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The Effect of Dyeing Properties of Fixing Agent and Plasma Treatment on Silk Fabric Dyed with Natural Dye Extract Obtained from Sambucus Ebulus L. Plant

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Abstract

In this study, the natural dye extracted from the fruits of Sambucus Ebulus L. (Dwarf Elder) was used to dye silk fabric. Prior to the dyeing process, the samples were exposed to oxygen plasma pre-treatment at low frequency for 1 and 5 minutes. Following the plasma pre-treatment, the samples were dyed with the natural dye by the use of conventional and microwave dyeing methods. After dyeing procedure, the samples were treated with three different fixing agents. Finally, the effect of plasma pre-treatment and microwave energy, and type of fixing agent as a dyeing procedure on colour fastness to light, rubbing and washing and also the colour strength of the samples were investigated. According to the results, the increase on plasma treatment time and microwave energy increased the colour strength of samples. The fixing agent treatment did not affect much as the colour fastness properties of dyed samples.

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Keywords: Natural Dye, Fixing Agent, Plasma Pre-Treatment, Microwave Method, Colour Yield.

1. Introduction

In textile industry, to synthetic dyestuffs and pigments are widely used because of their various range of colours, better colour fastness properties and low prices (Angelini et al., 2003). However, synthetic dyestuffs and pigments are ruled out by many producers because of their toxicity and carcinogenic effect, being not bio-degradable as well ecological (Ibrahim et al., 2010). Recently, the textile finishing industry tends to restrict the use of such synthetic dyestuffs and pigments in order for human health and environmental purposes. As a result, the use of natural dye has

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begun to increase for their better properties as being bio-degradable, non-toxic, origination no problem to human health and waste water contaminant (Becthtold et al., 2007; Barka et al., 2009; Baliarsingh et al., 2012). The researches have already studied the dye ability of textiles by use of such natural dyes originating from acacia cyanophylla flowers (Ghouila et al., 2012), weld (Mirjalili et al., 2011), terminalia arjuna, punica granatum, rheum emodi (Vankar et al., 2007), rutin (Nasirizadeh et al., 2012), mader (Nateri, 2011), bixa orellana seeds (Kamel et al., 2007), lak natural dyes (Kamel et al., 2007), henna (Shaukat et al., 2009), cochineal (Kamel et al., 2009) and turmeric (Ibrahima et al., 2010). Recently, the textile industry has investigated ecological production as an alternative use to conventional production (Sahahidi et al., 2010). The plasma technology which is one of the alternative ecological methods is used as a pre-treatment in textile industry (Omerogullari and Kut, 2012). Moreover, the plasma technology offers a dry method as well as ecological, low water, energy and chemical consumption (Bhat et al., 2011). In addition, the application of microwave energy in dyeing processes, the energy consumption is reduced as well the duration of the process. By the effective heating power of the microwave system, the dyeing rate increases resulting decrease in duration of dyeing process (Ahmed and El-Shishtawy, 2011).

In this study, silk fabrics were dyed with natural dyes extracted from Sambucus Ebulus L. fruit, by use of conventional and microwave method. Before dyeing process, firstly the sericin was removed from silk and then treated with oxygen plasma. The aim of this study is the investigation of plasma treatment and microwave energy on the effects of the light, rubbing, washing fastness and colour strength of the dyed materials. Furthermore, the effect of plasma application time is investigated on the dyeing procedure. After the dyeing process, the samples having better colour yield were treated with fixing agent to see if the fastness properties of dyed samples would be increased.

2. Materials 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 dyes of sambucin derived from anthocyanins chemical structure (Figure 1), extracted from Sambucus Ebulus L. by use of microwave and conventional dyeing methods. The fruit of Sambucus which consists of 3% tannins and several anthocyanins (Sambucin, Sambucyanin, Sambunigrin and chrysanthemin, all of cyanidin glycosides) used in folk medicine for snake bites, wounds, hemorrhoids and high fever (Fujita et al., 1995). Figure 1 shows the chemical structure of sambunigrin and image of Sambucus Ebulus L. plant.

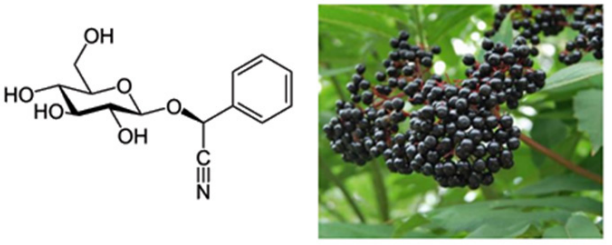


Fig. 1. Chemical structure of sambunigrin and image of Sambucus Ebulus L. plant (Shokrzadeh and Saravi, 2010)

2.2. Plasma Treatment

The plasma treatments of silk fabrics were carried out with oxygen gas in Diener Vacuum Plasma with low frequency at 40 kHz for 1 and 5 minutes. The fabric sample was placed onto the anode and then the pressure of the chamber is adjusted at 0.3 mbar.

2.3. Dyeing Process

The samples treated with oxygen plasma for 1 and 5 minutes then dyed with natural dyes by use of conventional and microwave dyeing methods with 1: 200 liquor ratio. In the conventional method, the samples were dyed at boiling temperature for 30, 45, 60, 75 and 90 minutes. And then, the samples were rinsed with cold water and left to dry. In

the microwave method, microwave energy applied to the sample by use of Kenwood Mark Type 467 at a frequency of 2.45 GHz. The fabric specimens were immersed into the cool dye solution, and dyed in microwave oven for 0.5, 1, 3, 5 and 7 minutes. And then, the samples were rinsed with cold water and left to dry.

2.4. After Treatment

The dyed samples were applied to on after treatment with three different fixing agent solution prepared at 2% concentration. The structure of fixing agents were quaternary polyammonium type cationic polymer fixing agent releasing no formaldehyde (Setafix, Setas Colour Center) and cationic modified type fixing agent (Denafix, Setas Colour Center).

2.5. Colour Measurements

The reflectance values of the dyed fabrics were undertaken by using Gretaq Macbeth – Colour Eye 2180UV spectrophotometer and the CIELab values were calculated using illuminant D65 and 10° standard observer values. The colour strength (K/S) values of samples were calculated with the Kubelka-Munk equation (Eqn 1) (Fairchild, 1997) and the reflectance values (R) at the maximum absorption wavelength (λmax). The calculation of K/S values was carried out with regard to the maximum absorption at 520 nm.

$$K/S = (1-R)^2/2R \quad (2.5.1)$$

The K, S, and R represent the absorption coefficients of the substrate, the scattering coefficient of the substrate, and the reflectance of the dyed fabric at λmax, respectively.

2.6. Measurements of Colour Fastness

The colour fastness to light was measured by use of Atlas Alfa 150S test instrument according to EN ISO 105-B02, and colour fastness to washing by Gyrowash/James H.Heal Co.Ltd. test instrument according to ISO105-C06, and finally the colour fastness to rubbing by James H. Heal 255A crockmeter according to ISO X12 (EN ISO 105-B02 Standard, ISO 105-C06 Standard, ISO 105 X12 Standard).

2.7. Analysis of Chemical Groups

The chemical groups of plasma treated and untreated silk fabrics were analyzed by using Perkin Elmer Spectrum 100 ATR-FTIR spectrophotometers in order to see the effect of plasma treatment.

3. Results and Discussion

3.1. Colour Measurements

The samples were applied oxygen plasma pre-treatment for 1 and 5 minutes after the samples were dyed with natural dyes extracted from Sambucus Ebulus L. After the dyeing process, the samples were treated with three different fixing agents in order to develop the fastness properties of samples.

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

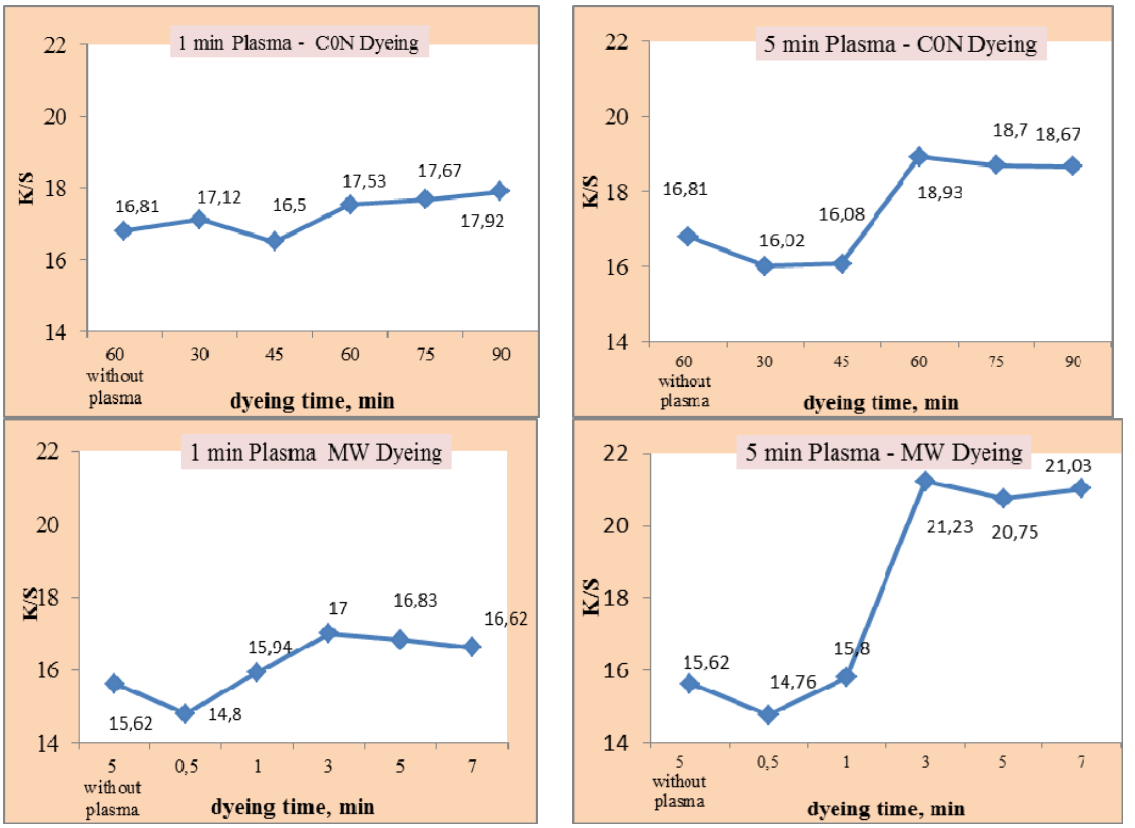


Fig. 2. The K/S values of the samples dyed by conventional and microwave method by the application of 1 and 5 min plasma

Figure 3 shows the K/S values of silk fabric applied oxygen pre-treatment then dyed by conventional and microwave method at 380 nm wavelength. It is seen from the results that the increased time on plasma treatment increase the K/S values of samples. This increase can be attributed to the roughness of fiber surface. As a result, the number of physical and chemical bonds between silk fibers and dyes seemed to be increased (Liu et al., 2011). In conventional method, the results were evaluated in terms of the duration of dyeing as seems the optimum duration of dyeing was 60 minutes. In microwave method, the results indicated that the K/S values of samples were highest in 3 minutes dyeing time. Microwave heating is described as the volumetric heating in which heat diffuses around the instrument from the surface of the material. In volumetric heating, the materials can 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 by causing more pores, resulting to increase the fixing rate of dyes into the polymeric textiles (Oner at al., 2013).

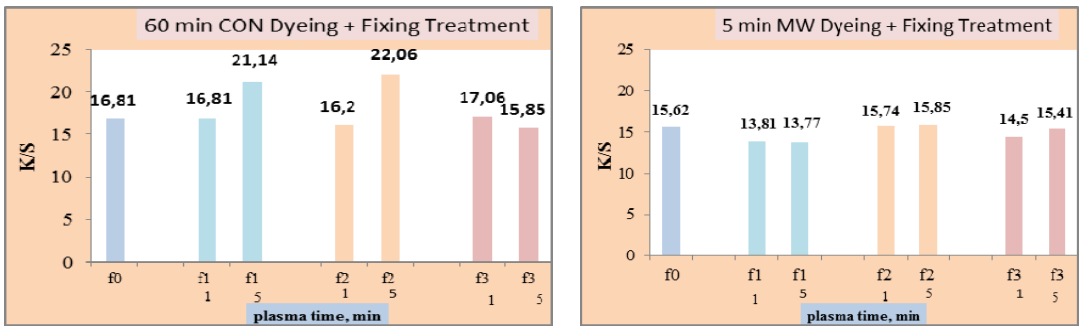


Fig. 3 shows the K/S values of samples which treated with fixing agent after the dyeing process.

Fig. 3. The K/S values of the samples treated with different types of fixing agent and then dyed for 60 mins by conventional and for 5 mins by microwave methods, subjected to 1 and 5 mins plasma treatment (f0: no fixing agent and plasma; f1: cationic polymer type fixing agent releasing no formaldehyde ; f2: quaternary polyammonium type fixing agent; f3: cationic modified resin type fixing agent)

It can be seen from Figure 3 that, the use of cationic polymer and quaternary polyammonium type fixing agent increases the K/S values where the cationic modified resin type fixing agent shows almost no difference on conventional dyeing, whereas the microwave dyeing shows almost no effect, as this might be attributed the fixing agents by stopping the effect of microwave process.

3.2. Fastness Properties

The results of light fastness, washing fastness and rubbing fastness properties of samples dyed by conventional and microwave method were given in Table 1 and 2. Table 3 shows the results of fastness properties of samples treated with fixing agent after the dyeing process.

Table 1. The fastness properties of the samples dyed by conventional dyeing method

		Light fastness	Washing fastness							Rubbing fastness	
			Color Change	Staining						Dry	Wet
1min plasma application				**CA**	**CO**	**PA**	**PES**	**PAN**	**Wo**		
Conventional Dyeing	**Dyeing time, min**										
	30	2	3	5	3	3	5	5	3	5	5
	45	2	3	5	4-5	4-5	5	5	4-5	5	5
	60	2	3	4-5	3-4	3-4	5	5	5	5	5
	75	2	3	5	3	3	5	5	3	5	5
	90	2	3	5	3	3	5	5	4	5	5
5min plasma application											
Conventional Dyeing	**30**	2/3	3	4-5	3-4	2-3	5	5	4	5	5
	45	2	3	4-5	3-4	3-4	5	5	3-4	5	5
	60	2	3	4-5	3-4	3-4	5	5	5	5	5
	75	2	3	5	4	4	5	5	4	5	5
	90	2	3	4	3	3	5	5	4	5	5

The results on Table 1 show that the duration of plasma treatment and dyeing procedure did not affect the fastness properties of the samples.

Table 2. The fastness properties of the samples dyed by microwave method

		Light fastness	Washing fastness							Rubbing fastness	
			Colour Change	Staining						Dry	Wet
1min plasma application				**CA**	**CO**	**PA**	**PES**	**PAN**	**Wo**		
MW Dyeing	**Dyeing time, min**										
	0,5	2	3	5	4	4	5	5	5	5	5
	1	2	3	5	4	3-4	5	5	5	5	5
	3	2	3	4-5	3-4	3-4	5	5	4	5	5
	5	2	3	4-5	4	4	5	5	4-5	5	5
	7	2	3	5	4-5	4-5	5	5	4-5	5	5

5min plasma application											
MW Dyeing	0,5	2	2	5	4-5	4-5	5	5	5	5	5
	1	2	2	5	4	4	5	5	5	5	5
	3	1/2	3	5	4	4	5	5	4	5	5
	5	1/2	3	5	3	4	5	5	5	4	5
	7	1/2	2	5	3	3	5	5	4	5	5

Moreover, the duration of plasma treatment and microwave dyeing also did not change much of the fastness properties as well.

Table 3. The fastness properties of the samples subjected to plasma treatment for 1 and 5 mins, and dyed for 5mins by microwave and 60 mins by conventional dyeing method

			Light fastness	Washing fastness							Rubbing fastness	
				Colour Change	Staining						Dry	Wet
					CA	CO	PA	PES	PAN	Wo		
5 min MW Dyeing	Plasma application time, min											
	f_1*	1	1/2	3	5	3	3-4	5	5	5	5	5
		5	1/2	3	5	3-4	3-4	5	5	5	5	5
	f_2**	1	2	3	5	3-4	3	5	5	5	5	5
		5	2	3	5	3-4	3-4	5	5	5	5	5
	f_3***	1	2	3	5	3-4	4	5	5	5	5	5
		5	2	3	5	3-4	3-4	5	5	5	5	5
	Without plasma MW dyeing		2	3	5	3	3	5	5	5	5	5
60 min CON Dyeing	f_1*	1	2	3	5	3	3-4	5	5	5	5	5
		5	2	3	5	3-4	3-4	5	5	5	5	5
	f_2**	1	2	3	5	3-4	3-4	5	5	5	5	5
		5	1/2	3	5	3-4	3-4	5	5	5	5	5
	f_3***	1	2	3	5	3-4	3-4	5	5	5	5	5
		5	1/2	3	5	3-4	3-4	5	5	5	5	5
	Without plasma CON dyeing		2/3	3	4	3	3	5	5	4	5	5

In the Table 3, f_1* is cationic polymer type fixing agent releasing no formaldehyde, f_2** is quaternary polyammonium type fixing agent and f_3*** is cationic modified resin type fixing agent, respectively.

Table 3 shows the fastness properties of samples treated with fixing agent after the dyeing process. The results show that the type of fixing agent did not increase the fastness properties.

According to the fastness results, Table 1, 2 and 3 show that the light fastness properties of all samples were quite low, of where in the literature also such light fastness is quite low. The washing fastness results were quite acceptable, whereas the rubbing fastness were very high.

3.3. Analysis of Chemical Groups

The FTIR-ATR analysis of plasma treated and raw silk fabrics were given in Figure 4.

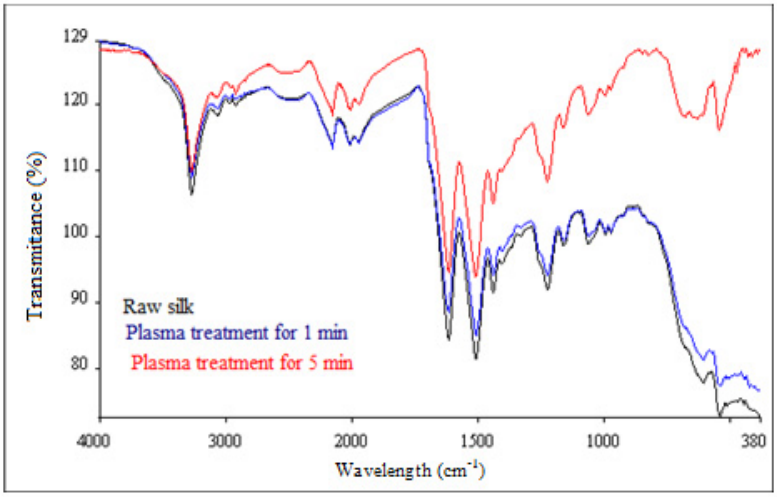


Fig. 4. FTIR-ATR analysis of raw silk and plasma treated silk fabric

FTIR-ATR values of samples dyed with natural dyes extracted from Sambucus Ebulus L. applied to different dyeing methods were given in Figure 4. As seen in FT-IR ATR spectra, the existence of the groups of Amid I (1655 cm^{-1} – 1630 cm^{-1}) and Amid II (1530 cm^{-1} – 1575 cm^{-1}) demonstrated that silk fabrics are of the structure of β molecular (Arai et al., 2004; Shao et al., 2005; Mathur et al., 1997). 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. Moreover, according to the results, plasma treatment did not affect the chemical bonds of silk fabric.

4. Conclusion

In this study, silk fabrics were dyed with the natural dyes extracted from the fruits of Sambucus Ebulus L. by conventional and microwave methods. Before the dyeing process, silk fabrics were treated with oxygen plasma for 1 and 5 minutes in order to see if there is an increase in dyeing properties. After the dyeing process, the samples having deep shades were treated with three different fixing agent causing increase the fastness properties of samples. According to the results, the colour strength of the samples increased with increase of the duration of the plasma treatment. Furthermore, the results show that the microwave energy caused to increase the colour strength of samples. As known from the literature, 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 as a result the colour strength of samples increased with the microwave energy. According to the results of fastness, the plasma pre-treatment did not affect the fastness properties of samples. Besides, the dyeing method did not cause to change the fastness properties of samples. Moreover, according to the results, as opposed to conventional method, dyeing by microwave methods were conducted in shorter periods of time, saving energy and water and had better dyeing properties. Ultimately, the fixing agent treatment and type of fixing agent did not cause any better fastness properties of dyed samples.

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