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Colour and Fastness Properties of Silk Fabrics Dyed With Colours Obtained From The Flowers of The *Papaver Rhoeas* L. (Common Poppy)

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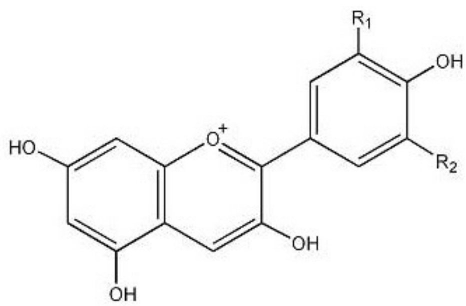
In present study, flowers of the *Papaver rhoeas* L. plant were used. Usability of the flowers of the *Papaver rhoeas* L. which has a wide expansion area throughout the world as natural dyes in dyeing the silk fibres was examined. After the silk material was treated with a mordanting process with the common mordanting agents, it was dyed with the dye extracted from the plant through the conventional and ultrasonic methods. L*a*b* values of the dyed material were measured and analyzed by using the CIE L*a*b* colour space system of the samples. Moreover, washing and light fastness properties of dyed materials were examined. It was observed that colour yield increased in dyeing made with natural dyestuff extracted from the *Papaver rhoeas* L. plant and through the dyeing method performed with environment-friendly ultrasonic energy.

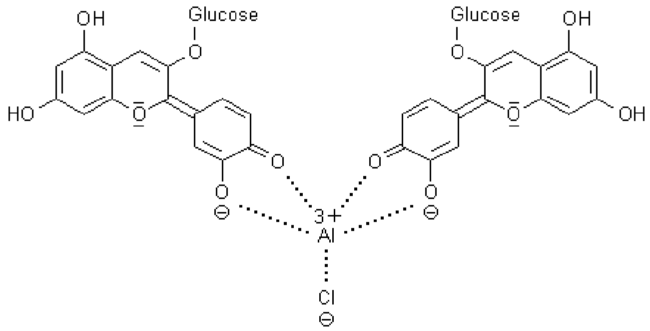
Key Words: *Papaver rhoeas* L., Vegetable dye, Silk, Ultrasonic method, Conventional method.

INTRODUCTION

Natural dyes are found in the root, stem, leaf, flower, fruit and fruit coat compositions of the plants that are available in the nature or generally in the organisms of the crustaceans, marine insects, snails and cochineals. Textile manufacturers have been prompted to seek for new methods on the grounds that synthetic dyes affect human health adversely when they come into contact with the body or that dyestuff that waste waters contain and the chemical substances used along with this dyestuff pollute the environment. Recently, ecotextile practices such as clothes used in their natural colours, recyclable textile products, colouring that is not harmful to the natural life have started to become widespread[1]. Photogenetic natural dyes are extracted from certain parts of many plants that grow in flora available in the nature such as coat, root, stem, leaf, flower, fruit and seed[2]. Dyestuffs containing different chromogen structures are extracted from the plants and are used as natural dyes. There are many natural chromogen structures as indigoid (blue), naphthaquinones (brown, pink, purple), anthraquinones (yellow, pink and red), phenalones (yellow), tannis (reddish brown, black, yellowish brown, bluish black) anthocynanins (blue, bluish red, mauve, brown, orange, reddish brown), carotenoids (orange, yellow, pink, brown colour), chlorophyll (green)[3]. *Papaver rhoeas* L. is an annual plant species from the Papaveraceae family and it has a wide expansion area throughout the world. *Papaver rhoeas* L. which covers the nature with its red flowers are used as vegetables in some regions while red flowers of the plant are consumed as syrup in Anatolia. Chromophore extracted from the *Papaver rhoeas* L. is in the form of anthocyanin. Some metals like Fe^{3+} and Al^{3+} establish decisive coordination complexes with anthocyanins that are in the form of *ortho*-dihydroxyphenyl (Formula 1)[4].



R_1 = H;	R_2 = H:	Pelagonidin
R_1 = OH;	R_2 = H:	Cyanidin
R_1 = OH;	R_2 = OH:	Delphinidin
R_1 = OCH_3;	R_2 = OH:	Petunidin
R_1 = OCH_3;	R_2 = OCH_3:	Malvidin



Formula 1. Main molecular structures of the anthocyanidins present in *Papaver rhoeas* L. flowers and aluminium complex of cyanidin-3-glucoside

Dystuffs extracted from various dye plants are used in dyeing the natural fibres such as wool, silk and cotton. There are some researches where fastness properties and colour values of the plant dyes[5], effects of the anionic agents on dyeing[6], effects of the process parameters on dyeing[7], mordant enzyme complex applications reducing the usage amounts of the mordants that may have a environmental burden[8], UV protection and antimicrobial properties of the natural dye are examined[9].

Silk has always preserved its importance and actuality among the textile raw materials for its naturality, lustre, softness, beauty and its charm resulting from all these properties since the earliest ages of the civilization. Silk fibre is a natural fibre which is composed of different α-amino acids and is in the form of a long chain with condensation and polymerization. It includes protein in the ratio of 97 % and it also includes wax, carbohydrate, pigments and inorganic compounds. Silk fibre protein is composed of an almost 75 % fibroin and 25 % serisin by weight. Silk fibre is a biodegradable natural protein fibre with a smooth structure and high crystallinity. Silk representing the unique and important class of the structural proteins of the nature is an excellent material in the fields of textile, cosmetics and biotechnology[10,11].

Ultrasonic energy is transmitted *via* waves as in the case of any sound wave. Power of the ultrasonic energy reveals its chemical effect through cavitation. These waves cause compression and relaxation in the molecular structure of the medium through which they pass. When sufficient negative pressure is applied to the liquid, degradation is observed in the liquid and cavitation bubbles appear. At the subsequent compression periods, these bubbles reveal a huge amount of energy by clashing each other[12]. There are many studies where ultrasonic energy is used in the pretreatment of the textile materials. The results obtained in these studies included the process time length reduced in hydrogen peroxide bleaching with ultrasonic method and whiteness degree increased in spite of operating at low temperatures[13]; sonication had positive effects on breaking strength, wettability and whiteness degree of the material in bio-cleansing of the raw cotton with pectinase[14]. There were effects of the ultrasonic method on bleaching performed on cotton fabric with laccase enzyme[15]; treatment of the raw cotton through the combined enzyme/ultrasound bio-preparation with the ultrasonic method provided advantages such as a shorter time of process, a better uniformity in the processes and less use of the expensive enzymes[16] and effects of the Persian silk fibres on the weight loss, breaking strength and elongation properties of the material were examined in terms of sonication time, soap, ultrasound-enzyme, enzyme concentration, degumming time and enzyme mixture ratios through the ultrasonic-soap and ultrasonic-enzyme methods which use alcalase, savinase and the mixture of these two enzymes as enzyme[17]. Cationized cotton fabrics were dyed with lac natural dye through both conventional and ultrasonic techniques. Many studies are available where the fact that colour yield and light fastness properties are better in the ultrasonic method in terms of pH, salt concentration, ultrasonic power, dyeing time and temperature[18] was observed, where dyeing properties of the cotton material treated with biomordant were examined by dyeing it through the ultrasonic technique with anthraquinone reddish orange dyes extracted from *Rubia cordifolia* products[19] and where effect parameters such as ultrasound power, particle size, extraction temperature and time were analyzed by dyeing the cotton fabric cationised with Solfix E using Cochineal dye through the conventional and ultrasonic techniques[20].

EXPERIMENTAL

Throughout this experimental work, 100 % silk, pretreated plain woven fabric was used. The weight of the fabric was 80 g/m^2.

Methods

Mordanting: Silk fabric treated with preliminary scouring and cleared of its serisin was mordanted with different mordants separately prior to dyeing under the conditions specified in the Table-1.

Preparation of the dye extract: *Papaver rhoeas* L. flowers used in this study were obtained by gathering only the flowers in May carefully without damaging the plant and its seeds. 20 L treated water was used per 1000 g dry flowers and dye was extracted by soaking the flowers in citric acid at 10 % of the dry flower weight for a week and it made ready for use by being filtered at the end of the period.

TABLE-1
MORDANTS (o.w.f.)

Code	Concentration (%)	Mordant	pH	Condition
1	30	Clay	7	Material: 10 g Liquor ratio: 20:1 Temperature:Boiling Time: 1 h Cooling, pressing, drying
2	10	Citric acid	3	
3	5	Tartaric acid	5	
4	4	Ferrous(II) sulphate	5	
5	3	Sodium dichromate	6	
6	4	Copper(II) sulphate	5	
7	15	Alum	4	
8	8+5	Alum + tartaric acid	4	
9	5	Oxalic acid	3	

Dyeing: 10 g material samples treated with preliminary mordanting and not treated with preliminary mordanting were boiled for 1 h with the extract prepared beforehand and the bath prepared in the 300:1 liquor ratio. At the end of the period, dyed silk was taken out of the extract and left to cooling and then it was rinsed in cold water and left to drying.

The samples of 2 g were dyed by conventional and ultrasonic methods at boiling temperature for 1 h at a liquor ratio of 300:1. Eventually the bath was then cooled and dyed fabrics were rinsed three times, cold rinsing (2.5 L water), warm rinsing (0.5 L boiled water) and cold rinsing (0.5 L water).

Objective evaluation of the colours: Colours of the dyed fabrics were evaluated with CIEL*a*b* colour coordinates (L*,a*,b*,C* and h) and colouring power (K/S) calculated by using Kubelka-Munk equation (eqn. 1). Measurements of the reflectance values % of the samples were performed using GretaqMacbeth-ColorEye 2180 UV colorimetric device and Dyematch computer program. In the measurements, samples obtained with the conventional method were accepted as standard and 10º observer values and D/65 light source were used. Studies were repeated once more in order to control the accurateness of each test (eqn. 2) was used in the calculation of the colour values according to CIE L*a*b* system[21].

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

where R is the reflectance value of the fibre in the wavelength in maximum absorption; K is the coefficient of absorption and S is the coefficient of scattering. Effect of the ultrasonic energy was determined with % relative colouring power (eqn. 3).

$$\text{Strength}\,(\%) = \left[\frac{(K/S_c)}{(K/S_u)}\right] \times 100 \quad (2)$$

$$\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2} \quad (3)$$

L* is the value of lightness-darkness; a* is the value of redness-greenness, b* is the value of yellowness-blueness. h angle formed by the straight line drawn from the colourless point to the colour point with the a* axis is a scale for the colour tone (type).

ΔE* value of the samples represents the colour difference. If the equation is ΔE* < 1, difference between the colours is slight and if it is ΔE* > 1, the difference is significant. Provided that ΔL* value is (–), this means that the sample is darker than the standard and if it is (+), this means that it is brighter. As for ΔC* value, if it is (+), this shows high saturation. Colour turns to red as a* value increases and to green as this value decreases; the colour turns to yellow as b* value increases and to blue as it decreases. Hue angle "h" (in terms of degree) displaying an increase from red to yellow is a measurement of the colour. For instance, h = 0º corresponds to a red colour tone, h = 90º means a yellow colour tone and h = 270º shows a blue colour tone. Limit values were accepted as follows for colour differences in the colour evaluation: ΔE* (total colour difference): 0.5; ΔL* (lightness-darkness difference): 0.5; Δa* (redness-greenness difference): 0.3; Δb* (yellowness-blueness difference): 0.3, ΔC* (saturation difference): 0.3; ΔH* (angular colour difference): 0.3.

Fastness: The colour fastness to washing and light tests were carried out in accordance with ISO 105-C06 and ISO 105-B02, repectively.

RESULTS AND DISCUSSION

Colour measurements: It was found that total colour difference ΔE* values were beyond the accepted tolerances as could be seen in the Fig. 1. when the conventional method was accepted as standard in colour measurements of the silk fabric samples dyed through conventional and ultrasonic methods with the dye extracted from the flowers of *Papaver rhoeas* L. which has anthocyanin chromophore after it was mordanted with nine different mordants. In the study, the biggest ΔE* value was observed in Code 3, 4 and 9 [tartaric acid, ferrous(II) sulphate and oxalic acid mordants]. ΔE* values are, respectively 6.5, 6.4 and 6.4. Sonication was influential in the total colour difference change as it was generally ΔE* > 1 in all the samples. Results beyond these specified limits are accepted as rejection while standard colour evaluation is performed. The fact that colour difference values were beyond the tolerances means that the applied ultrasonic method affected the colour, in other words, it contributed to the colour yield.

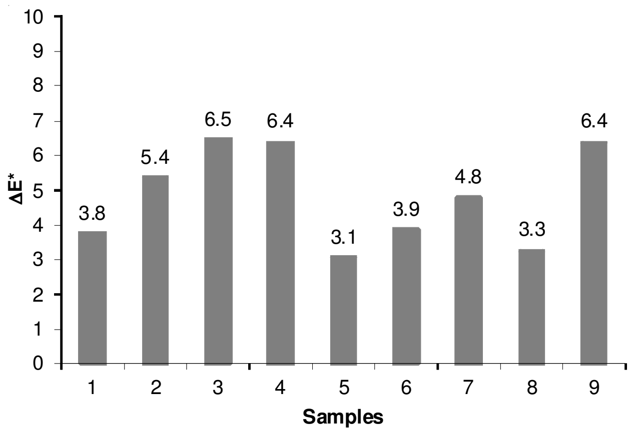
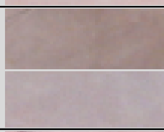


Fig. 1. Colour differences (ΔE*)

It was detected that dyeing processes performed through the ultrasonic method generally result darker dyes when compared to reference or conventional dyeing. When ΔL* values were analyzed, it was observed that the darkness difference was above the tolerance values determined between the reference sample and the other samples (tolerance value ΔL* = 0.5). CIEL*a*b* unit). This shows that the ultrasonic energy had a positive effect, in other words, it increased the penetration of the natural dye into fibre structure.

As can be seen in the Table-2, ultrasonic energy converted the colour tone of the dyed samples to red in most of the samples. When Δa* values were examined, it was detected that all the tests displayed change beyond the tolerances when compared to the reference (tolerance value Δa* = 0.3 CIE L*a*b* unit). It was observed that the use of ultrasonic energy usually converted the colours of the samples to a yellowish tone. Except for the samples mordanted with alum, alum + tartaric acid and oxalic acid, in the other samples, ultrasonic energy ensured the saturation or high chroma of the colour (ΔC*).

TABLE-2
SILK FABRICS DYED BY USING VARIOUS MORDANTS ACCORDING TO CONVENTIONAL (C) AND ULTRASONIC (U) METHODS AND CHANGE IN ΔL*, Δa*, Δb*, ΔC*, ΔH* VALUES WITH DIFFERENT MORDANTS

Code	Mordant	Method	Samples	ΔL*	Δa*	Δb*	ΔC*	ΔH*
1	Clay	C U		-1.49	0.53	3.46	1.63	3.10
2	Citric acid	C U		-4.65	-0.73	2.56	0.21	2.66
3	Tartaric acid	C U		-4.63	1.98	4.10	3.28	3.16
4	Ferrous II sulphate	C U		-0.78	1.79	6.13	2.05	6.05
5	Sodium dicromate	C U		-2.79	1.06	-0.76	0.59	1.16
6	Copper II sulphate	C U		-1.41	3.29	1.49	3.61	0.03
7	Alum	C U		-4.22	-1.41	1.67	-0.88	2.00
8	Alum + tartaric acid	C U		2.31	-1.09	2.10	-0.30	2.35
9	Oxalic acid	C U		-4.59	-4.38	0.68	-3.87	2.16

ΔH* value represents the hue angle difference between the standard fabric and the sample. Maximum difference occurs when ferrous II sulphate is used as mordant and minimum difference is observed when copper(II) sulphate is applied as mordant.

Effect of ultrasonic power: K/S values of the fabric were determined from the diffuse reflectance by using the Kubelka-Munk equation. The diffuse reflectance was measured at wavelength of 400 nm for *Papaver rhoeas* L. Effect of the ultrasonic power was determined thanks to these values.

Colour strength values of the silk fabric samples are higher as ultrasonic energy is an additional effect factor in deaggregation of the dye molecules and it enables a more rapid movement and effective blending, dye diffusion and a better dyeability when compared to the conventional method[22].

Fastness results: Washing and light fastness properties of the samples dyed with *Papaver rhoeas* L. are shown in Table-3. Light fastness properties of dyeing depend on the mordant and the mordanting method that are used. This is because metal dyeing complexes whose light strengths are different occur. Metal can have an either positive or negative catalytical effect in photochemical degradation of the dye[23].

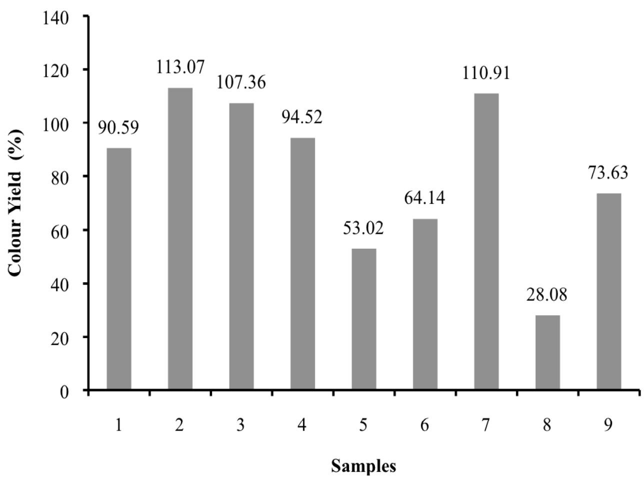


Fig. 2. Relative colour yields (%) of dyed samples with *Papaver rhoeas* L (400 nm)

TABLE-3
FASTNESS TEST RESULTS OF DYED FABRICS

Samples	Washing fastness														Light fastness	
	Colour change		Staining													
			CA		Co		PA		PES		PAN		Wo			
Unmordant	4	5	2/3	3/4	2	2/3	1	1/2	5	5	5	5	3	4	1/2	2
Clay	5	5	5	4/5	4/5	3/4	4	3	5	5	5	5	5	5	2	1/2
Citric acid	5	5	5	4/5	4/5	3	4/5	3/4	5	5	5	5	5	5	2	1/2
Tartaric acid	5	5	5	4/5	4/5	3	4	3	5	5	5	5	5	5	2	1/2
Ferrous(II) sulphate	4	5	3	5	2/3	4/5	1	4/5	5	5	5	5	3	5	2/3	2/3
Sodium dichromate	5	5	5	4	4	2/3	4/5	1/2	5	5	5	5	3	5	1/2	2
Copper(II) sulphate	5	5	5	3	4/5	3	4/5	1	5	5	5	5	4	3	2	2/3
Alum	5	4	5	3	4	2/3	4/5	1	5	5	5	5	5	4	2	2
Alum + tartaric acid	5	5	5	5	4/5	4/5	4	4	5	5	5	5	5	5	2	2
Oxalic acid	4	4	3/4	3/4	2/3	2/3	1/2	1/2	5	5	5	5	4	4	2	1/2

It was determined that light fastness was rather low in both methods. It can be thought that this is caused by the asymmetrical dye molecule. It was also found that ultrasonic energy did not have an important effect on the washing properties of dyeing.

Conclusion

When the results of the dyeing processes conducted through the conventional and ultrasonic methods with the dyes extracted from the flowers of the *Papaver rhoeas* L. plant are evaluated in a general sense (CIE L*a*b* colorimetric coordinates), it is observed that ultrasonic energy method yielded better results. This is caused by the sonication power of the ultrasonic energy. This method which is also environment-friendly provides savings on energy, auxiliary chemicals, water and time and it yielded positive results in terms of fastness values. Environment-friendly dyeing studies performed with natural dyes obtained from the flowers of the *Papaver rhoeas* L. which are more natural than the synthetic dyestuff used in the study have yielded considerable positive results.

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