

# Eco-friendly Dyeing of Cotton Fabric using Ultrasonic Energy with Natural Colorants Extracted from Mulberry Leaves

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**Abstract.** The dyeing of cotton fabrics using mulberry leaves (*Morus alba* L.) as a natural dye has been studied in both conventional heating and ultrasonic techniques. The extractability of Mulberry leaves dye from natural origin using power ultrasonic was also evaluated in comparison with conventional heating. The mulberry leaves extract to provided brown-green to green color, depending on the pH concentration. The results of dye extraction indicate that power ultrasonic is rather effective than conventional heating at low temperature and short time. The effects of dye bath pH, dyeing time, dyeing temperature and mordants were studied and the colorimetric parameters  $L^*$ ,  $a^*$ ,  $b^*$  of dyed fabrics with ultrasonic and conventional techniques were compared.

## Introduction

There is a growing demand for eco-friendly and non-toxic colorants [1]. Dyes derived from natural sources for applications such as food and dyeing of textile have emerged as an important alternative to harmful synthetic dyes [2-3]. Most natural dye colors are found in the roots, bark, leaves, and flowers of plants. However, production cost of natural dyes from direct harvesting are high as compared to production costs from waste and byproducts. Mulberry (*Morus alba* L.) leaves are generally used as a food source of silkworms in Asian regions [4-5]. Use of mulberry leaves as byproduct of silk and food industry is a promising concept which can lower costs involved in natural dye production [6]. The application of ultrasound power to textile dyeing has a significant role in the concept of clean technology for textile processing i.e. enhance the dyeing rate; economize energy and time consumption without causing any apparent fibre damage [7-8]. In this study, the dyeing of cotton fabrics using mulberry leaves as a natural dye has been studied in both conventional heating and ultrasonic techniques

## Experimental Methods

### Materials

The leaves of *Morus alba* L. were collected fresh from Samut Prakan province, Thailand. The cotton fabric, purchased locally, was soak in a detergent solution containing 2.5 g/L at 80 – 100°C for an hour to remove starch and other stiffening agents. The material to liquor (M:L) ratio was maintained at 1:80, after which the fabrics were washed with distilled water.

Mordants such as copper sulphate ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ) and alum [ $\text{Al}(\text{NH}_4)(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ ] were procured Sigma-Aldrich Chemical Co. Distilled water was used in extraction, preparation of all chemical solutions, and dyeing processes.

### Extraction using conventional heating (CH) method

The leaves of *Morus alba* L. were collected fresh and cut into small pieces with average size of 1 cm and used for the experiments. Typically 2 g of sample was taken and 100 ml distilled water was added in a glass beaker. The beaker was covered using aluminium foil to prevent loss of solvent by evaporation. The temperature of the extraction bath for CH process was maintained at 80°C.

*Extraction using ultrasound (US)*

2 g of sample was taken and 100 ml distilled water was added in a glass beaker. The beaker was covered using aluminium foil to prevent loss of solvent by evaporation. The beaker was set in the ultrasonic bath and the ultrasonic power set at 80 W. The temperature was maintained around 55°C. Samples were taken at every 10 min and the optical density was determined with the help of UV-VIS spectrophotometer.

*Spectrophotometric analysis*

The extract samples were analyzed using a UV-visible spectrophotometer JASCO. The UV-VIS spectrum of the extract samples were obtained in the visible region of 400-800 nm. Then the dye present in the extracted solution was analyzed by measuring the absorbance value at a wavelength of peak absorbance at 638 nm.

*Gravimetric analysis*

At the end of the extraction process, the samples taken from both ultrasound and control extracts were filtered and taken in clean, dried and weighed glass vessels. The extracts were dried in a hot-air oven until all the water evaporated and only the extract was left. The vessels were then cooled in a desiccators and the constant weight of the colorant extract obtained per gram of the plant material used were calculated. The yield was calculated using the equation [8]:

$$\% = \frac{\text{natural dye extract obtained (g)}}{\% \quad m \quad f \quad p \quad m \quad s \quad (g)} \quad (1)$$

$$\% \text{ Improvem} \quad \text{sound} = \frac{\% \quad f \text{ (US process-CH process)}}{\% \quad f \text{ CH process}} \quad (2)$$

*Dyeing of cotton fabrics*

The cotton fabrics were dyed in 0.1 M NaOH aqueous solution at a liquor ratio of 30:1 for 1 hour. In case of conventional heating (CH), the dyeing was carried out glass beaker set in a 45°C water bath. In case of ultrasonic dyeing (US), experiments were carried out in a glass beaker set in ultrasonic bath and the ultrasonic power at 80 W. The temperature was maintained around 45°C. After filtration and certain dilution, the optical density of the dye liquor at 638 nm was measured.

*Method of mordanting*

The post-mordanting method involved using 1% solutions each of  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  and  $\text{Al}(\text{NH}_4)(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ . Mordanting was carried out for 30 min at room temperature. The fabrics were then washed and dried.

*Dye absorbance, color strength and color depth measurements*

The absorbance of the dye solutions was recorded before and after dyeing on UV/VIS spectrophotometer. The amount of dye absorbed was calculated by using the relation :

$$\% \text{ Dy} \quad \text{bsorbance} = \frac{\text{abs} \quad f \quad -\text{abs} \quad f}{s \quad f} \times 100 \quad (3)$$

The Color strength and color depth of dyed samples were determined by light reflectance technique using a Hunter Lab spectrophotometer Color Quest XE. The color strength (K/S) value of samples was evaluated using the Kubelka-Munk equation [9];

$$K/S = (1-R)^2 / 2R \quad (4)$$

Where R is the reflectance of the dyed sample; K is the absorption coefficient and S is the scattering coefficient.

The color of the dyed samples are given in CIELab coordinates ( $L^*$ ,  $a^*$ ,  $b^*$ ):  $L^*$  corresponding to the brightness (100 = white, 0 = black),  $a^*$  to the red – green coordinate (+ve = red, -ve = green) and  $b^*$  to the yellow – blue coordinate (+ve = yellow, -ve = green)

### 3. Results and discussion

#### *Extraction of mulberry leaves*

UV-VIS spectrum of natural dye obtained from mulberry leaves was shown in Fig. 1(a). The absorbance values for dye extract obtained by CH method and US method at the wavelength of 638 nm are shown in Fig. 1(b) and 1(c). Extraction of mulberry leaves with alkali condition gives higher yield compared to aqueous extract. The results of gravimetric analysis indicate that there is about 22 % improvement in the % yield of aqueous extract and 23% improvement in the % yield of alkali extract due to the use of ultrasound as compared to the CH process (Table 1). The spectrophotometric analysis showed the similar results (Fig. 1)

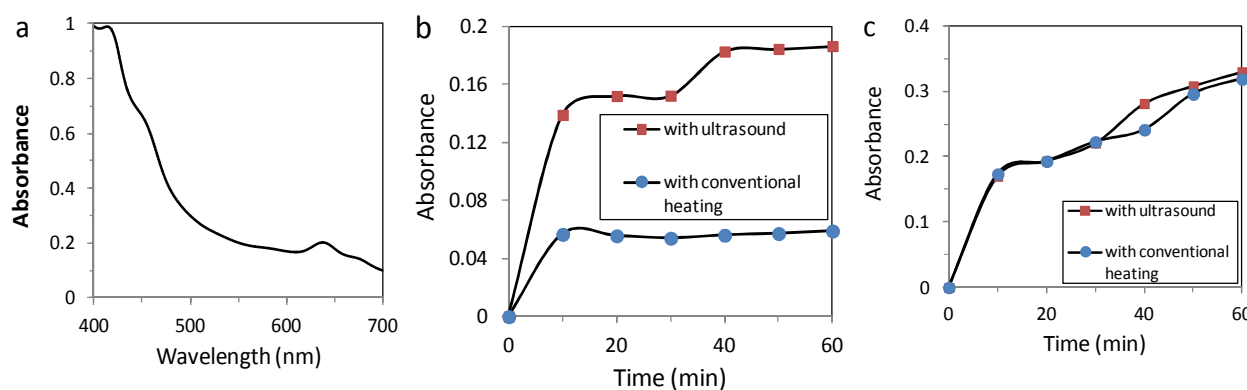


Fig. 1. (a) UV-VIS spectrum for mulberry leaf extracted (1 g in 50 ml 0.1 M NaOH aq.) with ultrasound for 1 hr. (b) The effect of ultrasound, 80 W on extraction of mulberry leaf (1 g in 50 ml water) and (c) The effect of ultrasound, 80 W on extraction of mulberry leaf (1 g in 50 ml 0.1 M NaOH aq.) at  $\lambda_{\max}$  – 638 nm.

Table 1. The effect of ultrasound on the yields

Solvent	CH – yield (%) (A)	US – yield (%) (B)	% improvement due to ultrasound (B-A)/(A) × 100
water	2.25	2.75	22.2
0.1 M NaOH	13	16	23

#### *Dyed Cotton*

As shown in Table 2, dyeing with mulberry leaves extract using ultrasound gives better dye uptake as compared to the CH process (14% improvement in the % dye absorption). The K/S values and CIELab values are also shown in Table 2. It was clear that the color strength increased for cotton dyed with US process. With copper sulphate and alum, brown-green and green color shade are obtained, respectively, whereas non-mordanting showed almost non-color. Hence, this dye, which can be taken in cold method, is well suited for cotton because of its environment friendly due to less of thermal energy.

Table 2. Absorption (%) K/S values and L\*, a\*, b\* values for dyed cotton with mulberry leaves dye

Method	Mordants	Absorbance		Absorption (%)	K/S	Color co-ordinates		
		Before dyeing	After dyeing			L*	a*	b*
CH	Nil				0.9	94.09	2.10	-9.60
	CuSO <sub>4</sub> 5H <sub>2</sub> O	0.9959	0.8908	10.552	12.0	81.00	-11.57	11.04
	Al(NH <sub>4</sub> )(SO <sub>4</sub> ) <sub>2</sub> 12H <sub>2</sub> O				7.70	82.91	-4.32	12.01
US	Nil				1.1	93.88	1.90	-8.98
	CuSO <sub>4</sub> 5H <sub>2</sub> O	0.9959	0.8762	12.021	13.4	79.87	-11.97	11.39
	Al(NH <sub>4</sub> )(SO <sub>4</sub> ) <sub>2</sub> 12H <sub>2</sub> O				8.23	80.31	-3.81	15.18

## Conclusions

It may be concluded that ultrasonic proved effectiveness in the dye extraction and dye-uptake of cotton fabrics with mulberry leaves dye. The enhanced effect was about 23% and 14% more than conventional heating, respectively. This technique in addition to its advantage of saving the processing time and energy offers better environmental impact.

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