

Original Research Article

Environmentally friendly dyeing of Silk fabrics Using Microwave Heating

E.M.El-Khatib, N.F.Ali* and M.A.Ramadan

National Research Centre, Textile Research Division, El Buhouth St., Dokki,
Cairo, Po. 12622, Egypt

*Corresponding author

ABSTRACT

Keywords

Dyeing,
Microwave,
Silk fabrics,
Saffron,
Natural
Dyes

Silk fabrics were dyed with natural coloring matter extracted from Saffron (*Crocus sativus*) using a traditional and microwave heating methods. Factors affecting the dyeing properties such as dye extracted quantity, dye concentration, pH values, temperature and duration of dyeing bath were studied. Environmentally friendly pre-treatment with chitosan were used instead of mordant. Color strength (K/S) values was measured for dyed Silk fabrics. The fastness properties including light, washing, and perspiration of dyed fabrics were assessed. The dye extraction and Color strength (K/S) of Silk fabrics indicate that microwave heating is more effective than traditional heating. Other additional features about microwaves are economical (saving time and energy), eco-friendly, and produce a higher dye uptake as compared to conventional techniques. The results show also good fastness properties. It is necessary to promote non-polluting natural dyes, which involve inexpensive equipment and small-scale operations. The exploration of natural dyes can be environmentally and economically viable. Our aim is to show the feasibility of providing high-quality natural dyes extracted from plants, this improving our environment and giving opportunities to the fabric industry to catch up with the current consumer trends towards more aesthetic fabrics with natural products.

Introduction

In recent years, the textile industry must go towards developing of new technologies to reduce the energy and water consumption. The use of microwave in textile wet processing is one way for this purpose. The advantages of microwaves, which it is use much less liquid, they can exhaust or save dyes and leave no waste of liquid dye compared to conventional methods. Microwave dyeing has other advantages

such as less power consumption, easy production of desired shades, as some synthetic dyes are being banned by the Western countries due to their toxic, carcinogenic and polluting nature. A natural dye is a colorant (dye or pigment) obtained from vegetable or animal matter without any chemical processing (Singh, 2000, Shin, and Cho 2003).

In recent years, there has been a manifested interest in natural dyes. The reasons are manifold, including the ecological movement, biodegradability and higher compatibility of natural dyes with the and quick dyeing (Saima et al., 2008; Bechtold et al., 2003, Bechtold et al., 2006).

On the other hand natural dyes have been used since ancient times for coloring and printing fabrics. Several studies on the application of natural dyes have been reported (Singh, 2000; Miquel et al, 2002, Kamel et al., 2005)) to protect the environment for indiscriminate exploitation and pollution by industries. Recently, the potentiality of using natural dyes in textile coloration as UV-protection and anti microbial has been investigated (Ali et al., 2011)

There has been a revival of interest in natural dyes throughout the world environment (Teli et al., 2000; Lee et al 2001). Some natural dyes require the inclusion of one or more types of metallic salt (mordents), such as aluminum, iron, chromium, copper and tin, to ensure reasonable color fastness to sunlight and washing. These metallic salts combine with the dyestuff to produce dye aggregates, which cannot be removed easily (Hebeish et al., 2012).

However, natural dyes do not necessarily help improve the environment because some of the mordents like chromium and copper are deadly poisons. Only non-hazardous chemicals like aluminum, tin and iron help produce favorable colors without harming the eco-cycle (Tiwari et al., 2001). There are only two or three places in the world where Saffron (*Crocus sativus*) grows. Kashmir has the proud privilege of being one of these places. Saffron (*Crocus sativus*) plants very

small and its flower is the only part which is seen above the ground. The blooming time of this flower is autumn. Saffron (*Crocus sativus*) has a unique sweet smell and it is widely used as natural dye in dyeing, cosmetic industry and cooking (Miquel et al., 2002).

It is commonly known as Crocus, it consists of dried stigmas and upper parts of styles of plant *Crocus sativus* Linn. The image of the Crocus plant was showed in the Fig. 1.

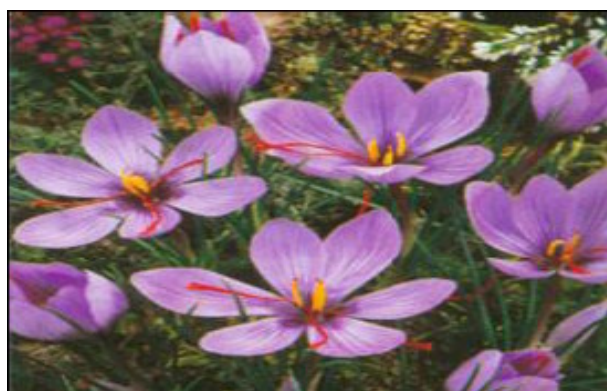


Figure 1: Saffron plant

The present work is undertaken with a view to establish a green strategy for dyeing. So that environmentally friendly pre-treatment with chitosan were used instead of using mordents.

In this work silk fabrics were dyed with natural dye extracted from Saffron (*Crocus sativus*) powder (Duran et al., 2002), using a traditional and microwave heating methods (Parton 1997).

Experimental

Silk Fabric

Silk fabric (53 g/m²), supplied by Akhmim Upper, Egypt, was treated with a solution containing 2 g/l anionic detergent and 2g/l sodium carbonate at 45°C for 30 min, thoroughly washed and then air dried at

room temperature.

Microwave Equipment

The microwave equipment used in this experiment was the Samsung M 245 with an output of 1,550 watts operating at 2450 MHz.

Dyestuff

Scientific classification

Saffron plant: Saffron (*Crocus sativus*) was classified based on chemical nature

Class – Carotenoid pigment

C.I. name – Natural Yellow 6

Part used: The dried stigmas of flowers.

Kingdom : Plantae

(unranked) : Angiosperms

(unranked) : Monocots

Order : Asparagales

Family : Iridaceae

Subfamily : Crocoideae

Genus : *Crocus*

Species : *C. sativus*

Binomial name: *Crocus sativus*

Chemistry of pigments

The main constituents of saffron are crocin, crocetin, picrocrocin and safranal. α -Crocin is a carotenoid pigment which is primarily responsible for saffron's golden yellow-orange color. The bitter glycoside picrocrocin is responsible for (saffron's flavor and taste). It is a union of an aldehyde sub-element known as safranal, which is responsible for the (odor and aroma) of the saffron. Structures of the chemical constituents illustrated in Figure. 2.

Methods

Conventional Extraction

Conventional extraction was carried out in 100ml boiled distilled water using varying amounts of the dried stigmas of flowers

(0.1-1.5%) for 60 minutes. After filtration and dilution, the optical density of the dye liquor was measured at λ_{max} 440 nm. (Tiwari, et al., 2001).

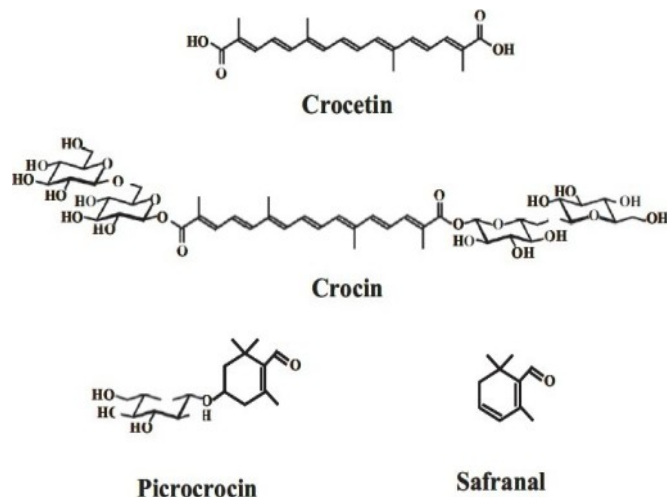


Figure 2: Structures of the chemical constituents of saffron

Microwave extraction

Microwave extraction was carried out as mentioned above in 100ml distilled water using varying amounts of the dried stigmas of flowers (0.1- 1.5%). After filtration and dilution, the optical density of the dye liquor was measured at λ_{max} 440 nm (Tiwari, et al., 2001)The best concentration at higher optical density of the dye liquor was carried out for different time periods (1-6 minutes).

Pretreatment with chitosan

Chitosan (high molecular weight) solution was freshly prepared by dissolving (2.0 g/l) of chitosan in distilled water containing acetic acid (4g/l). The silk fabrics were immersed in this solution at a liquor ratio 20:1. Fabrics were then squeezed to a wet pick of 100%. The so padded samples were dried at 100°C for 3 min, followed by curing at 150 °C for 3min then washed in distilled water and finally dried at ambient conditions

(ALi and EL-Khatib, 2010)

Dyeing Procedure

In a dye bath containing different concentrations (1- 15 g/l) of saffron dye with a liquor ratio 1:100, the silk fabric was dyed by conventional heating at different path pHs (2-8) for different time periods (10-60 minutes) and at the boiling temperature. For comparison, the same dyeing condition was made for microwave heating for different concentrations of saffron dye (1- 15 g/l) and time periods (1-6 minutes). Thus, in a dye bath containing (10 g/l) saffron dye with a liquor ratio 1:100 at pH 3. The dyed samples were rinsed by warm water and then cold water, washed in a bath containing 5g/l non-ionic detergent at 50°C for 30 minutes, then rinsed and dried in shade at room temperature.

Measurements

Color Strength (K/S value)

The reflectance was measured against the soaped samples in the Perkin-Elmer Lambda 3B UV/Vis spectrophotometer. The values of color strength (K/S) values were determined by the Kubelka-Munk equation (Judd, 1975).

$$K/S = (1-R)_{\infty} / 2R$$

UV/Vis Absorption Spectra

The spectra of UV/Vis absorption in water were recorded by the Shimadzu UV/Vis spectrophotometer. The quantity of dye uptake was estimated by the following equation:

$$Q = (C_o - C_f) V/W$$

Where Q is the quantity of dye uptake (mg/g), C_o and C_f are the initial and final concentrations of dye in the solution (mg/l) respectively. V is the volume of the dye solution in (L). The concentrations of the

dye solutions were determined after reference to the respective calibration curves of saffron dye using the Lambert-Beer's law.

Fastness Properties

The dyed samples were tested according to the ISO-standard methods. The color fastness tests are: rubbing (ISO 105 – X12, 1987), washing (ISO 105 – CO₂, 1989) and light (ISO – BO₂, 1989) using the Tera Light Fastness Tester. The color changes of the samples were assessed against an accurate Gray scale.

Results and Discussion

Dye Extraction

Effect of Dye Amount

The comparative extractability of saffron dye using microwave irradiation at 5 minute and conventional heating at the boiling temperature was measured spectrophotometrically at λ_{max} 440 nm. Figure 3 illustrates that as the dried stigmas of flowers amount increases, the absorption of extracted dye increases when using either microwave heating or traditional heating. The figure also illustrates that the obtained values of dye extraction are higher when using microwave irradiation.

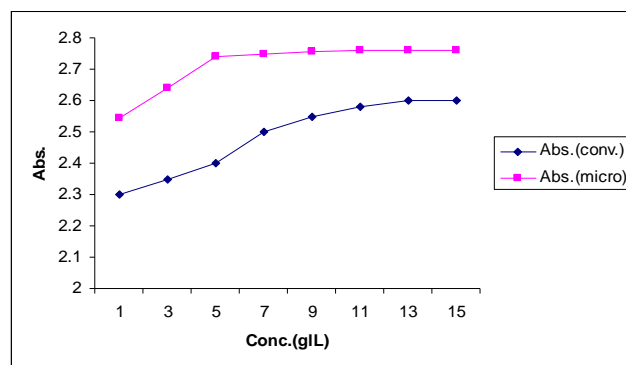


Fig.3 Effect of the dried stigmas of flowers conc. on dye extraction by using conventional heating and microwave

irradiation

Effect of Extraction Time

The extraction of saffron (0.1g/100ml water) by microwave irradiation was carried out for different lengths of time (1-6 minutes). Figure 4 shows that dye extraction by microwave irradiation is higher than that by traditional heating and the maximum values were obtained after 4 minutes respectively. This can be attributed to microwave irradiation producing an intensive movement in the liquor due to the wave-guides. The resulting distribution of microwaves gives a uniform exposure to which any material moves through.

Effect of time of dye extraction

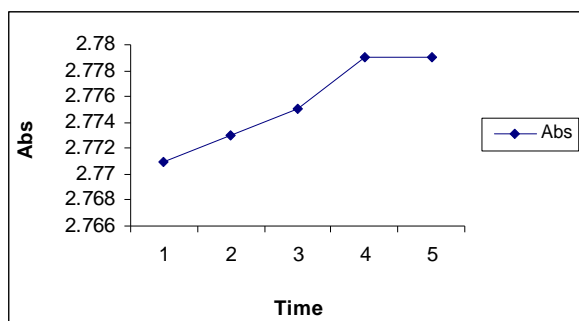


Fig. 4: Effect of time on the extent of dye extraction by using microwave irradiation

Dyeing

Effect of Conc. of dye

Silk fabrics treated with chitosan and untreated dyed by saffron dye at different concentrations (1- 15 g/l) of a liquor ratio 1:100, the silk fabrics were dyed by conventional heating at boiling for one hour and microwave method for 5 minutes.

The results show that the K/S was increased by increasing the concentration of the dye till 13g/l in conventional method.

In case of microwave method the K/S increased till 10g/l of the dye. Fig. 5 and 6 illustrate that the microwave dyeing is more

effective than conventional dyeing.

Effect of Conc. of dye by Conventional method

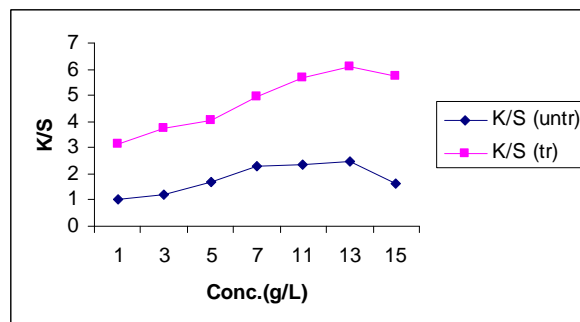


Fig. 5: Effect of conc. of dye on dyeing silk fabrics (treated and untreated) by Conventional method on color strength (K/S).

Effect of Conc. of dye by microwave method

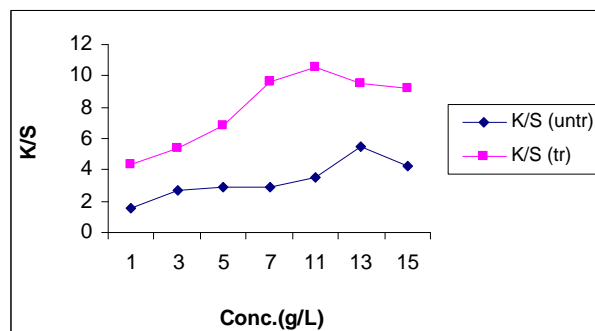


Fig.6: Effect of conc. of dye of dyeing silk fabrics (treated and untreated) by microwave method on color strength (K/S).

Effect of pH Level of Dye Bath

Figure 7 shows that the pH values of the dye bath have considerable effect on the silk fabric's dye ability when using either dyeing technique (expressed as K/S). The results illustrate that microwave irradiation and traditional heating enhances dye ability at pH 3. The effect of the dye bath's pH level can be attributed to the waves correlating dye molecules and the silk fabric. Since saffron dye contains terminal carboxylic and

hydroxylic groups, it interacts ionic ally with the protonated terminal amino groups of silk fibers at the acidic condition via the ion exchange reaction. The weak carboxyl and hydroxyl anion of dye present at an acidic medium replaces that of the acid due to its higher affinity. The anion of dye has a complex nature, and when it is bound to the fiber, another kind of interaction takes place together with the ionic force. This ionic interaction leads to an increase in the silk fabric's dye ability as shown in Fig. 7.

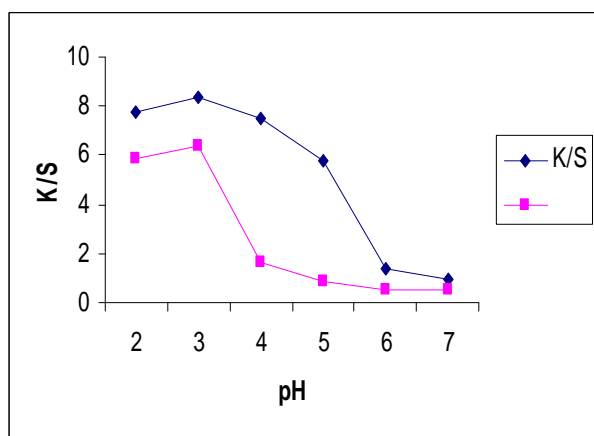


Fig .7: Effect of dye bath pH on dyeing by Conventional and microwave methods on color strength (K/S)

Effect of Dyeing Time

The effect of dyeing time was also studied. Figures 8 and 9 illustrate the effect of dyeing time on the color strength. From the figure, it can be observed that the color strength increases as dyeing time increases when using either microwave or traditional heating. The effect of microwaves is greater as dyeing time increases up to 5 minutes, while it takes 50 minutes to achieve the same effect when using traditional heating. Under microwave irradiation, the dyed fiber reveals significantly improved levelness. In case of traditional heating, the absorbed dye is distributed unevenly on individual fibers,

and is concentrated in only a few parts of the outer areas. The use of microwave irradiation leads to a greater degree of dye penetration. Microwave irradiation results in a higher and more uniform concentration of the dyestuff on the fiber surface, making it available for diffusion into the fiber interior.

Effect of time on dyeing by Conventional method on color strength (K/S)

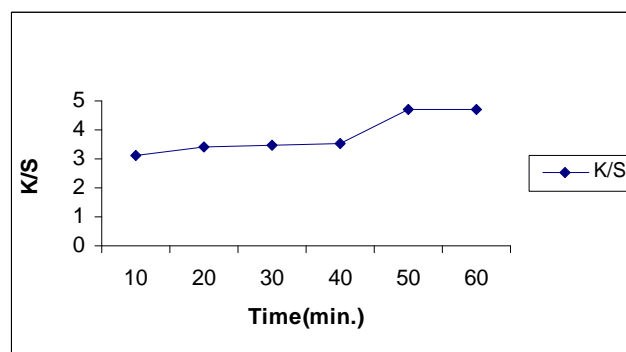


Fig.8 Effect of time on dyeing by Conventional method on color strength (K/S)

Effect of time on dyeing by microwave method on color strength (K/S)

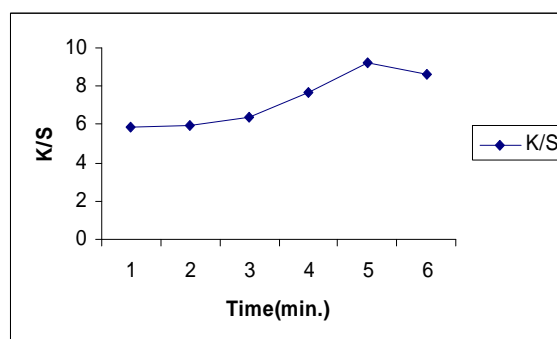


Fig.9. Effect of time on dyeing by microwave method on color strength (K/S)

Fastness properties

The fastness properties of washing, rubbing, perspiration and light in terms of microwave irradiation and traditional heating are shown in Table 1.

Table.1 Fastness properties of dyed silk fabrics using microwave irradiation and traditional heating

Dyeing methods	Washing fastness			Rubbing fastness		Perspiration						Light
	A	C	w	Rd	Rw	Aa	Ca	Wa	Ab	Cb	Wb	
Microwave irradiation	4-5	4-5	4	4-5	3-4	4-5	4	3-4	4	4	3-4	6
Traditional heating	3	2-3	3	2-3	2-3	3-4	3-4	3	3	3	2-3	4-5

A= changing in colour,
 C= staining on cotton,
 W= staining on wool,
 Rd= dry rubbing,

Rw= wet rubbing,
 subscript a= acidic perspiration,
 subscript b= alkaline perspiration.

The results indicate “good to excellent” fastness properties of the dyed samples when using microwave irradiation and “fair to good” when using traditional heating.

Microwaves were found effective in dye extraction from saffron and dye uptake by silk fabrics. The enhanced effect was about 50% more than traditional heating, Other additional features about microwaves are that they are cheaper, more economical and eco-friendly. Saffron shows good fastness properties (washing, rubbing, perspiration and light) of silk fabrics pretreated with chitosan in presence of microwave heating. Compared to conventional methods, microwave dyeing has other advantages such as less power consumption, easy production of desired shades, and quick dyeing, as in the case of silk which takes only 30 minutes, while conventional dyeing requires few hours of heating or boiling. Environmentally friendly pre-treatment of silk fabrics with chitosan instead of using mordant improved the color strength and the fastness properties.

So, that this article offer a new viable method for dyeing silk fabrics by a green strategy and saving energy.

References

- Ali N. F. and E. M. EL-Khatib.(2010).Modification of Wool Fabric to Improve Its Dye ability, *Journal of Natural Fibers*.7:276–288
- Ali N.F., R.S.R. EL. Mohamedy and E. M. El- Khatib. (2011). Antimicrobial Activity of Wool Fabric Dyed with Natural Dyes, *Research Journal of Textile and Apparel*. 15: 1-10
- Bechtold T.. Turcanu.A., Ganglberger, E . & Gessler.S.. 2003, Natural dye in modern textile dye houses- how to combine experiences of two centuries to meet the demands of future? *J.Clean . Prod*, 11: 499-509.
- Bechtold T., Mahmud-Ali, A. & Mussak, R.,2006, Natural dyes for textile. A comparison of methods to asses s the quality of Golden rod plant material . *Dyes and Pigment*, 70: 1-7
- Duran N, Teixeira MFS. Conti R. De, Esposito E. 2002; *Critical Reviews in Food Science and Nutrition*. 42: 53.
- Hebeish A.A.1, N.F. Ali. and J.I. Abd El-Thalouth3, (2012). Green Strategy for Development of Antimicrobial Printed Textile Fabrics, *Research Journal of Textile and Apparel*. 16 :77-81

- Kamel, M.M. R.M. el-Shishtawy, B.M. (2005). Yussef, H. MASHALY, Ultrasonic assisted dyeing III. Dyeing of wool with lac as a natural dye., *dyes and pigments.*, 65:103-110.
- Miquel J, Bernd N, Sempere JM, Diaz (2002), *Alperi J, RamirezA, Gerontol. Arch, Geriatr.* 34: 37–46.
- Singh, O.P. 2000, 'Natural dyes: the pros & cons', *Indian textile journal*, 42.
- Tiwari, Vandana & Vankar, Padma, S. (2001), 'Unconventional natural dyeing using microwave and sonicator with alkanet root bark', *Asian textile journal*, 10:54-57.
- Parton, K. (1997), 'The dyeing of silk: past, present, and future', *JSDC*, 113,. 341.
- Saima Umbreen, Shaukat Ali, Tanveer Hussain and Rakhshanda Nawaz, (2008) "Dyeing Properties of Natural Dyes Extracted from Turmeric and their Comparison with Reactive Dyeing", *Research Journal of Textile and Apparel*, 12: 1-11.
- Teli M.D, Paul, R. and Pardesi, P.D. 2000, Natural Dyes: Classification, Chemistry and Extraction Methods Part 1, *J. Colourage.* 60: 43-48
- Lee H. H., Eom, S. I.. & Kim, J. P. 2001. UV absorber after treatment to improve light fastness of natural dyes on protein fibers., *J. Colour Technol*, 117: 134-138.
- Shin, Y.S. and A. Cho, (2003). Natural Dyeing Using the Colorants Extracted from American Fleabane (I) – Dyeing Properties On Wool. *Journal of korean., Soci. Cloth. Tex.*, 27: 1434-1440.