



## Optimization of Extraction and Dyeing Conditions of Natural Dye from Ratanjot Roots Using Ultrasonic Waves

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### Authors' contributions

This work was carried out in collaboration among all authors. Author ORD designed the study, performed the statistical analysis, wrote the protocol and wrote the first draft of the manuscript. Authors SB and SG managed the analyses of the study. Author SG managed the literature searches. All authors read and approved the final manuscript.

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### ABSTRACT

The present work involves the optimization of extraction and dyeing recipes to standardize the procedure for natural dye obtained from the roots of Ratanjot (*Onosma echiodes*) using ultrasonic waves. The resultant dye obtained at optimized extraction conditions was applied on wool fabric to study the effect of temperature, dye concentration, time and dyeing pH. Optical density and colour strength (K/S value) were studied for evaluation of optimum extraction and dyeing conditions. It was observed that extraction and dyeing parameters have significant effect on colour characteristics of dyed wool fabric. The optimized extraction conditions were; dye extraction pH 7, extraction time 60 min and temperature 60°C while optimized dyeing parameters were; dyeing temperature 60°C, dye concentration 2 g/g, dyeing time 75 min and dyeing pH 5. It was also observed that the optimum dye extraction time and pH is same with the conventional heating method but there is reduction in the temperature (60°C), dyeing time and dye concentration consumption in ultrasonic extraction and dyeing method, thus saves the energy, time and amount of dye requirement. Therefore, ultrasound wave represents a promising method for increasing diffusion of dye by the effect of cavitation, as well as for improving the effectiveness of processes as compare to conventional heating method.

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## 1. INTRODUCTION

Traditionally, textile artisans used material from natural sources for the purpose of colouring fabric before the synthetic dyes were invented and commercialized. With the change in time period, demand of the textile product increased, manufacturer faced a hard time in fulfilling the need of customers. Textile dyers/ manufacturers moved towards the use of synthetic dye, because of the easy availability of different types of synthetic dye, they produced a wide variety of shades, had shorter as well as simplified dyeing process and the cost being less as compare to natural dyes [1]. Since synthetic dyes are synthesized from petroleum by-products, they produce toxic or carcinogenic amines. Both, the human as well as animals are affected by the use of these chemicals, because of which the need for natural dyes has evolved which possess the health-promoting as well as eco-friendly properties [2]. With the adverse impact of synthetic dyes, the human mentality is shifting towards the organic products, protecting environmental degradation, promoting the use and production of natural dye in textile industries [3]. Therefore, it provides an opportunity to reintroduce the use of natural dyes for dyeing purposes.

Ratanjot is the local name for plants belonging to the borage family (including *Alkanna tinctoria* or *alkannet*). Traditionally, it was used for colouring textiles, food (such as rogan josh), vegetable oils, wines, medicines, cosmetics and varnishes [4]. It is also used to cure eye diseases, bronchitis, abdominal pains, etc. It is a historical natural dye and also holds immense importance in the field of pharmaceutical, cosmetics and food colorants. It is a red dye obtained from the roots of various Boraginaceous plants species [5]. Dark red, purple and browns are the most typical colours achieved. The major constituents of Ratanjot extracts are naphthoquinones. Plant dyes rich in naphthoquinones such as lawsone from henna, juglone from walnut and lapachol from *alkannet* are reported to show antibacterial and antifungal activities [6,7].

Due to the high demand of textile materials in the market, there is needed to develop an effective extraction technique for natural dyes from plant materials. The application of ultrasonic waves is gaining popularity in textile sector. Application of Ultrasonic waves holds a promising future in the

field of textiles industry. Ultrasound has numerous advantages such as energy saving by dyeing at lower temperature or shortens the process; lower the consumption of auxiliary chemical, lesser processing costs, which lead to increase in the competitiveness among the industry, process improvement by controlling the colour shade [8]. One study used ultrasonic cleaner for the extraction of natural dye obtained from *Cochineal* dye. They reported that application of ultrasonic waves improved dyeability as well as colour fastness properties of the dyed fabric [9].

Many research related on the traditional dye extraction of natural dye were reported but dye extraction with ultrasound waves is a novel concept which gave positive benefits on the colour yield, reduce energy and time requirement. In this study, ultrasonic cleaner approach was used in extraction and dyeing process. Ultrasonic cleaner produced uniform and wider waves as compared to ultrasonic probe. As a result, the main objective of the study is to optimize the extraction and dyeing conditions of *ratanjot* using ultrasonic waves.

## 2. MATERIALS AND METHODS

### 2.1 Materials

#### 2.1.1 Raw materials

The Ratanjot plant (*Onosma echiodes*) was collected from Herbal garden of Botany Department, PAU, Ludhiana, and Punjab. The roots were separated from plant and kept for dried under the sun, then ground and stored at room temperature before use. A plain weave wool fabric was procured from the local market of Ludhiana, Punjab for dyeing.

#### 2.1.2 Chemicals

Acetic acid and sodium carbonate were used for regulating pH. These chemicals were supplied by Thames chemicals, Ludhiana. All the chemicals used were of laboratory grade.

### 2.2 Methods

#### 2.2.1 Optimization of extraction conditions

Dye material of 1g was weighed and placed in the beaker along with the 50ml of deionized water. The beaker was then placed into the

ultrasonic cleaner for extraction process. Parameters optimized were dye extraction pH (4-8), extraction time (30, 45 and 60 min) and temperature (30, 45 and 60°C). The extracted dye solution was then strained through filter paper. Dye solution of 0.2 ml each was taken out from dye bath and diluted 100 times. The diluted dye solution was then centrifuged at 3000 rpm for 20 min. The diluted dye solution was poured in the cuvette to measure the optical density of dye solution. The optical density of extracted sample was measured using Labindia UV spectrophotometer at the wavelength range from 400 to 600nm. The sample showing the maximum optical density was considered as optimized value.

### 2.2.2 Optimization of dyeing conditions

After extraction, effects of dyeing parameters such temperature, dye concentration, dyeing time and dyeing pH were noted for process optimization. Wool fabrics were dyed by ultrasonic dyeing method in ultrasonic cleaner using filtered extract at 30, 45 and 60 °C for 1 h for temperature optimization. To study the effect of dye concentration, dyeing was done using 1, 2, 3, 4 and 5 gm of dye material at 60°C for 1 h. In order to observe the effect of dyeing time, dyeing was carried out for 30, 45, 60 and 75 min and 90 min. Different dyeing pH (4, 5, 6, 7 and 8) were used to investigate the effect of pH on dyeing.

### 2.2.3 Colour strength

The colour strength (K/S) of dyed fabrics was measured according to CIE system using Colorflex Hunter Lab. The colour strength values are calculated using the following "Kulbelka – Munk" equation:

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

Where K is the absorption co-efficient, R is the decimal fraction of the reflectance of dyed fabric and S is the scattering coefficient at wavelength of maximum adsorption.

## 3. RESULTS AND DISCUSSION

### 3.1 Optimization of Extraction Conditions

#### 3.1.1 Effect of wavelength on dye extraction pH

The optical density of the dye solution was measured at different wavelength using a UV

spectrophotometer. The optical density of the dye solution decreases with increase in the wavelength up to a certain level, i.e 530 nm, as shown in Table 1. Beyond that the optical density started increasing. The optical density increases with increase in pH from 4 to 7. This trend was studied for the pH values ranging from 4 to 8. It was found that the optical density was maximum at pH 7 and wavelength 540 (nm).

#### 3.1.2 Effect of dye extraction pH

Table. 2 clearly shown that optical density of extracted gradually increase with increase pH up to 7. The maximum optical density value was found at pH7. And, with increase the extraction temperature and time, the optical density value also increases. Hence, pH is considered as optimum dye extraction pH.

#### 3.1.3 Effect of dye extraction time

The optical density of extracted dye gradually increases with increase in extraction time of dye as shown in Table 2. It was observed that the optical density was higher at dye extraction time of 60 min. Therefore, dyeing for 60 min is considered as optimized.

#### 3.1.4 Effect of dye extraction temperature

The optical density of dye solution increases with increase in extraction temperature as shown in Table 2. It was observed that the optical density was maximum at 60°C. With the increase in temperature, cell wall ruptures and more dye component comes out into the extract and dissolves. At high temperature, solubility of dye is increased so it comes more in filtrate and less in residue. One study also reported that the maximum dye exhaustion percentage at dye extraction temperature of 60°C for dyeing cotton and silk yarn with *Acalypha wilkesiana* (copper red colour dye) by using ultrasonic dye extraction method [10].

### 3.2 Optimization of Dyeing Conditions

#### 3.2.1 Effect of dyeing temperature on colour strength

It is evident from the Fig.1 that the colour strength values increases with increase in temperature. It could be due to fibre swelling and deaggregation of dye at higher temperatures.

### 3.2.2 Effect of dye concentration on colour strength

It is clear from Fig.2 that dye absorption increases when the dye material was increased from 1 to 2 g/g of wool fabric, but decrease in dye absorption with increase in the dye concentration from 3 to 5g / 100 g of wool.

### 3.2.3 Effect of dyeing time

As the time for dyeing increase, colour strength value increases but there is a decrease in colour strength by further increasing dyeing time from 75min. This could be due to shift in equilibrium of colorant from fabric to dye bath. From the colours strength values given in the Fig.3, it is clear that dyeing for 75 mins shows higher colour strength. So dyeing for 75 min was considered as optimized.

### 3.2.4 Effect of dyeing pH on colour strength

It is inferred from Fig.4 that there is gradual increase in dye absorption when dyeing pH increase from 5 to 6 but decrease from 6 to 8. Since, pH6 is considered as optimum dyeing pH for ratanjot dyed wool fabric.

### 3.3 Comparison of Traditional and Ultrasonic Dyeing Methods of Ratanjot Natural Dye

The optimized extraction conditions of traditional / conventional heating dyeing method were; dye extraction pH 7, extraction time 60 min and temperature 100 °C while optimized dyeing parameters were; dyeing temperature 100°C, dye concentration 3g/g, dyeing time 90 min and dyeing pH 7 as described by previous research work [11]. The optimum dye extraction time and pH is same with the application of ultrasonic waves but there is reduction in the temperature (60°C), dyeing time and dye concentration in ultrasonic extraction and dyeing method, thus saving the energy, time and amount of dye requirement. Similarly one study optimized dyeing conditions for cotton by using reactive dyes with the application of ultrasonic wave. The optimum dyeing conditions were 1 per cent dye concentration, 45-55°C dyeing temperature and 60 minutes dyeing time by using ultrasonic dyeing technique, whereas, in conventional dyeing technique, the optimum dyeing conditions were 2 per cent dye concentration, 100°C dyeing temperature and 120 minutes dyeing time [12].

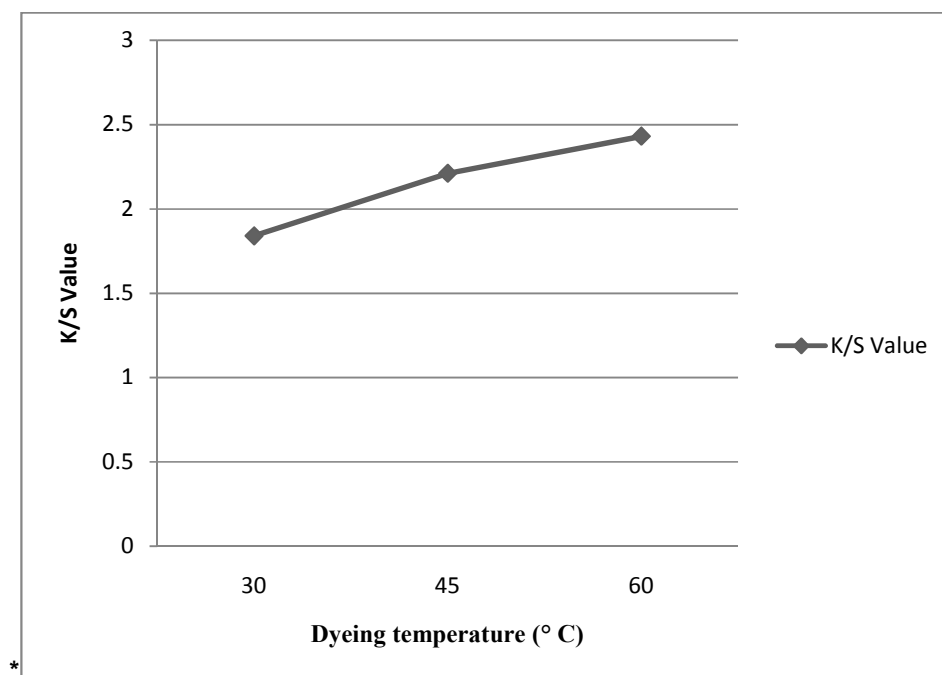


Fig. 1. Effect of dyeing temperature on colour strength (Dyeing done at dyeing pH 7, dyeing time 60 min and dye concentration 1 g/g)

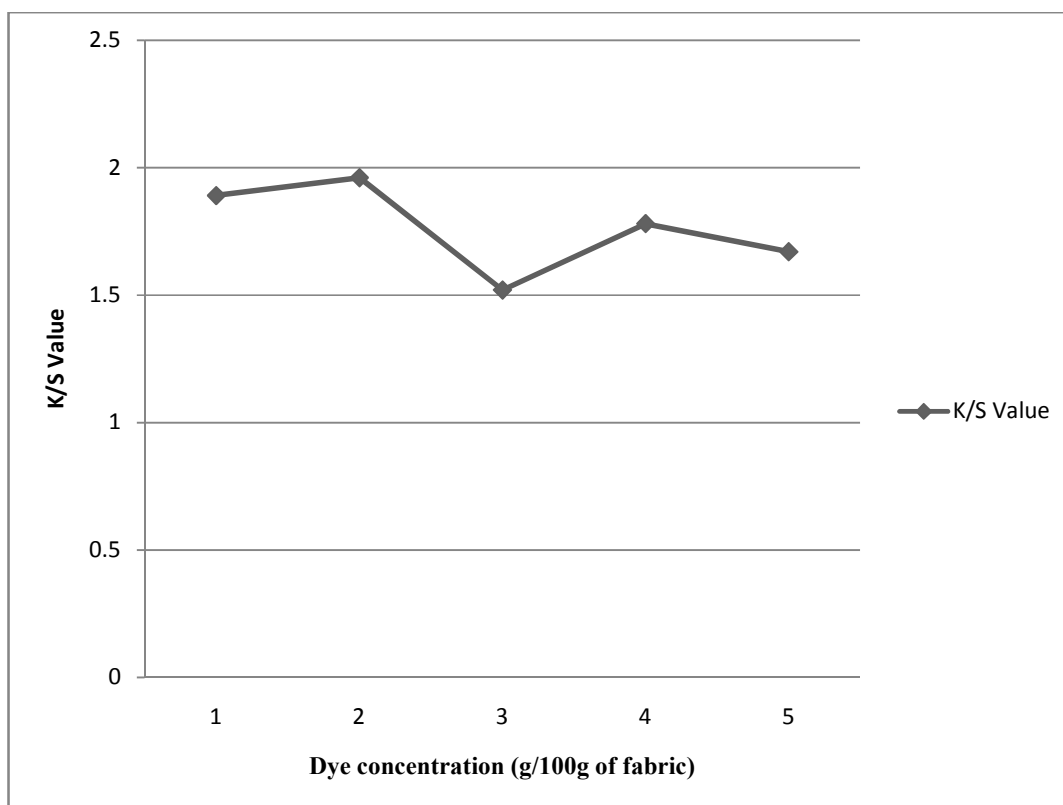


Fig. 2. Effect of dye concentration on colour strength (Dyeing done at dyeing pH7, dyeing time 60 min and dyeing temperature 60°C)

Table 1. Effect of wavelength on dye extraction pH

| Wavelength(nm) | pH media |       |       |       |       |
|----------------|----------|-------|-------|-------|-------|
|                | 4        | 5     | 6     | 7     | 8     |
| 400            | 0.329    | 0.417 | 0.546 | 0.761 | 0.528 |
| 410            | 0.297    | 0.376 | 0.456 | 0.693 | 0.449 |
| 420            | 0.297    | 0.366 | 0.422 | 0.630 | 0.433 |
| 430            | 0.295    | 0.339 | 0.424 | 0.623 | 0.452 |
| 440            | 0.294    | 0.337 | 0.401 | 0.596 | 0.448 |
| 450            | 0.259    | 0.296 | 0.360 | 0.531 | 0.388 |
| 460            | 0.173    | 0.227 | 0.256 | 0.433 | 0.307 |
| 470            | 0.180    | 0.223 | 0.256 | 0.426 | 0.299 |
| 480            | 0.250    | 0.296 | 0.298 | 0.477 | 0.368 |
| 490            | 0.252    | 0.307 | 0.313 | 0.478 | 0.383 |
| 500            | 0.253    | 0.297 | 0.304 | 0.461 | 0.365 |
| 510            | 0.241    | 0.290 | 0.286 | 0.444 | 0.363 |
| 520            | 0.232    | 0.274 | 0.269 | 0.408 | 0.339 |
| 530            | 0.213    | 0.251 | 0.241 | 0.389 | 0.352 |
| 540            | 0.814    | 0.830 | 0.833 | 0.992 | 0.908 |
| 550            | 0.791    | 0.808 | 0.806 | 0.960 | 0.883 |
| 560            | 0.771    | 0.787 | 0.781 | 0.931 | 0.858 |
| 570            | 0.749    | 0.765 | 0.757 | 0.900 | 0.836 |
| 580            | 0.728    | 0.741 | 0.730 | 0.870 | 0.809 |
| 590            | 0.705    | 0.716 | 0.702 | 0.838 | 0.783 |
| 600            | 0.684    | 0.695 | 0.678 | 0.810 | 0.759 |

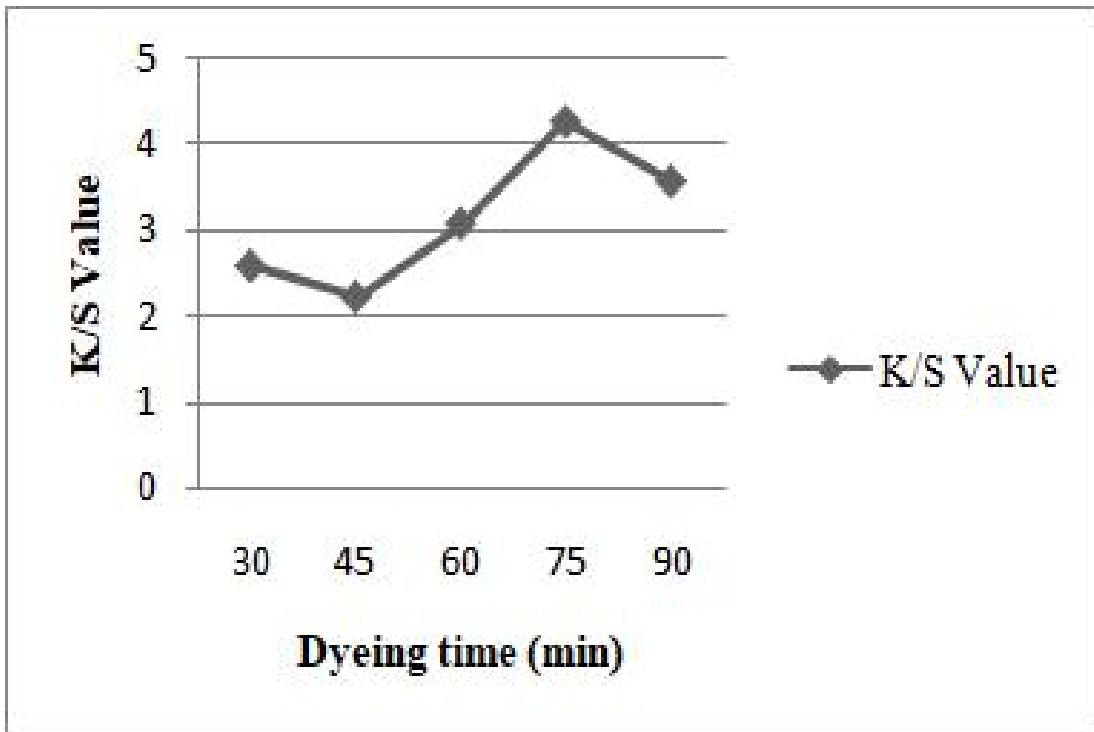


Fig. 3. Effect of dyeing time on colour strength (Dyeing done at dyeing pH7, dye concentration 2 g/g and dyeing temperature 60°C)

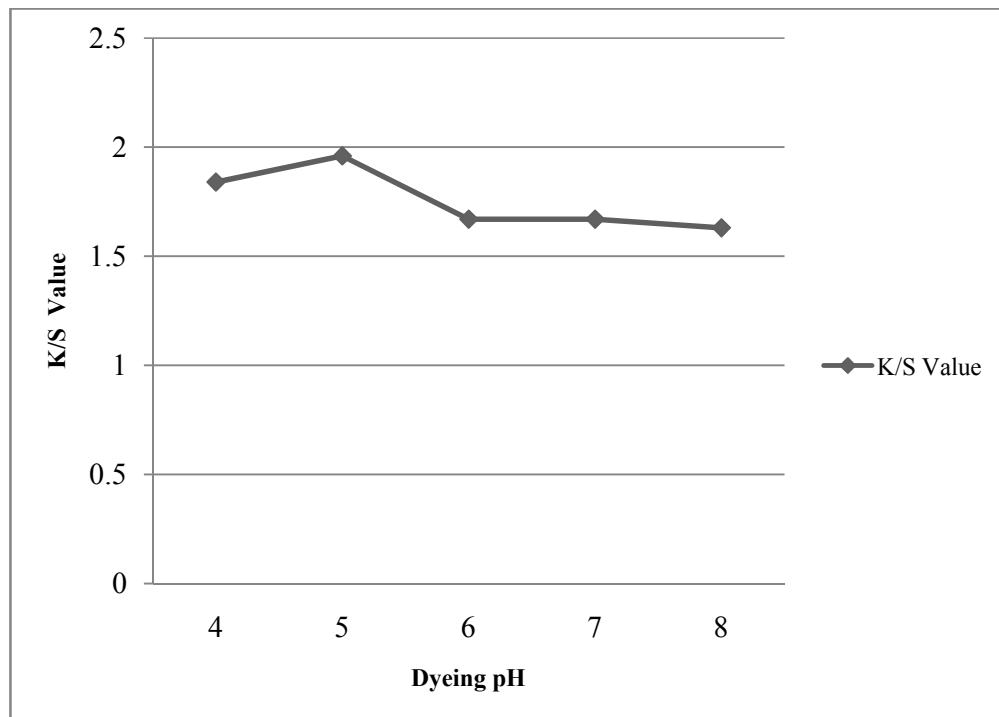


Fig. 4. Effect of dyeing pH on colour strength (Dyeing done at dye concentration 2g/g, dyeing time 75 mins and dyeing temperature 60°C)

**Table 2. Optimization of extraction conditions**

| Time (mins) | Temperature (°C) | pH    |       |       |       |       |
|-------------|------------------|-------|-------|-------|-------|-------|
|             |                  | 4     | 5     | 6     | 7     | 8     |
| 30          | 30               | 0.667 | 0.678 | 0.684 | 0.810 | 0.759 |
|             | 45               | 0.684 | 0.695 | 0.702 | 0.870 | 0.783 |
|             | 60               | 0.716 | 0.781 | 0.787 | 0.908 | 0.810 |
| 45          | 30               | 0.705 | 0.716 | 0.730 | 0.904 | 0.809 |
|             | 45               | 0.728 | 0.741 | 0.757 | 0.943 | 0.836 |
|             | 60               | 0.808 | 0.833 | 0.808 | 0.985 | 0.870 |
| 60          | 30               | 0.771 | 0.787 | 0.781 | 0.909 | 0.858 |
|             | 45               | 0.791 | 0.808 | 0.806 | 0.982 | 0.883 |
|             | 60               | 0.814 | 0.830 | 0.833 | 0.990 | 0.908 |

The application of ultrasonic waves improved extraction of dyes from different parts of various plants resources. The improvement could be due to better discharges of natural dyes materials from cell wall membranes of plants and mass transfer to solvent assisted by cavitation provided by ultrasonic waves.

#### 4. CONCLUSION

Optimization of dyeing conditions is essential to minimize the investment cost and to avoid discrepancy in the dyed fabric quality. The study reveals that extraction and dyeing parameters have significant effect on colour strength and quality of wool fabric. The optimized extraction conditions were; dye extraction pH, extraction time 60 min and temperature 60 °C while optimized dyeing parameters were; dyeing temperature 60°C, dye concentration 2g/g, dyeing time 75 min and dyeing pH 5. When compare the dyeing conditions with conventional heating method, the optimum dye extraction time and pH is same, however, there is reduction in the temperature (60°C), dyeing time and dye concentration consumption in ultrasonic extraction and dyeing method, thus saving the energy, time and amount of dye requirement. Therefore, the application of ultrasound waves represent a promising method for increasing diffusion of dye by the effect of cavitation, as well as for improving the effectiveness of processes.

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#### COMPETING INTERESTS

Authors have declared that no competing interests exist.

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