.31	14.48	13.83	16.90
Plasma Pre-treatment	51.27	9.14	-0.52	9.16	356.72	20.29	19.50	21.21

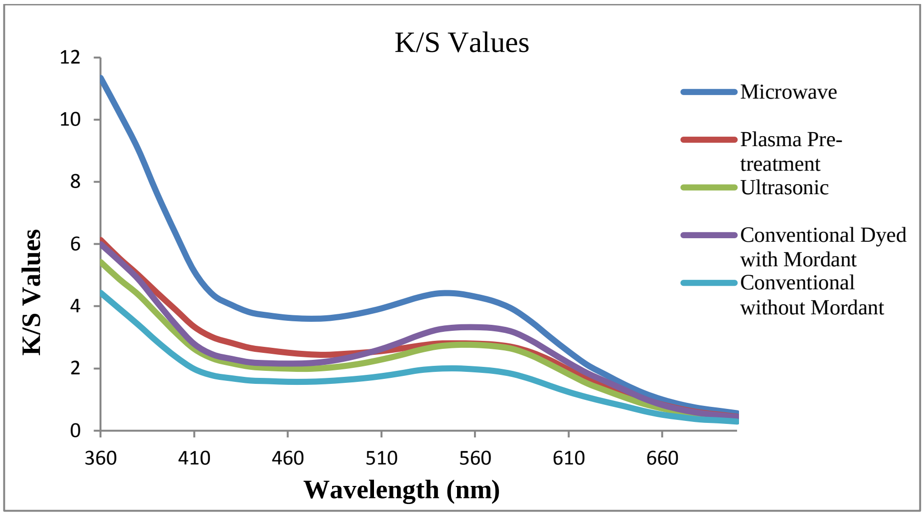


Figure 2. The K/S values of the dyed silk samples

According to the colour strength results (K/S) of samples dyed via different methods, the sample dyed via microwave method was slightly better than the other samples dyed via different methods in a short time of dyeing.

Furthermore, the SEM images indicated that no damages to the samples dyed via ultrasonic, microwave, and conventional method were observed. Microwave heating is characterized as the volumetric heating in which heat diffuses around the instrument from the surface of the material. In volumetric heating, the materials can directly and internally absorb microwave energy and convert it into heat, providing rapid, controlled, selective, and uniform heating. Moreover, microwave heating results in increasing the diffusion of organic molecules into polymers, which in turn increase the fixing rate of dyes into the polymeric textiles [25]. The results demonstrated that the volumetric heating is more effective the plasma treatment, mordant and ultrasonic energy. Ultrasonic power, used in ultrasonic method, diffuses like sound waves in liquid, which results in stretching and relaxation in the structure of liquid. The application of negative pressure on liquid causes fission of liquid and cavitation bubbles. Under pressure, collusion of these bubbles with each other results in diffusion of a great amount of energy, which in turn were used in the textile wet process [25,26]. Compared to the ultrasonic and conventional process, the ultrasonic energy accelerates the chemical and physical reactions in the dyeing process [27,28]. For these reasons, the duration of the dyeing process is short and uniform.

3.2. Results of Fastness

The fastness properties of dyed samples are given in Table 4.

As shown in Table 3, the values of colour change with respect to the reference samples of silk materials were quite low. In this regards, it was illustrated that these processes did not have much effect on the colour change in terms of washing fastness.

According to the washing fastness results, it was observed that there were not much differences among the staining values in terms of conventional, ultrasonic, and microwave method, and

Table 4. The fastness properties of dyed fabrics

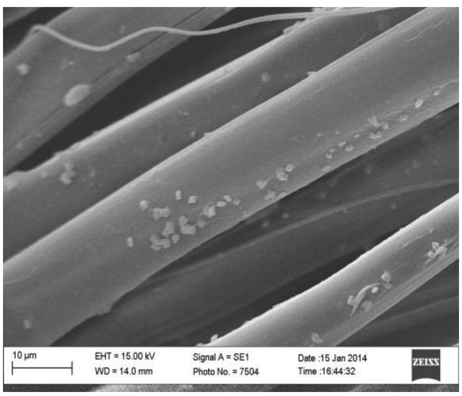
Dyeing method	Light fastness	Washing fastness							Rubbing fastness	
		Colour change	Staining						Dry	Wet
			CA	CO	PA	PES	PAN	WO		
Conventional dyeing without mordant	1	1	4-5	4-5	3-4	4-5	5	5	5	5
Conventional dyeing with mordant	2	1	4-5	4-5	3-4	4-5	5	5	5	5
Ultrasonic dyeing	2	1-2	5	5	5	5	5	5	5	5
Microwave dyeing	1/2	1	5	1-2	4	5	5	5	5	5
Plasma pre-treatment	2	1	5	5	5	5	5	5	5	5

applying plasma pre-treatment. In addition, according to the rubbing results, these methods does not effect on the rubbing fastness.

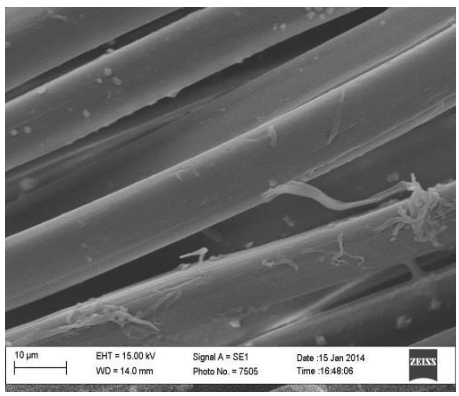
Furthermore, the dyeing methods do not effect on the light fastness properties of samples. The light fastness of all samples is low. The reason of this result is deemed that the chemical structure of natural dyes damaged very quickly with light.

3.3. Characterization of the Surfaces of Dyed Samples

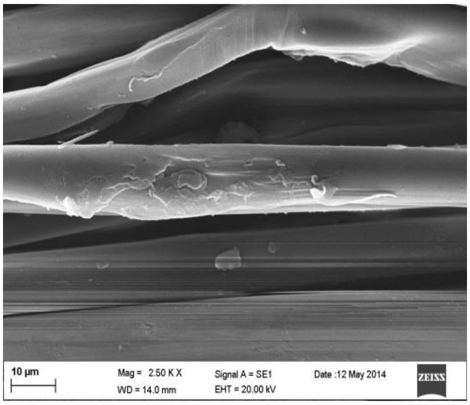
The surfaces of silk samples are characterized with SEM and the SEM images are given in Figure 3.



(a)



(b)



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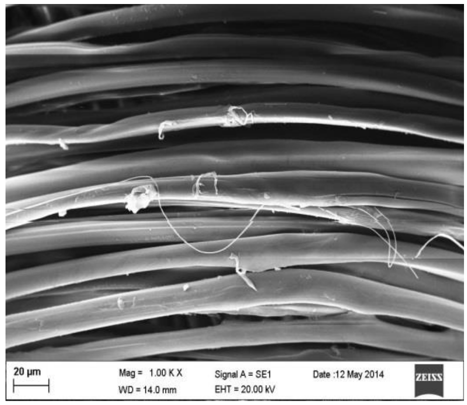

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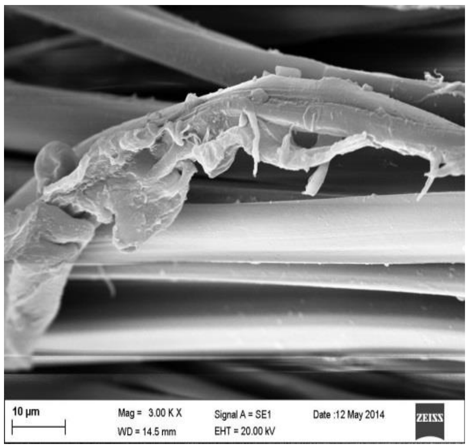

(e)

Figure 3. SEM images for silk fabrics ((a) conventional dyeing without mordant; (b) conventional dyeing with mordant; (c) ultrasonic dyeing; (d) microwave dyeing; (e) plasma pre-treatment)

From the SEM results, one can conclude that oxygen plasma treatment caused deformation on the surface of silk fibers (Figure 3e). Considerable deformation was not observed on the surfaces of samples dyed via conventional, microwave, and ultrasonic methods. It was deemed that the deformation of the plasma treatment increased in the capillarity of plasma treated sample.

3.4. Fourier Transform Infrared Spectroscopy Spectra Dyed Samples

The results of FT-IR ATR spectra of samples are given in Figure 4.

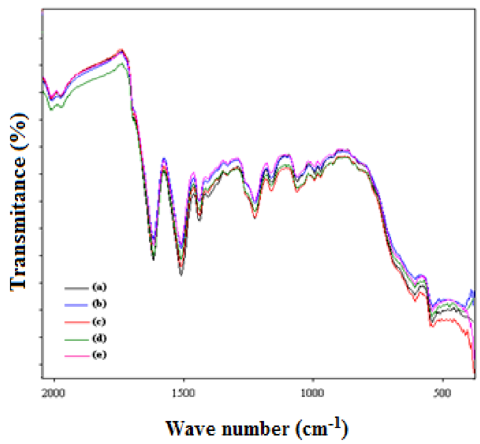

Figure 4. FT-IR ATR spectra: (a) conventional dyeing without mordant; (b) silk fabric with plasma treatment; (c) conventional method with mordant; (d) microwave method; (e) ultrasonic method

FTIR-ATR values of samples dyed with blueberry extract via different dyeing methods were given in Figure 4. As seen in FT-IR ATR spectra, the existence of the groups of Amide I (1655 cm^{-1} – 1630 cm^{-1}) and Amide II (1530 cm^{-1} – 1575 cm^{-1}) demonstrated that silk fabrics are of the structure of β molecular [29-31]. According to FTIR-ATR graphic, the bonds between C-C, C-N, C-H, and N-H stress were shown ranged from 1466.78 cm^{-1} to 1399.69 cm^{-1}, 1260.30 cm^{-1},1580.40 cm^{-1} and, 1635.63 cm^{-1}, respectively. Furthermore, according to the results, plasma pretreatment, microwave and ultrasonic energy did not affect the chemical bonds of silk fabric.

4. CONCLUSIONS

In this study, natural textile materials such as silk fabric were dyed with natural dye extracted from blueberry via different dyeing methods. According to the results, the colour of the sample dyed with microwave energy was darker than those of other samples. The values of colour differences of silk fabrics were not accepted. Besides, the dyeing methods did not affect the improvement of the light fastness of samples. Likewise, it was demonstrated that the processes are not much effective on the washing and rubbing fastness of dyeing. In conclusion, it should be specified that plasma pre-treatment, ultrasonic and microwave methods provide an eco-friendly dyeing process for the silk materials dyed with the natural dyes extracted from blueberry. Moreover, according to the results, as opposed to conventional method, dyeing via ultrasonic and microwave methods were conducted in shorter periods of time and energy.

Besides, according to the results of the SEM analysis, ultrasonic and microwave dyeing method did not harm silk fibers as opposed to plasma pre-treatment. Further, FT-IR analysis indicated that plasma pre-treatment, ultrasonic, and microwave energy did not change the chemical bonds of silk fabrics.

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