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Ecofriendly ultrasonic natural dyeing of wool fabric with natural dyes obtained from *wrightia tinctoria*

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ABSTRACT

Natural dyes are extracted from plants, insects and minerals. Certain limitations of the natural dyes, they were withdrawn with the invention of synthetic dyes. In the long run, synthetic dyes were found to be harmful to the chemicals. As a result natural dyes have come to be used for their many intrinsic values. The main reason being, then availability of local plants as the main source of natural colorants. Almost all the parts of the plants, namely stem, leaves, fruits, seeds, barks etc are used for extracting natural colour. In addition, they are antimicrobial antifungal, insect repellant deodorant, disinfectant and they also have medicinal values. The present study was conducted to evaluate the colouring component and extraction method of plant dyes. Leaves of the plant are the source of dye. This dye was used to dye wool by applying different mordanting methods with different mordant. Their easy availability in the country being zero cost effective and planted for other purposes are the main reasons for utilizing them as natural dyes.

KEYWORDS: Ultrasonic dyeing; Natural dye; *Wrightia tinctoria*; wool

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INTRODUCTION

Wrightia tinctoria belongs to the family of apocynaceae [1]. This is a small deciduous tree. It generally grows up to 1.8 m tall and often under 60 cm girths, sometimes upto 7.5 high and it in distributed all over India. “Indrajav” is its common name. It is known for its traditional heading quality and it is widely recognized as a medicinal plant [2]. As it is a medicinal plant, almost every part is useful pungent leaves chewed for relief from tooth ache; bark and seeds are antidysenteric, antidiarrhoel, and antihaemorragic [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 mainly in food industry [4] and also in textile industry. There are reports stated that the ultrasound approach improved dyeability as well as enhancement in the fastness properties of the dyed fabric [5]. They reported that the use of ultrasonic energy showed a significant improvement in the dye uptake representing the sonication efficiency in textile dyeing with lower temperature (60^{o}C). Normally, conventional dyeing temperature for wool fabric is 60 to 80^{o}C. Dyeing temperature and time are important parameters which influenced silk dyeing. It is known that dyeing silk at high temperature for a long period of time tends to decrease the strength of wool fiber [6]. In this study ultrasonic cleaner approach was used in both extraction and dying process. Using ultrasonic in dying process can improve efficiency dye uptake to fabric and color fastness properties without longer dyeing time and higher temperature compared to traditional dyeing method [7].

MATERIALS AND METHODS

Chemicals

Laboratory grade chemicals Alum, Stannous chloride, potassium dichromate, Nickel suphate, copper sulphate and ferrous sulphate were supplied by Maruthi trader Agencies, Kancheepuram. A natural mordant myrobolan, Turmeric, Cow dung, Banana sap juice was used for the study.

Source

Wrightia tinctoria leaves were collected from padavedu, Thiruvannamalai district.

Figure 1: *Wrightia tinctoria. L* tree

Figure 2: Leaves of *Wrightia tinctoria*

Figure 3: Dry leaves of *Wrightia tinctoria*

Substrate

100% wool cloth was purchased from kamala Departmental Stores, Thanjavur.

Equipment used in the present work

Weighing balance (Ciezen)

Codyson CD-4820 Ultrasonic Cleaner

Extraction of Dyes

Wrightia tinctoria leaves were cleaned by washing with water in order to remove dirt as shown in Fig.2. The cleaned leaves were dried under direct sunlight as shown in Fig.3. Then the leaves were ground into very small pieces in a grinder as shown in Fig 6. A fine strainer was used to remove the wastages. Finally, the contents were weighted. After all these, process, 1 kilogram leaves weighed 318 gram. Then, it is put in 75% ethanol 25% water and heated in a breaker which in kept over a water bath for 2 hour. After this the contents were filtered and kept in a separate beaker.

Effect of M: L Ratio

The wool samples were dyed with dye extracts keeping various M: L ratio as 1:10, 1:20, 1:30 and 1:40. It was observed that the dye uptake was good in M: L ratio 1:30.

Dyeing of Fabrics

The 100% bleached wool fabrics were dyed with the dye extract keeping M: L Ratio at 1:30. Dyeing was carried out at 60^oC and continued for 1 hour in ultra sonicator.

Mordanting

Different metallic salts and natural mordant were used to dye bleached wool fabrics.

(i) Pre-mordanting

Bleached wool fabric was mordant before dyeing using 3% of any one of the chemical mordant like alum, stannous chloride, potassium dichromate, ferrous sulphate, nickel sulphate, copper sulphate and natural mordant like myrobolam, turmeric, cow dung and banana sap juice at 60^oC for 1 hour with MLR of 1:30. The wool fabric thus treated with metal salts were dyed using the dye extract.

(ii) Post- mordanting

Bleached wool fabrics dyed with dye extract were made to become wet and put into different dye baths which contains the required amount of dye extract and water. Acetic acid was added to it after 20 minutes. The wool fabric was dyed for about one hour at 60^oC. The fabrics thus dyed were removed, squeezed and put to treatment with metal salts without washing. Different metal salts were used for treatment using 3% of any one of the chemical mordant like alum, stannous chloride, potassium dichromate, ferrous sulphate, nickel sulphate, copper sulphate and natural mordant such as myrobolan, turmeric, cow dung, Banana sap juice at 60^oC for 30 minutes with MLR of 1:30. The dyed fabrics were washed repeatedly in all the three methods in water and dried in air. At last, the dyed fabrics were put to soap with 2gpl soap solution at 60^oc for 10 minutes. The fabrics were repeatedly washed in water and dried under sum.

(iii) Simultaneous mordanting

Here, the treatment of bleached wool fabrics in carried out simultaneously using dye extract and metal salt using 3% of any one of the chemical mordant like alum, stannous chloride, potassium dichromate, ferrous sulphate, nickel sulphate, copper sulphate and natural mordant such as myrobolan, turmeric, cow dung , Banana sap juice at 60^oC for 1 hour with MLR 1:30.

Dyeing in sonicator

Ultrasound mainly generates cavitations in liquid medium and thus it enhances a wide variety of chemical and physical processes. To suitably induce cavitations, sonicator of 20 KHz frequency is used [8]. To facilitate better dye uptake, cavitations should cause formation and collapse of micro bubbles. For this sonicator is found to be very effective show in Figure 4. Initially the micro bubbles are unstable and gradually grow in the process of oscillation. At test, improve micro bubbles implode violently and thus generate high pressures and temperature which are localized momentarily chemical reaction takes place between the fabric and the dye by this activated state as it results in forming shock waves and severe shear force which in capable of breaking chemical bonds.

Figure 4: Dyeing in sonicator

Effect of ultra sound

Ultrasound dyeing process is preferred because better dye uptake is ensured even in lower temperature and lower chemical combinations; most of the textile industries are highly impressed and interested in adopting ultrasound technique because of this advantage. But, in spite of the better results from lat- scale studies, the ultrasonic backed wet textile methods have not been implemented on an industrials scale. The main aim of this study is developing a specific ultrasonic energy device to be adapted on to the jigger or winch for industrial in liquid media is the source of sonochemical activity. The acoustic cavitations occurring near a solid surface generates micro jets. Then micro jets will effectively facilitate the liquid to move with a higher velocity. It results in the diffusion of solute considerably inside the pores of the fabric. Localized rise in temperature and swelling effect due to ultrasound in the sonication process may result in the improvement of diffusion. The bubbles remaining in stable cavitations oscillate and it results in the enhancement of molecular movement and string effect of the ultrasound. In particular, the effect of stable cavitations cans the realized at the interface of the fabric and dye solution in dyeing wool fabrics. As a result of the intense and accelerated mass transport, wet transport using a conventional approach like increasing temperatures is not always feasible. This study of wring ultrasonic dyeing process has arrived at a conduction that exhaustion of dyes after an hour of dyeing in ultrasound method was 45% and 25.5% while it was

89% and 84% by using the conventional method. The dyeing process of pre – treated fabrics with natural dyes in a sonicator bath at 1:30 ratio commences with the temperature of the dye bath nothing less than 60^0C. This temperature is maintained for one hour. The dye uptake in the sonicator dyeing process showed considerable acceleration.

Colour fastness

The dyed samples were tested according to IS standards. Colour fastness to washing, light and rubbing, perspiration were determined from standard test methods IS-105-C03, IS-2454-85, IS-766-88 and IS-105-E04 respectively.

Measurements of colour strength

The K/S value of the undyed and dyed wool fabrics was determined [9] by measuring surface reflectance of the samples using a computer-aided Macbeth 2020 plus reflectance spectrophotometer, using the following Kubelka Munk equation with the help of relevant software:

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

Where R is the decimal fraction of the reflectance of the dyed samples at λ. K is the absorption coefficient and S is scattering coefficient [10].

RESULTS AND DISCUSSION

Wrightia tinctoria leaves discharged colour easily in alcoholic water. If the quantify of leaves was increased from 5g to 20g in 100 ml (75%alcohol and 25% water) boiled for one hour, it showed an increase in colour strength and also depth in colour. The colour thus obtained was blue.

As power ultrasound offers potential cost savings in time, chemicals, energy and reduced effluent [11, 12], it has been recognized for many years. Hence, its utilization in textile coloration loading to better dyeing and less dye effluent based on the negative nature of both dye and substrate by using wool for enhancing its dye ability with natural dye becomes as study of intetst.

Preparation and optimization aqueous extract of Wrightia tinctoria

Alcoholic leaves extract of *Wrightia tinctoria* were found to discharge colour in 75% ethanol very easily. Increasing the quantity of leaves 5 g to 20 g per 100 ml (75%alcohol and 25% water) water boiled for 1 hour is accompanied with the increase in colour strength and depth in colour [13]. It was observed that, colour of the dye extract was dark blue colour as shown in Figure: 6

Figure 5: Alcoholic extract from the leaves of *Wrightia tinctoria*

Dyeing behavior of the extract

The dye extract was found to be suitable for wool. The wool fabrics were dyed with chemical and natural mordants. It was observed that, the dye uptake was found to be good in post-mordanting method is shown in Figure 11.

Effect of dye bath pH

P^H values of the dye bath having a considerable effect on the dye ability of wool fabrics with natural dye under both US and CH conditions is shown in fig.6. US have clearly improved the dye ability of the fabrics at P^H 3 and 3.5 values. The P^H decreases the dye ability under both CH and US conditions. The effect of dye bath P^H may be due to the correlation between the dye structure and that of wool fibres.

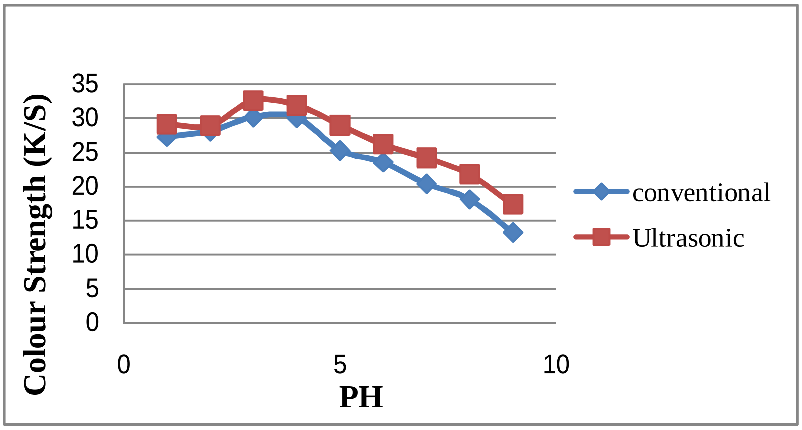


Figure 6: Effect of dye bath pH on the colour strength of dyed wool fabrics.Dyeing conditions: 500 W, MLR 1:30, 30 ml aqueous dye Extracts (3% w/v), 1 h, at 60^0C.

Effect of dyeing time

In order to assess the effect of dyeing time, high concentration of the dye at 3g/100 ml water for revealing the effect of power ultrasonic on the de-aggregation of dye molecules in the dye both by higher dye-uptake is applied. The colour strength thus obtained increased in proportion to the increase in time in both US and CH methods a shown in fig 8, with much higher colour strength at all points in the US case. In the US methods, a plateau is attained after to upto 70 minutes. After this a slight decline started as the time was prolonged. But, in the CH case, the decline in colour strength started only after 60 minutes. Such decline in the dye ability may be due to the description of the dye molecules as a result of long dyeing time.

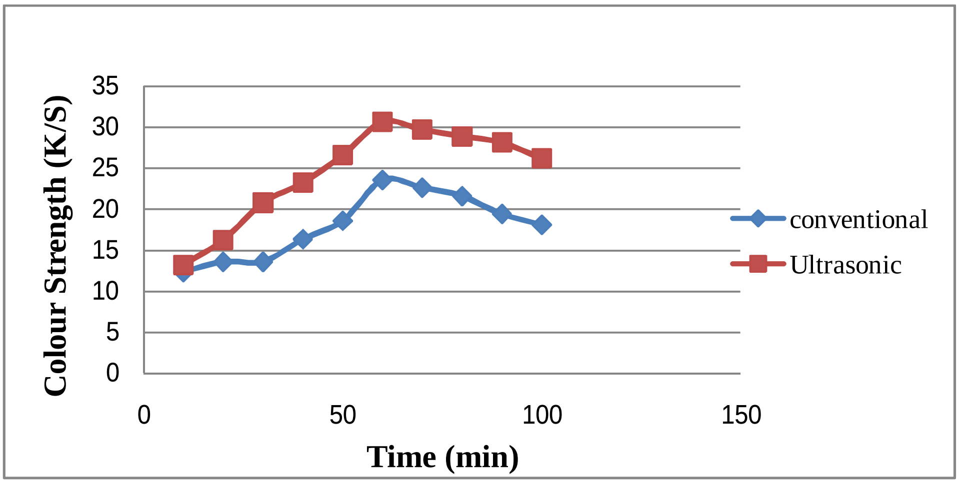


Figure 7: Effect of dyeing time on the colour strength of dyed wool fabrics.Dyeing conditions: 500 W, MLR 1:30, 30 ml aqueous dye extract (3% w/v), pH 3.5, at 60^{0}C.

Effect of temperature

The effect of temperature on the dye ability of the wool fabrics with natural dye was conducted under US and CH conditions at different temperatures (30-80 °C). As shown in Fig. 8, it is clear that the colour strength increases with increase in dyeing temperature in both cases of US and CH methods with pronounced increase in the US case than the CH. At 60 °C or above a plateau value of *K*/*S* was observed in CH, the case that was absent in US method. Generally, the increase in dye uptake can be explained by fiber swelling and hence, enhanced dye diffusion. Also, the ultrasonic power provides other additional factor of de-aggregation of dye molecules and thus leading to further enhancement of dye diffusion and better dye ability than CH.

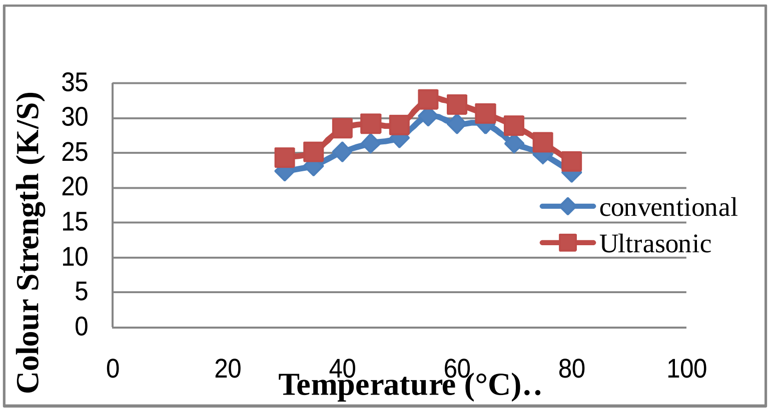


Figure 8: Effect of dyeing temperature on the colour strength of dyed wool fabrics. Dyeing conditions: 500 W, MLR 1:30, 30 ml aqueous dye extract (3% w/v), pH 3.5, at 60^{0}C.

Effect of ultrasonic power

Fig.9, shows the colour strength of dyed fabric to be directly proportional to the power supplied. Different levels of power from 100 to 600W were applied. This once again establishes the assisting effect of power ultrasonic on the dye ability of wool fabrics with natural dye. This may be due to breaking up of micelles and high molecular weight aggregates into uniform dispersions in the dye bath [14] degassing, i.e., expulsion of dissolved or entrapped gas or air molecules from fiber into liquid and removal by cavitation thus resulting in dye fiber contact and diffusion expediting the rate of dye diffusion inside the fiber by penetrating the insulating layer covering the fiber (if any) and speeding up the interaction or chemical reaction(if any) between dye and fiber.

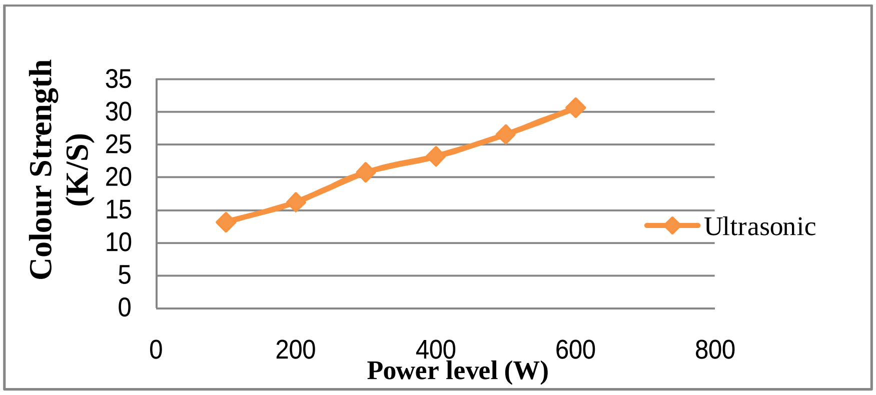
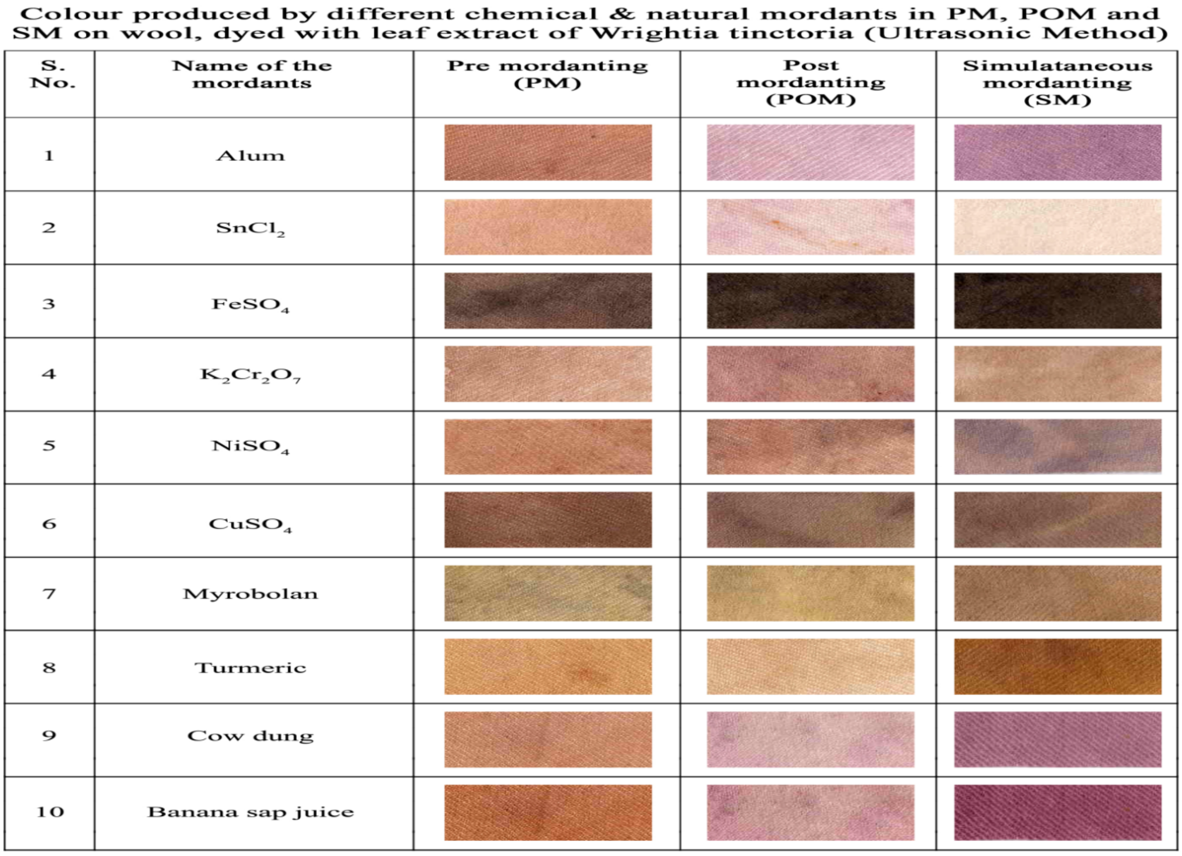


Figure 9: Effect of power level on the colour strength of dyed wool fabrics. Dyeing conditions: 500 W, MLR 1:30, 30 ml aqueous dye extract (3% w/v), pH 3.5, at 60^{0}C.



Colour produced by different chemical & natural mordants in PM, POM and SM on wool, dyed with leaf extract of Wrightia tinctoria (Ultrasonic Method)

S. No.	Name of the mordants	Pre mordanting (PM)	Post mordanting (POM)	Simulataneous mordanting (SM)
1	Alum			
2	$SnCl_2$			
3	$FeSO_4$			
4	$K_2Cr_2O_7$			
5	$NiSO_4$			
6	$CuSO_4$			
7	Myrobolan			
8	Turmeric			
9	Cow dung			
10	Banana sap juice			

Table 1: Effect of dyed with leaves extract of *Wrightia tinctoria.L* mordants on the colour strength of wool fabric

Dyeing Method	Ultra sonicator				
Colour strength	Process	L*	a*	b*	K/S Value **K/S(λ=420 nm)**
Without mordant	Pre	56.57	8.88	12.80	4.38
	Post	65.36	5.82	3.24	5.12
	SM	57.36	7.49	5.24	3.15
Alum	Pre	67.67	9.28	10.88	14.68
	Post	75.47	10.07	-0.59	16.38
	SM	69.23	8.13	2.52	13.25
$SnCl_2$	Pre	82.22	3.37	13.89	12.02
	Post	80.5	5.79	6.57	16.28
	SM	81.2	4.32	10.25	11.62
$FeSO_4$	Pre	45.03	5.03	4.71	**28.51**
	Post	35.8	4.25	4.71	**32.62**
	SM	38.3	3.52	3.41	**25.05**
$K_2Cr_2O_7$	Pre	64.74	7.41	13.53	16.38
	Post	63.11	9.8	9.58	19.1
	SM	61.2	8.15	12.69	13.62
$NiSO_4$	Pre	59.5	9.00	13.82	17.32
	Post	61.97	7.52	9.09	24.31
	SM	60.5	8.19	12.60	18.12
$CuSO_4$	Pre	49.0	7.38	13.15	**24.71**
	Post	54.65	7.49	5.10	**29.01**
	SM	52.3	7.24	8.31	**21.92**
Myrobolan	Pre	74.86	0.91	26.14	6.38
	Post	73.03	2.52	28.64	7.32
	SM	71.0	1.25	27.31	4.38
Turmeric	Pre	22.24	6.10	32.42	8.72
	Post	76.2	3.66	27.23	9.92
	SM	74.81	4.52	30.15	7.79
Cow dung	Pre	63.57	10.88	15.80	5.38
	Post	73.36	7.82	4.24	6.62
	SM	68.36	9.49	7.24	4.15
Banana sap juice	Pre	66.06	10.37	14.94	6.92
	Post	75.55	8.5	3.18	8.1
	SM	72.16	9.51	3.92	5.02

Table 2: Fastness Properties for Wool Fabric Dyed with *Wrightia tinctoria.L*

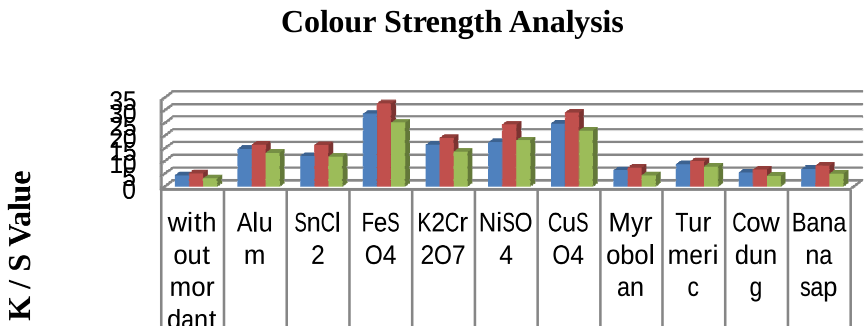
Dyeing Method	Ultra sonicator						
Fastness	Process	Washing fastness (IS-105-C03)	Light fastness (IS-2454-85)	Rubbing fastness (IS-766-88)		Perspiration (IS-105-E04)	
Mordant concentration: 3%				Dry	Wet	Acid	Alkali
Without mordant	Pre	4	4	4	4	4	4
	Post	4	4	4	4	4	4

	SM	4	4	4	4	4	4
Alum	Pre	5	5	5-6	5	5-6	5
	Post	5-6	5	5-6	5	5-6	5
	SM	5-6	5	5-6	5	5-6	5
$SnCl_2$	Pre	5	4-5	5	5	5	5
	Post	5	5	5	5	5	5
	SM	5	5-6	5	5	6	5
$FeSO_4$	Pre	6	5	6	5	6	5
	Post	5-6	5	6	5	6	5
	SM	6	5-6	6	5	5-6	5
$K_2Cr_2O_7$	Pre	5	5	5	5	5	5-6
	Post	5	5	5	5	6	5-6
	SM	5	5-6	5	5	6	5
$NiSO_4$	Pre	5	5	5	5	5-6	5
	Post	6	5	6	5	5-6	5
	SM	6	5	6	5	5-6	5
$CuSO_4$	Pre	6	5	6	5	5-6	5
	Post	6	5	6	5	6	5
	SM	6	5	6	5	6	5
Myrobolan	Pre	4-5	4	4-5	4	4-5	4-5
	Post	4-5	4	4-5	4	4-5	4
	SM	4-5	4	4-5	4	4	4
Turmeric	Pre	4-5	4-5	4	4	4-5	4
	Post	4-5	4	4	4	4-5	4
	SM	4-5	4	4	4	4	4
Cow dung	Pre	4	3-4	4	4	4	4
	Post	4	4	4	4	4	4
	SM	4	4	4	4	4	4
Banana sap juice	Pre	4-5	4-5	4-5	3-4	4	4-5
	Post	4	4-5	4-5	3-4	4-5	4
	SM	4-5	4-5	4-5	4	4-5	4

Optimization of mordant with K / S values and colour dye changes

Various shades of colour were obtained from pre mordanted, simultaneous mordanted and post – mordanted methods of dyeing different mordants determined the various shades of colour on the dyed fabric according to K/S values. The different L*, a*, b* and K/S values show in table 1. The L* values indicates perceived lightness or darkness where values of 0 indicates black and 100 indicates white. The values of a* and b* indicate red (+a) and green (-a) while b* values indicate yellow (+a) and blue (_b). In pre-mordenting method K/S values $FeSO_4$ is 28.51, $CuSO_4$ is 24.71, and the shades of colour in found to be darker. But when L* values are lower, the hues of colour obtained will be darker. In post-mordanting method K/S values for $FeSO_4$ is 32.62, $CuSO_4$, in 29.01

and the shades of colour are found to be darker while the lower L* values show lighter shades. In simultaneous mordanting method, $FeSO_4$ is 25.05, $CuSO_4$ in 21.92 which shows the shades of colour are darker. But if the L* values are lower, the shades of colour will be darker. The effect of mordtants on colour values of on wool dyed with leaves of *Wrightia tinctoria* is shown in Figure 5.



	without mordant	Alum	SnCl2	FeSO4	K2Cr2O7	NiSO4	CuSO4	Myrobolan	Turmeric	Cow dung	Banana sap
Premordaning	4.38	14.68	12.02	28.51	16.38	17.32	24.71	6.38	8.72	5.38	6.92
Post mordanig	5.12	16.38	16.28	32.62	19.1	24.31	29.01	7.32	9.92	6.62	8.1
Simultaneous mordaning	3.15	13.25	11.62	25.05	13.62	18.12	21.92	4.38	7.79	4.15	5.02

Figure 11: Surface Colour Strength (K/S Values) of Dyes Wool Fabrics after Pre, Post and Simultaneous Mordanting Methods

Colour fastness Properties

The fastness properties of dyed wool fabrics are shown in Table 2. It was observed that, dyeing with *Wrightia tinctoria.L* gave very good washing, light and rubbing, perspiration fastness properties. Overall, it could be used for commercial purposes and attain acceptable range.

Ultrasonic efficiency

Ultrasonic efficiency (ΔK %) in accelerating the dyeing rate was examined by introducing the following equation:

$(\Delta K\ \%) = 100\ (K_{us}-K_{ch})/K_{ch}$

Where K_{us} and K_{ch} are the rate constants of dyeing with ultrasonic and conventional heating, respectively. The value of ultrasonic efficiency is positive for dyes indicating a favorable Effect of ultrasonic power on the dyeing process.

CONCLUSIONS

The dye uptake as well as the fastness properties of the wool fabric was found to enhance when metal mordant was used in conjugation with ultra sonication for the extract of *Wrightia tinctoria***.** Thus ultrasound has been shown to be an effective tool in textile dyeing for cleaner production. In this respect, for the further studies it will be interesting to investigate addition of

functionality to the cotton slivers during the dyeing with different herbal sources which have different medical
Properties.

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