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Ultrasonic dyeing of cotton fabric with roots of ratanjot (*Onosma echioides)* natural dye

Oinam Roselyn Devi[1*], Sandeep Bains[2], Sumeet Grewal[3]
[1] Research Scholar, Department of Apparel and Textile Science, College of Community Science, Punjab Agricultural University, Ludhiana, Punjab, India
[2] Professor, Department of Apparel and Textile Science, College of Community Science, Punjab Agricultural University, Ludhiana, Punjab, India
[3] Assistant Professor, Department of Apparel and Textile Science, College of Community Science, Punjab Agricultural University, Ludhiana, Punjab, India

Abstract
Traditionally, the dyeing of natural dye from plant materials requires longer time and higher temperature to get good colour fastness. Thus, this study explored a suitable technique for more efficient natural dyeing using mordant to improve colour fastness of natural dyes. This study used ultrasound wave as a technique for dyeing the colourant from a selected plant (Ratanjot) to a selected fabric (cotton) and compared the conventional and ultrasonic dyeing methods in terms of its colour strength, CIE Lab values and color fastness to lightness, washing, rubbing and perspiration. The dye uptake percentage was found to be maximum by using ultrasonic dyeing technique as compared to conventional dyeing technique. Moreover, the cotton samples achieved by ultrasonic dyeing technique gave darker shade. Also, the fastness grades to light, washing, rubbing and perspiration also improved for the samples dyed with ultrasonic waves even at lower temperature. Therefore, the utilization of ultrasound wave for dyeing of natural dye found to be significantly improved in dye uptake and colour fastness with lower dyeing temperature compared to conventional dyeing technique.

Keywords: conventional dyeing, natural dye, ratanjot, ultrasonic wave

Introduction
India is one of the richest countries in the world in terms of biodiversity. India houses a wealth of beautiful floral resources; a wide range of natural products (*flora and fauna*) can be found. India has been the largest textile exporter for hand woven and naturally processed dyes. Nearly 450 plants are found to be good source of natural dye in India. These dyes are very popular for their soft, lustrous colour endurance and biodegradable nature. Natural dyes are very soothing to the human eyes as compared to synthetic dyes. Therefore, the use and production of natural dyes is growing day by day in textile sector. Natural dyes has been used for producing value added diversified products by the printers, craft dyers, artisan community and in the handloom sector [1]. The reason behind is that these are eco-friendly, nontoxic, biodegradable, natural and impart no allergic reactions which are associated with the synthetic dyes.

The name *Ratanjot* is attributed to the roots of various *Boraginaceous* plant species. It was regarded as one of the important herbal drugs of indigenous systems of medicine. It is used for imparting pleasing red colour to foodstuff, oils and fats. The root, which forms the actual drug, is considered to be anthelmintic, antipyretic, antiseptic and claimed to be useful in treating the diseases of the eye, bronchitis, abdominal pain, itch, etc. [2]. Previously, ratanjot was reported as one of the potential natural dyes sources by many researchers [3].

Recently, there is a growing demand for developing suitable efficient dyeing technique for natural dyes from plant materials. Ultrasonic approach has been widely used in food industry and also in textile industry. Ultrasonic cleaner has a more uniform and wider waves compared to ultrasonic probe and ultrasonic-assisted extraction based on central composite design. Ultrasound dyeing method save energy by dyeing at lower temperature or shortens the process, lower the consumption of auxiliary chemical, lesser processing costs, which lead to increase in the competiveness among the industry, process improvement by controlling the color shade [4]. In this study, ultrasonic bath approach was used in both extraction and dyeing process. One study used ultrasonic bath for dyeing cotton fabric using colourant from Cochineal dye and reported that ultrasound approach improved dyeability as well as colour fastness properties of dyed fabric [5]. Another study also used ultrasonic bath for dyeing the silk fabric with the sticta coronate lichen. They reported that the use of ultrasound wave improved the dye uptake with lower temperature (60 °C). Normally, conventional dyeing temperature for silk fabric is 60 to 80 °C [6]. It is known that dyeing at high temperature for longer period of time tends to decrease the strength of silk fabric.

In view of the above, the present study was undertaken to compare the dyeing and fastness properties of cotton and wool fabric using ratanjot as natural dye in both conventional and ultrasonic methods.

Materials and Methods
Materials
Collection of raw materials
The cotton fabric, natural mordants namely amla and babool powder were purchased from local market of Ludhiana, Punjab.

Roots of Ratanjot plant were dried under the sun and finely ground into powder form. The chemicals namely urea, sodium chloride, sodium carbonate, acetic acid, synthetic mordants (alum and tannic acid) were supplied by Thames Chemicals, Ludhiana, India.

Preparation of samples

In order to remove impurities from the fabric, the cotton was soaked in a warm 0.5 percent solution of a mild non-ionic detergent for 3 hours keeping M: L ratio of 1:30. After kneading and squeezing, the cotton fabric was rinsed in tap water and dried in shade. Since cotton had less affinity towards natural dyes, therefore, the cotton was again pre-treated with 20 per cent of myrobalan, soaked for overnight, squeezed and lastly dried in the sun [7].

Methods

Optimisation of dyeing variables

Experiments were conducted for optimizing the dyeing and mordanting conditions of cotton with roots of ratanjot dye using four different natural and synthetic mordants. The conditions namely - dye material extraction pH (4-8), dye extraction time (15, 30, 45, 60 minutes), dye extraction temperature (30, 45 and 60 °C), dyeing concentration (1, 2, 3, 4 and 5g/g), dyeing time (30,45, 60, 75 and 90 minutes), dyeing pH (5-9) and dyeing temperature (30, 45 and 60 °C) were optimized.

Optimisation of mordant concentration

Mordant concentration of 0.1, 0.2, 0.3, 0.4 and 0.5gm of amla and babool mordants were mordanted simultaneously at optimum dyeing pH, for optimum time at optimum temperature. The concentrations of alum were 5, 10, 15, 20 and 25gm/100g of fabric. The concentrations of tannic acid were 1, 2, 3, 4 and 5 per 100g of fabric. The sample giving maximum K/S value was taken as optimum mordant concentration.

Final dyeing of cotton fabric

Conventional dyeing methods

Conventional dye extraction was carried out as described by Bains *et al*., (2002) [3]. The optimized dyeing conditions for conventional dyeing techniques were dye concentration of 3%, dyeing pH 7 dyeing time of 45 min and 100 °C dyeing temperature.

Ultrasonic Dyeing methods

The extracted dye solution was poured in the ultrasonic bath and maintained the MLR of 1:250. A fabric strip / pre-soaked sample was fixed on a stainless-steel frame and immersed into the dye solution and subjected to ultrasound at optimized temperature for optimized time. The simultaneous mordanting method was performed at optimized mordant concentration. After dyeing, the baths were allowed to cool down for some time to avoid sudden change during rinsing. The samples were taken out, washed in mild detergent solution and rinsed in tap water to remove unfixed dye material. After thoroughly rinsing, the samples were finally dried in shade. The dyeing with ultrasound was carried out at 220V/50Hz.

Colour strength and CIE Lab values

The colour yield of the dyed fabrics was evaluated by Colorflex Hunter Lab in terms of CIE Lab values (L*, a*, b*) and colour strength (K/S) values. The colour strength values of the dyed fabrics were calculated using the 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 scatting coefficient at wavelength of maximum adsorption.

Colour fastness tests

The light, wash, rubbing and perspiration fastness values of all dyed samples were determined by Digital light fastness tester, launder meter, crock meter and per spirometer, tested according to ISO Bureau of Indian Standard.

Result and Discussion

Various experiments were carried out in order to determine the role of ultrasound wave for dyeing of *Ratanjot* dye on cotton in terms of dye uptake and colour fastness properties.

Optimised dyeing conditions

Table 1 and 2 shows the optimum dyeing conditions of ratanjot roots dye and observed that the maximum dyeability was found at dye extraction pH 7, dye extraction time of 60 minutes and dye extraction temperature 60 °C. And, the maximum dye absorption on cotton was achieved by using dye material concentration 3g/g of fabric, at dyeing pH 6, dyeing temperature 60 °C and dyeing time for 45 minutes. The optimum mordant concentration values have been presented in table 3. The optimum mordant concentrations were found to be 0.5 for amla, 0.4 for babool powder, 10 g for alum and 2g for tannic acid respectively.

Table 1: Optimized dye extraction conditions

Dye extraction conditions	Optimum Value
Dye extraction pH	7
Dye extraction time (min.)	60
Dye extraction temperature (°C)	60

Table 2: Optimised dyeing conditions on cotton

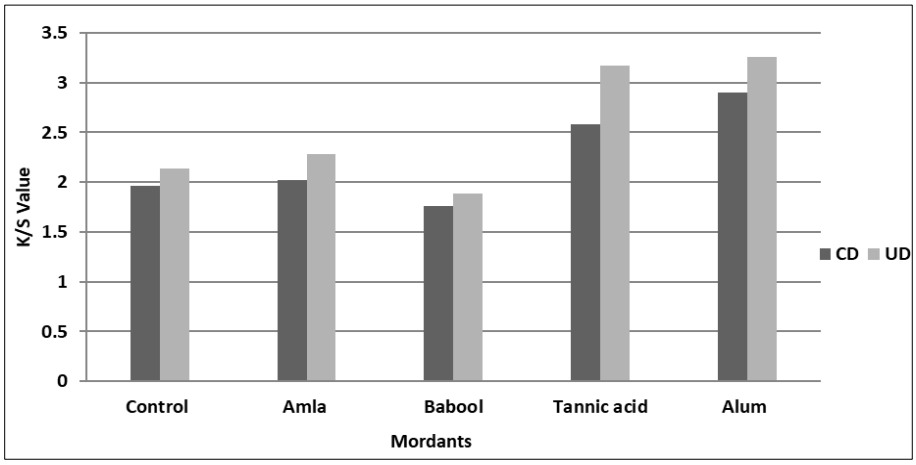
Dyeing conditions	Optimum value
Dye concentration (g/g)	3
Dyeing pH	6
Dyeing temperature (°C)	60
Dyeing time (min.)	45

Table 3: Optimised mordant concentrations on cotton

Mordants	Optimum value (g/g)
Amla	0.5
Babool	0.4
Alum	10
Tannic acid	2

Colour strength

The colour strength (K/S values) of dyed fabric using both conventional and ultrasonic dyeing methods is shown in Fig.1. The K/S values of dyed fabric achieved by ultrasound were higher as compared to dyed fabric achieved by conventional dyeing. The reason may be due to acceleration of dye movement from solution to a fibre by ultrasound energy and reaction take place between dye molecules and cellulose OH groups. Among the four mordants, the K/S value was found to be maximum in dyed fabric mordanted with alum. One study reported that higher dye uptake in cotton fibre when dyed with Parijataka (*Nyctanthes arbor tristis*) flower by using ultrasonic dyeing method (64.55) as compared to conventional dyeing method (53.71) [8].



CD = Conventional dyed samples, UD = Ultrasonic dyed sample

Fig 1: Comparison of the K/S value of Conventional and ultrasonic dyed fabrics

Colour measurement

The measurement of chromaticity values such as L*, a* and b* was used to evaluate the dyed cotton fabrics obtained by using both ultrasonic and conventional dyeing method. Tables 4 shows the values of L*, a*, b* and c* obtained from measurement using Color Flex Hunter Lab based on CIE system.

The colour measurement of dyed cotton fabric using ultrasonic and conventional dyeing method is tabulated in table 4. The value of L* indicate lightness and darkness effect for dyed fabric and found to be highly significant in dyed fabric achieved by conventional dyeing method which indicated that the ultrasonic dyed fabric gave a darker shade. This was supported by one study that dyed cotton gave a deeper shade with the use of ultrasound compared to that of conventional dyeing technique [9]. By using ultrasound dyeing technique, dyed cotton fabric showed more redness and yellowish effect compared to that of conventional dyeing technique. The C* values of ultrasonic cotton dyed samples are found to be higher than the samples obtained by conventional except the sample mordanted with tannic acid, which indicates that the samples obtained by ultrasonic wave are more saturated than conventional boiling methods.

Table 4: Chromaticity values of the dyed cotton fabrics

Samples	L*		a*		b*		C*	
	CD	U D	C D	U D	C D	U D	CD	U D
Unmordanted	69.63	69.22	-0.40	3.36	4.24	4.35	4.22	5.49
Amla	73.59	71.58	0.82	-0.61	9.33	14.44	9.37	14.43
Babool	67.38	63.21	4.41	5.80	0.41	2.64	4.42	6.37
Alum	67.80	67.22	4.07	4.72	1.07	4.54	4.21	6.53
Tannic Acid	71.10	63.39	2.24	2.70	7.94	6.64	8.25	7.17

Colour Fastness

Table 5 shows the comparison between fastness properties of dyed cotton fabrics using ultrasonic and conventional method.

The light fastness grade of dyed cotton was improved from poor to fair using ultrasound. The wash and rubbing fastness for dyed cotton fabric was good. However, by using ultrasonic dyeing technique, fastness properties to washing and rubbing were slightly improved. Excellent grades (5) were observed for ultrasonic dyed samples in acidic medium. In alkaline medium, the perspiration grade was good which improved to good to excellent (4/5) ultrasonic dyeing.

Table 5: Colour fastness grades of conventional and ultrasonic dyed cotton using Ratanjot dye

Sample	Light fastness grades	Washing fastness grades	Rubbing fastness grades		Perspiration Fastness Grades	
			Dry	Wet	Acidic	Alkaline
Unmordanted						
CDS	2	4	4/5	4/5	5	5
UDS	2	4/5	5	5	5	5
Amla						
CDS	3	5	5	4/5	5	4
UDS	¾	5	5	5	5	4/5
Babool						
CDS	2	4	5	5	5	5
UDS	2	5	5	5	5	5
Alum						
CDS	2	3	4/5	4	3	4
UDS	2	3/4	5	5	4	4/5
Tannic acid						
CDS	2	4	4/5	4	5	4
UDS	3	4	5	5	5	4

CDS = conventional dyed sample, UDS = ultrasonic dyed sample

Conclusion

The use of ultrasonic dyeing technique for the dyeing of cotton fabric with Ratanjot dye *(Onosma echioides)* was found to have

significant improvement in the percentages of dye absorbed to fabric and fastness properties of dyed cotton fabric with lower temperature compared to conventional dyeing technique. Therefore, this technique in addition to its advantage of saving the energy offers better environmental impact as it helps much in improving dye-uptake.

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