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Investigation on Colour and Fastness Properties of Wool Fabrics Dyed With Colours Obtained From the Flowers of the *Papaver Rhoeas* L. *(Common Poppy)*

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N. Merdan, M.Z. Saclıoglu, D. Kocak, B.Y. Sahınbaskan; Investigation on Colour and Fastness Properties of Wool Fabrics Dyed With Colours Obtained From the Flowers of the *Papaver Rhoeas* L. *(Common Poppy)*

ABSTRACT

In this research, usability of the flowers of the *Papaver rhoeas* L. which has a wide expansion area throughout the world as natural dyes in dyeing the wool fibres was examined. Wool fabric was dyed through the conventional and ultrasonic methods with the dye extracted from the plant after it was mordanted with the common mordanting agents. $L^*a^*b^*$ values of the materials were measured and compared by using the CIE $L^*a^*b^*$ colour space system of the wool fabric samples. Moreover, washing and light fastness properties of the 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, wool, ultrasonic method, conventional method

Introduction

Natural dyestuffs are extracted from animal and plant materials and they are used as colourant in many fields such as food and textile industries [1]. Use of the plant and animal dyes traces back to the earliest ages. Today, the most important natural dyestuff source is the plant dyestuffs. Plant dyes are extracted from the parts of the plants such as flower, cone, stem coat and root without any chemical process or as a result of minimum chemical processes possible. In today's world where environmental problems are increasing day by day, overwhelming dominance of the chemical dyes to the natural dyes continues. Nowadays, natural dyes have started to draw attention due to the fact that synthetic dyes are not sufficiently degradable, they are harmful to the environment and they cause allergy. Future of the dye plants which are among the non-food products used as the source of the natural dyes is promising. Herbal dyes are used in food, textile, cosmetics and drug preparation. Attention to the production of the ecotextiles based on natural dyes is increasing gradually in the use of ecological and economical sources. Yellow, red, green and dark gray tones can be obtained from various plant materials [2]. Dye plants include natural chromogenes such as indigoid, anthraquinones, naphthaquinones, phenalones, anthocyanins, tannis, carotenoids and chlorophyll that allow the various pigment colours to be obtained [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. Chromofore obtained from the *Papaver rhoeas* L. is merocyanine which is a derivative of the anthocyanins (Formula 1)[4].

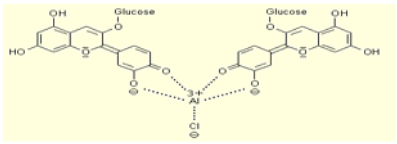
Main molecular structures of the anthocyanidins present in *Papaver rhoeas L.* flowers ve Aluminium complex of cyanidin-3-glucoside

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

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antimicrobial properties of the natural dye are examined [9]. Beside to these studies, it is stated that indigo carmine can be a dye source for wool and tin dyeing and single-path dyeing is advantageous in terms of consumption of energy, water and chemicals [10]. It is specified that colouring power, dye penetration values and fastness properties are high in wool dyeing performed with the natural dye obtained from red prickly pear by using different mordants [11].





(Formula 1)

The wool keratin molecule consist of a highly complex sequence of amino acids. The protein is mainly composed of eighteen amino acids with cystine, lysine, arginine, glutamic acid and aspartic acid. This complexity is not confined to the individual molecules.

The physical structure of wool fibre is equally complex, being organised on several hierarchical levels. The essential macroscopic components of the wool fibre are the cortex and the cuticle. The cortex has a bilateral structure in which approximately half of the crosssection is made up of ortho-cells, whilst the opposite side consists of para-cells. This unusual feature gives rise to several distinctive properties, including differential dye-affinity and swelling. The cortical cells are composed of complex arrangements of smaller structural units including micro and macrofibrils [12].

Ultrasonic energy is transmitted via waves as in the case of any sound wave. Ultrasonic sound waves have a frequency range of 20kHz-20MHz. 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 [13]. There are many studies where ultrasonic energy is used in the pretreatment of the textile materials. The results obtained in these studies included: process time length reduced in hydrogen peroxide bleaching with ultrasonic method and whiteness degree increased in spite of operating at low temperatures [14]; sonication had positive effects on breaking strength, wettability and whiteness degree of the material in bio-cleansing of the raw cotton with pectinase [15]; the effect of ultrasonic sound waves in the range of 35-39 kHz for investigate wool dyeing and finishing processes with reactive and acid milling dyes, ultrasonic technique was reported to have been an alternative method for chemical and energy saving [16]. Wool fabrics were dyed with lac natural dye through both conventional and ultrasonic techniques and it was stated in the comparison made in terms of pH, salt concentration, ultrasonic power, dyeing time and temperature that colouring power and fastness properties are better in the ultrasonic method [17]. Cotton, silk and wool yarns were dyed with the natural dye obtained from *Mahonia napaulensis* and it was found that dye penetration was better in sonicator dyeing [18].

Experimental:

Materials:

Throughout this experimental work, 100 % wool, pre-treated plain woven fabric was used. The weight of the fabric was 110 g/m^2.

Methods:

Mordanting:

Wool fabric treated with preliminary scouring was mordanted with different mordants separately prior to dyeing under the conditions specified in the TABLE-1.

Table 1: Mordants (o.w.f.)

Code	Concentration (%)	Mordant	pH	Condition
1	30	Clay	7	Materail: 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 Sulphate	5	
5	3	Sodium dicromate	6	
6	4	Copper Sulphate	5	
7	15	Alum	4	
8	8+5	Alum+Tartaric Acid	4	
9	5	Oxalic Acid	3	

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 liters 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.

Dyeing:

10 g material samples treated with preliminary mordanting and not treated with preliminary mordanting were boiled for an hour with the extract prepared before hand and the dye bath prepared in the 20:1 liquor ratio. At the end of the period, dyed wool 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 hour 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 CIE $L^*a^*b^*$ colour coordinates and colouring power (K/S) calculated by using Kubelka-Munk equation (Equation 1). Measurements of the % reflectance values of the samples were performed using GretaqMacbeth – ColorEye 2180UV colorimetric device and Dyematch computer program. In the measurements, samples obtained with the conventional method were accepted as standard and 10° observer values and D/65 light source were used. Studies were repeated once more in order to control the accurateness of each test (Equation 2) was used in the calculation of the colour values according to CIE $L^*a^*b^*$ system [19].

$$K/S = (1-R)^2/2R \qquad \text{(Equation 1)}$$

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 (Equation 3).

$$\% \text{ Strength} = [(K/S_c) / (K/S_u)].100 \qquad \text{(Equation 2)}$$

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

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 $\Delta E^* < 1$, difference between the colours is slight and if it is $\Delta E^* > 1$, 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=0° corresponds to a red colour tone, h=90° means a yellow colour tone and h=270° 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:

Colour Measurements:

It was found that total colour difference 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 wool 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 2, 3 and 7 (sitric acid, tartaric acid, alum mordants). ΔE^* values are respectively 16.31, 13.65 and 13.25. Sonication was influential in the total colour difference change as it was generally $\Delta E^* > 1$ in all the samples. Results beyond these specified limits are accepted as rejection while standard colour evaluation is performed. In this study, 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.

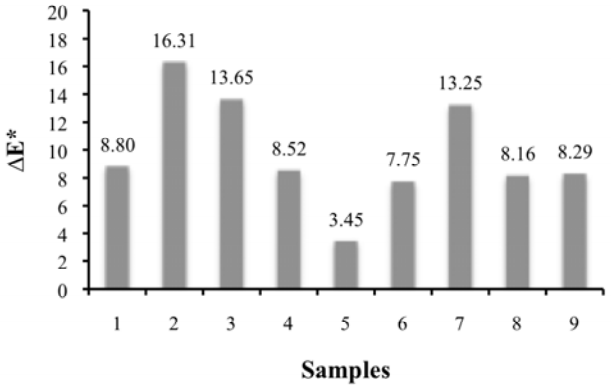


Fig. 1: Colour differences (ΔE^*).

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, CIE $L^*a^*b^*$ 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.

In most of the wool samples that were dyed, ultrasonic energy converted the colour tone of the dyed samples to red. When Δa^* values were examined, it was detected that all the tests showed changes 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 generally had the samples acquired a yellowish tone. Ultrasonic energy provided the saturation of the colour or a high level chroma in all samples.

ΔH^* value represents the hue angle difference between the standard fabric and the sample. Maximum difference occurs when sitric acid is used as mordant and minimum difference is observed when sodium dicromate is applied as mordant.

Effect of Ultrasonic Power:

K/S values of the fabrics 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 wool 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 [20].

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 [21].

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 dyed samples.

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 $L^*a^*b^*$ system), 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 natural dyes extracted from the flowers of the *Papaver rhoeas* L. which are more natural than the synthetic dyestuffs used in the study yielded practical and promising results in the dyeing studies.

Table 2: Wool Fabrıcs Dyed by Usıng Varıous Mordants Accordıng to Conventıonal and Ultrasonıc Methods.

Code	Mordant	Metod	Samples	ΔL*	Δa*	Δb*	ΔC*	ΔH*
1	Clay	**C**		5.96 (More light)	-0.41 (More green)	6.47 (More yellow)	4.61 (More saturated)	4.56
		U						
2	Citric Acid	**C**		14.68 (More light)	-3.48 (More green)	6.20 (More yellow)	2.50 (More saturated)	6.65
		U						
3	Tartaric Acid	**C**		12.57 (More light)	-1.73 (More green)	5.02 (More yellow)	2.79 (More saturated)	4.51
		U						
4	Ferrous II Sulphate	**C**		7.40 (More light)	-1.37 (More green)	3.99 (More yellow)	2.87 (More saturated)	3.10
		U						
5	Sodium dicromate	**C**		-2.07 (More light)	-0.25 (More green)	2.75 (More yellow)	2.26 (More saturated)	1.59
		U						
6	Copper II Sulphate	**C**		6.34 (More light)	-2.08 (More green)	3.95 (More yellow)	2.30 (More saturated)	3.83
		U						
7	Alum	**C**		12.64 (More light)	-1.70 (More green)	3.59 (More yellow)	1.69 (More saturated)	3.59
		U						
8	Alum + Tartaric Acid	**C**		7.49 (More light)	-1.17 (More green)	3.04 (More yellow)	1.58 (More saturated)	2.85
		U						
9	Oxalic Acid	**C**		7.46 (More light)	-0.52 (More green)	3.57 (More yellow)	2.34 (More saturated)	2.74
		U						

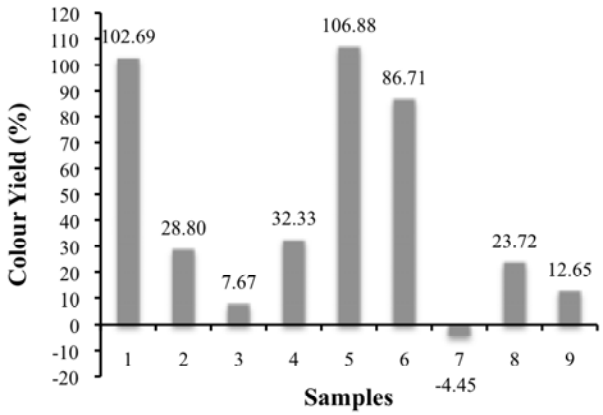


Fig. 2: Relative colour yields (%) of dyed samples with *Papaver rhoeas* L. (400 nm).

Table 3: Fastness Test Results of Dyed Fabrıcs.

Samples	Washing Fastness														Light Fastness	
	Colour Change		Staining													
			CA		*Co*		PA		*PES*		*PAN*		*Wo*			
	C	*U*	*C*	*U*	*C*	*U*	*C*	*U*	*C*	*U*	*C*	*U*	*C*	*U*	*C*	*U*
Unmordant	4	4	2/3	3	2	2/3	2	1/2	5	5	5	5	5	4	1/2	2
Clay	5	4	5	4/5	4/5	3/4	4	3	5	5	5	5	5	5	2	2
Sitric Acid	5	5	5	4/5	4/5	3	4/5	3/4	5	5	5	5	5	5	2	1/2
Tartaric Acid	4	4	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	2	4/5	5	5	5	5	3	3	2/3	3
Sodium dicromate	5	5	5	4	4	2/3	4	1/2	5	5	5	5	5	3	1/2	2
Copper II Sulphate	5	5	5	3	4/5	3	5	1	5	5	5	5	3	5	2	2/3
Alum	5	5	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	5	4	3/4	4	2/3	3	1/2	2	5	5	5	5	4	5	2	1/2

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