International Journal of Chemical Studies

P-ISSN: 2349–8528
E-ISSN: 2321–4902
IJCS 2018; 6(6): 1131-1136

Received: 04-09-2018
Accepted: 08-10-2018

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Management of bitter bush weed (*Chromolaena odorata*) by natural dyeing of silk using metallic mordants

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Abstract
Bitter bush is an alien, noxious and aggressive weed, which has destructing effects on the agricultural crops, forests, and other exposed areas. This weed can be used as a profitable source of eco-friendly natural dye, as potential use of it as dye source can also solve the problem of its invasion which is otherwise very difficult. The process of silk dyeing with twigs of Bitter Bush was optimized to produce varying shades of dye on silk fabric by using selected metallic mordants. Colour fastness of dyed and mordanted samples to light, washing, crocking and perspiration was also evaluated. Different variables viz. concentration of dye material, extraction time, dyeing time, mordants concentration and method of mordanting were optimized based on percentage of absorption of dye by the silk fabric as well as percentage of marks obtained by the dyed sample through visual evaluation. Colour fastness of samples was found satisfactory, so bitter bush dye can be profitably used for dyeing of silk fabric.

Keywords: natural dye, weed, silk dying, metallic mordants, colourfastness

Introduction
As the whole world slowly awaken to the ill-effects of various chemical products, textile dyeing sector also desperately required new natural dye sources which are renewable and at the same time commercially viable. The problem of renewable source of dye can only be solved by identifying new sources of natural dye which are present in abundance and their rate of renovation is fast. Bitter Bush (*Chromolaena odorata*) or *Kalabasa* (local name) is an alien, noxious and aggressive weed, which mainly occurs in agricultural areas, forest waste lands, road sides and other exposed areas in various parts of India. Invasion of this fast growing weed is presently a serious problem for the farmers, forest and archeological officials, as it destroys the crops, forests and monuments. Thus natural dye extracted from twigs of Bitter bush can be profitably used to dye silk fabric and farmer-friendly also. In the present investigation attempts has been made to optimize the process of dyeing of silk fabric with natural dye extracted from twigs of Bitter Bush (*Chromolaena odorata*), using different metallic mordants.

Experimental details
Materials
Bitter bush twigs were collected from hillsides of College of Forestry and Hill Agriculture, Ranichauri, Hill-campus: G.B. Pant University of Agriculture and Technology, Pantnagar, District Tehri Garhwal (Uttarakhand) and dried under shade and made into coarse powder. Metallic mordants used for the study were alum, copper sulphate, ferrous ammonium sulphate, tartaric acid and chrome. Laboratory grade chemicals were procured from Central Drug House (P) Ltd, New Delhi. The silk fabric used in this study was purchased from Gandhi Ashram, Pantnagar. The λ-max and optical density of dye solutions was measured by using PC based double beam UV/VIS spectrophotometer (ECIL). Chemicals used for changing the medium were Hydrochloric acid and Sodium carbonate. Analytical Rasayan grade (AR) chemicals were obtained from S.D Fine- Chem. Limited.

Methods
Dyeing variables viz. dye concentration, extraction time, dyeing time) were optimized on the basis of percent absorption and percent of marks obtained through visual evaluation, whereas selection of dye extraction medium,

concentration of mordants and methods of mordanting were optimized on the basis of maximum marks obtained in visual evaluation by panel of judges only. Percent absorption of the dye was calculated by recording optical density at given λ-max before and after dyeing. To calculate the percentage absorption, the absorbance of the dye solution at λ-max was recorded both before and after dyeing. The optical density was recorded and percent absorption was calculated by the following formula:

$$\%\,\text{Absorption} = \frac{\text{O.D. of the dye liquor before dyeing} - \text{O.D. of the dye liquor after dyeing}}{\text{O.D. of the dye liquor before dyeing}} \times 100$$

Percentage of marks obtained through visual evaluation was calculated by using following formula:

$$\%\ \text{of marks} = \frac{\text{Marks obtained}}{\text{Total marks}} \times 100$$

Preparation of Fabric: The silk samples were dipped in solution of 0.5 ml of mild detergent (genteel) per 100 ml of water heated at 50˚C, for 30 minutes followed by kneading, squeezing and rinsing with running water. Samples were then dried in shade and ironed while still damp. Prior to dyeing or mordanting the silk samples were soaked for half-an hour.

Dye extraction medium: The dye source was extracted in aqueous, acidic and alkaline medium. In neutral aqueous medium 100 ml distilled water was used for extraction of dye. Acidic medium was prepared by adding 1 ml of hydrochloric acid in 100 ml of distilled water. Alkaline medium was prepared by dissolving 1 g of sodium carbonate in 100 ml of distilled water. Dye solutions were prepared by extracting 2 g of each dyestuff in 100 ml of each aqueous, acidic and alkaline medium at 80˚C for 60 minutes. After that, the solutions were filtered and pre soaked silk samples of 1 g were dyed in these solutions for 60 minutes. The samples were then allowed to cool at room temperature, taken out, rinsed in water, dried in shade and ironed while still half wet.

Selection of mordants: Five metallic mordants (alum, copper sulphate, ferrous ammonium sulphate, tartaric acid and chrome) were taken at constant concentration of 0.01 g/1 g silk fabric/100 ml of distilled water. Mordant solution was prepared by dissolving mordant in 10 ml of pre prepared dye extract, strained and then added to remaining 90 ml of dye liquor. Simultaneous mordanting and dyeing was carried out with optimized dyeing time at 80ºC. After dyeing, rinsing, washing and ironing, the samples were evaluated visually and three mordants which gave best results with the dye were selected.

Dye concentration: To optimize concentration of dye material, five different concentrations of dye material i.e. 4 – 8 %, were prepared separately by boiling 4g, 5g, 6g, 7g and 8g of crushed twigs of bitter bush, in 100 ml of aqueous medium for 60 minutes at 80ºC. To see whether optical density was further increased with increase in concentration, one extra dye solution of 9g dye material / 100ml distilled water was also prepared. After cooling and filtration, 1 ml of dye solution was pipetted out from each beaker and absorbance was recorded after diluting it up to 20 times. Presoaked silk samples of 1 gm were dyed in these solutions for 60 minutes at 80ºC with continuous stirring using material and liquor ratio1:100. The dyed samples were removed from the dye bath, rinsed in tap water, dried in shade, and ironed when half wet. Again optical density for all the residual solutions was recorded using the same procedure as mentioned earlier. Best samples were selected based on maximum percentage absorption and highest percentage of marks obtained through visual evaluation for further study.

Extraction time: The optimum concentration of the dye material was taken in 5 beakers each containing 100 ml of selected extraction medium and boiled for 30, 45, 60, 75 and 90 minutes at 80ºC respectively. The dye extracts thus obtained were cooled and filtered. Absorbance of dye solutions was recorded and presoaked silk samples of 1 gm each were dyed in these solutions for 60 minutes at 80ºC. Dyed samples were removed from the dye bath and rinsed in tap water, dried in shade and ironed, when half wet. Optical density of residual solution was recorded. Samples were also evaluated visually and were selected on the basis of maximum percentage of absorption and highest percentage of marks obtained.

Dyeing time: Five solutions of dye were prepared with optimized concentration and extraction time. Optical density was recorded. Pre soaked samples of 1 gram of silk were dyed in these solutions for 30, 45, 60, 75, 90 minutes, respectively at 80ºC. Dyed samples were removed from the dye bath solution, rinsed in tap water, dried in shade and ironed. Optical densities of the residual solutions were recorded. Percent absorptions were calculated and samples were evaluated visually.

Preparation of blank sample: Silk sample weighing 2 g + 0.01 g was dyed with the twigs of biter bush using the optimized dyeing conditions, i.e. concentration of dye, dye extraction time and dyeing time.

Concentration of mordants: Each selected mordants was taken in five different concentrations, 0.01g, 0.02g, 0.03g, 0.04g and 0.05 g/ 100 ml of the dye extract/g of silk. Simultaneous mordanting and dyeing method was used for the determination of optimum concentration of mordant. Different concentration of each metallic mordant was dissolved in 10ml of extracted dye liquor. These solutions were mixed with the remaining 90ml dye liquor warmed and stirred. Pre-soaked silk samples were placed in the dye bath and the temperature was raised to boiling and then allowed to simmer at 80ºC till the optimum dyeing time. The samples were removed from the dye bath and rinsed under tap water. These were dried in shade and ironed when half wet. Best sample was selected on the basis of visual evaluation.

Optimization of methods of mordanting: Mordanting was carried out by three different methods i.e. pre mordanting, simultaneous mordanting and dyeing and post mordanting. The methods, which produced best colour, were selected on the basis of highest percentage of marks obtained by sample through visual evaluation.

Preparation of final samples: Final samples weighing 2.0 + 0.1g were prepared from the selected plant dye source with optimized dyeing conditions and optimum concentration of mordant and method of mordanting. The developed colours were named according to the shade card of Asian paints, Berger paints and Sandtex Matt paints.

Assessment of colourfastness: Standard methods i.e. light fastness test (IS: 686-1957), washing fastness test (IS: 3361-1979), fastness to Rubbing (IS: 766-1956), perspiration fastness (IS : 971-1956) were used to assess the colourfastness of final samples.

Statistical analysis: Weighted mean score was calculated to find out the mordant and mordanting method which is most suitable for dyeing of silk with Bitter bush dye. Marks were assigned to each factor i.e. light, washing, crocking and perspiration as per their importance for consumers. Using following formula, weighted mean score was calculated and different ranks were allotted to mordants along with their method of mordanting:

In case, k variate X1, X2 … Xk have known weights w1, w2 … wk, respectively, then the weighted mean is:

$$\mu = \frac{w_1X_1 + w_2X_2 + \ldots\ldots + w_kX_k}{w_1 + w_2 + \ldots\ldots w_k}$$

Observations and Results

Bitter bush dye extracted in aqueous medium was found to produce best results on silk fabric and was selected for further study. Range of shades was produced on silk by five metallic mordants i.e. alum, copper sulphate, ferrous ammonium sulphate, tartaric acid and chrome with Bitter bush dye. On the basis of visual evaluation alum, ferrous ammonium sulphate and tartaric acid were found to produce best results with Bitter bush dye. The λ-max of dye solution of Bitter bush dye was found to be 572 nm.

Maximum percent absorption and highest percentage of marks in visual evaluation was obtained with 6 % concentration of dye material. The results of percent absorption and marks obtained by samples on visual evaluation are given in Table 1. It shows that in Bitter bush dye, the percentage absorption increased from 4 to 6 grams of dye concentration. Further, a sharp decline was found with increase in concentrations. With the higher concentration i.e. 9 grams of dye, percent absorption was minimum. Therefore, 6 gram concentration was selected optimum for further study as illustrated in Fig.1.

The maximum percent absorption was obtained when dye was extracted for 60 minutes. On visual evaluation, the same result was found that the maximum marks were obtained by the sample dyed with solution when dye was extracted for 60 minutes. Therefore 60 minutes dye extraction time was selected for further study as illustrated in Fig. 2.

Percentage absorption of Bitter bush dye by silk fabric increased with increase in dyeing time from 30 to 45 minutes. Thereafter decrease in percent absorption was observed with increase in dyeing time. The maximum percent absorption was found for sample dyed for 45 minutes. But on visual evaluation, the sample dyed for 60 minutes obtained the highest percentage of marks (Fig. 3).

The results of visual evaluation reveals that samples mordanted with 0.01 per cent concentration for tartaric acid and 0.04 per cent concentration for ferrous sulphate and alum obtained maximum scores. So, these concentrations were selected for further study. Difference in percentage of marks for different methods of mordanting with selected metallic mordants for Bitter bush dye is shown in Fig. 4. Colours developed through different methods of mordanting with selected metallic mordants using Bitter bush dye are shown in Sample Sheet. Fig.4. reveals that simultaneous dyeing and mordanting method gave best result with tartaric acid while post mordanting method was found best with ferrous sulphate and alum. Selection was done on the basis of visual evaluation. The blank sample dyed with Bitter bush dye showed Tata mimosa colour, whereas Olive green, Daffodil and pale cream colours were obtained with ferrous sulphate, tartaric acid and alum, respectively.

Results of colourfastness test reveals that blank sample dyed with Bitter bush dye showed negligible (5) change in colour as well as negligible staining (5) on cotton during both dry and wet crocking. The mordanted samples as well as blank sample were fast towards dry and wet crocking, but dry crocking was found better than wet crocking. Wash fastness test results reveals that in case of blank sample change in colour was found from slight to negligible (4-5), while negligible (5) staining on both cotton and silk was found. Increase in colour was observed in alum mordanted sample. In case of both acidic and alkaline perspiration fastness test blank sample showed slight to negligible (4-5) change in colour. Increase in colour was also observed in both the cases. Whereas, negligible (5) staining on both silk and cotton was found in acidic and alkaline perspiration. The results showed that in most of the cases, the fastness to perspiration deteriorated with the use of mordants. Blank sample showed poor (2) fastness to light. Sample mordanted with tartaric acid showed fair to fairly good (3-4) fastness to light whereas fairly good (4) fastness to light was found in case of ferrous sulphate and alum mordanted samples. It can be said that metallic mordants improved the light fastness of dyed samples when compared with blank sample.

On the basis of weighted mean score, different mordanted samples were given rank for their fastness. The sample that had highest weighted mean score was given rank I and so on. Detailed results are shown in Table 2. Overall ranking of metallic mordants for colour fastness indicated that ferrous sulphate was found best as it occupied first rank. Alum obtained second while tartaric acid mordant got lowest rank in overall ranking of mordants for colour fastness.

Discussion

The difference in results has been found in the optimization of dyeing time step, i.e. between maximum percent absorption (45 min) and highest percentage of marks (60 min) obtained through visual evaluation. It is the fact that consumers are the ultimate users, therefore 60 minutes dyeing time was selected as illustrated in Fig. 3. Optimum dyeing time was selected on the basis of visual evaluation that was carried out on different criteria including luster, evenness of dye, depth of shade and overall appearance of the colour which are considered important by consumers while selecting coloured textile materials. Since the percentage of marks obtained through visual evaluation is a subjective approach, the appearance of colour may or may not be related to maximum percentage of absorption. The increase in colour was observed during various colour fastness tests. The reason behind darkening may be presence of alkali, acid and temperature applied during tests, which can affect structure of dye molecule. Singh (1991) [1] also reported that the hue of silk sample dyed with Nargis leaf got darkened on washing.

Table 1: Percent absorption by silk fabric dyed with different concentration of Bitter bush dye material at 572 nm λ_{max}

Conc. of dye material (g)	O.D. before dyeing	O.D. after dyeing	% Absorption	% of marks obtained
4	0.041	0.035	14.63	61.25
5	0.045	0.031	31.11	65.00
6*	0.065	0.040	38.46	78.13
7	0.074	0.059	20.27	78.00
8	0.079	0.063	20.25	76.87
9	0.080	0.061	13.75	-

Selected concentration of dye

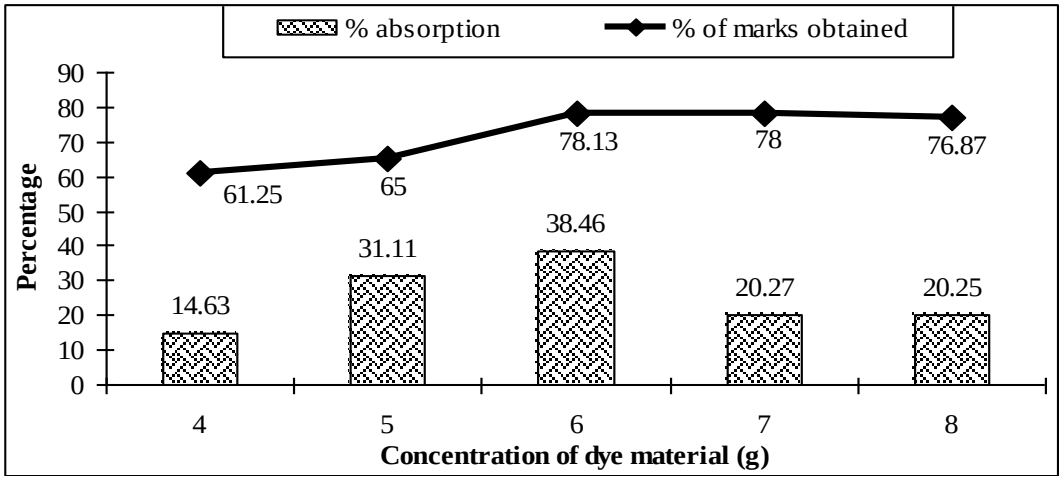


Fig 1: Percentage of absorption and percentage of marks of silk fabric dyed with Bitter bush dye with different concentrations

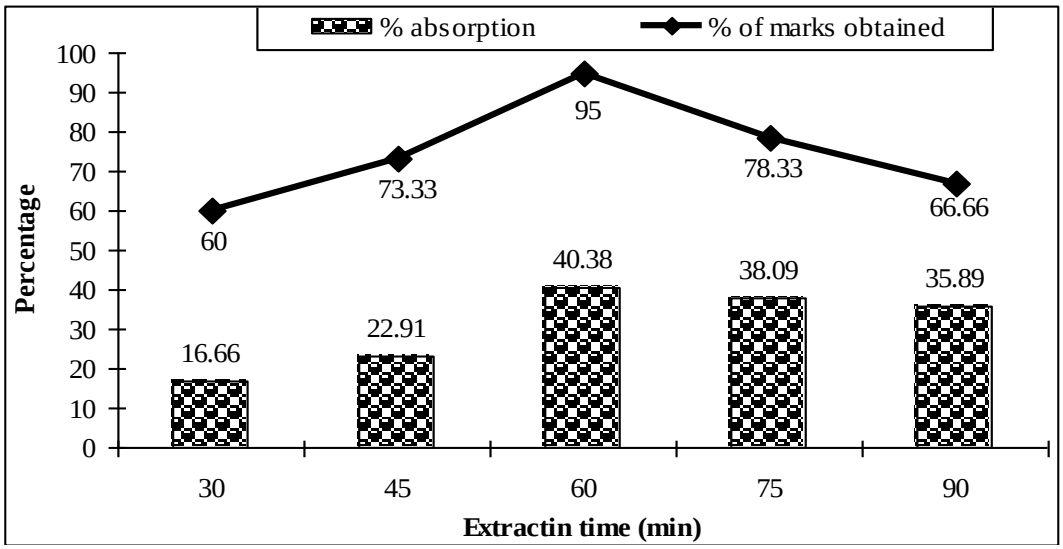


Fig 2: Percentage of absorption and percentage of marks of silk fabric dyed with Bitter bush dye with different extraction time

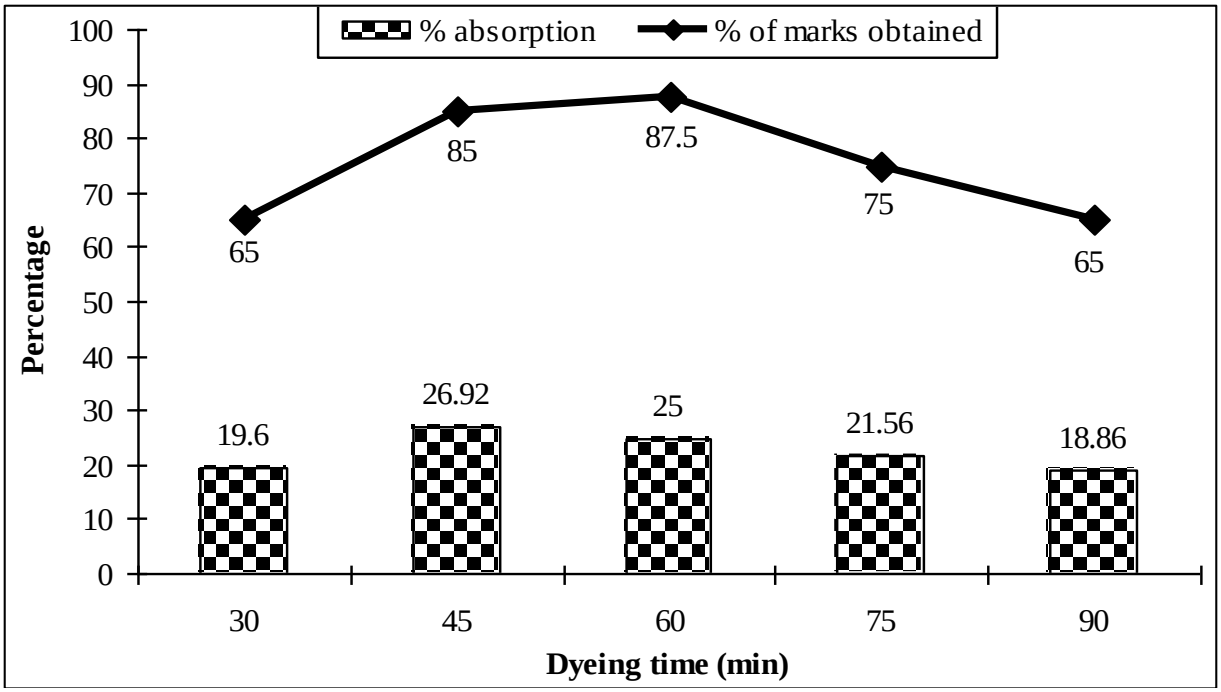


Fig 3: Percentage of absorption and percentage of marks of silk fabric dyed with Bitter bush dye with various dyeing time

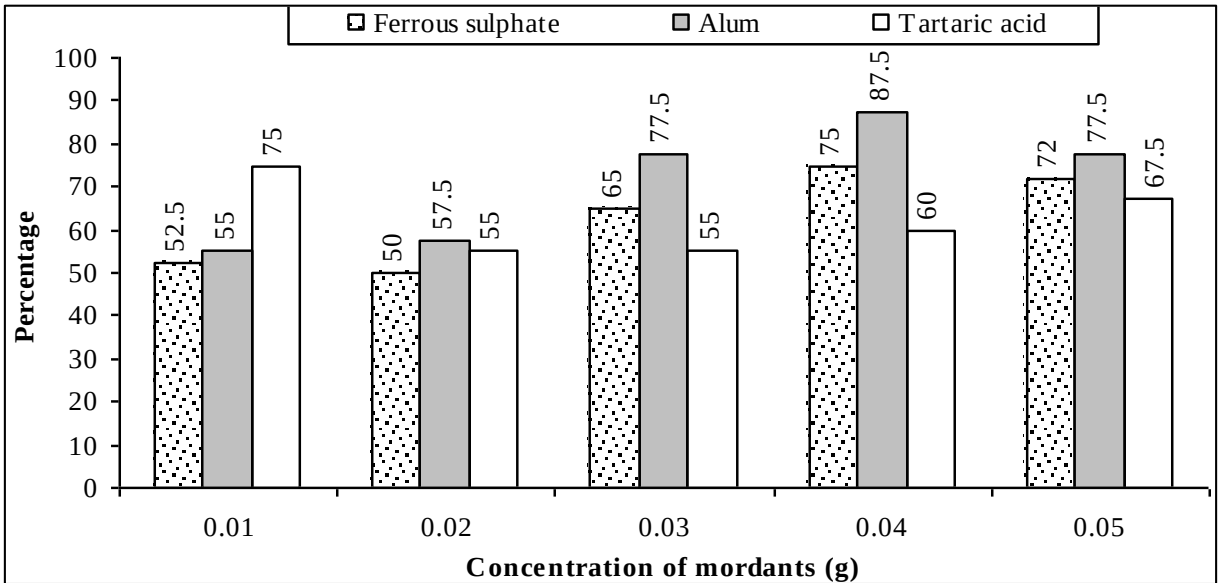


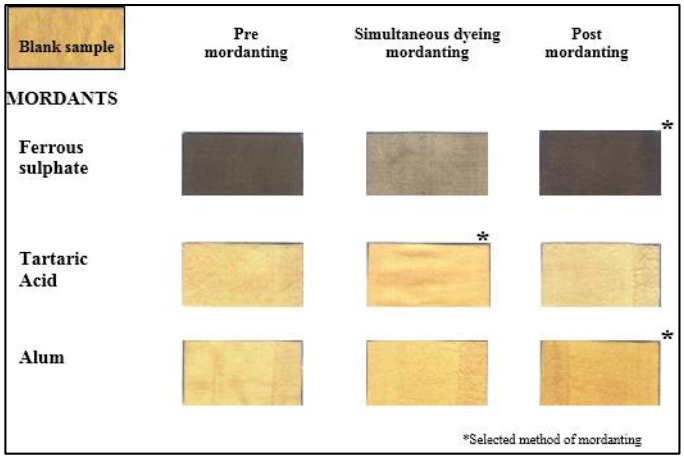
Fig 4: Percentage of marks obtained lby different concentrations of metallic mordants for Bitter bush dye

Table 2: Overall ranking of mordants against colour fastness

S No.	Mordants	Mordanting method	CF				WF			PF						LF	WMS	Rank
			Dry		Wet		CC	CSC	CSS	Acidic			Alkaline					
			CC	CSC	CC	CSC				CC	CSC	CSS	CC	CSC	CSS			
1.	Ferrous sulphate	III	5	5	4-5	3-4	5	5	5	4-5*	5	5	3-4	5	5	4	24.50	I
2.	Tartaric acid	II	5	5	5	4	5	4-5	4-5	3-4*	4-5	4	3-4	4-5	4-5	3-4	17.25	III
3.	Alum	III	5	5	5	4-5	4-5*	5	5	4-5*	5	4-5	3-4*	5	5	4	18.05	II
	BLANK	-	5	5	5	5	4-5	5	5	4-5*	5	5	4-5*	5	5	2	-	-

CF: Crocking fastness
CSC: Colour staining on cotton
PF: Perspiration fastness
LF: Light fastness
CC: Change in colour
WF: Washing fastness
CSS: Colour staining on silk
*: Increase in colour
I: Pre Mordanting
II: Simultaneous mordanting
III: Post mordanting
WMS: Weighted mean score
5: negligible or no change/ staining
4-5: slight to negligible change/ staining
4: Slight change/ staining
3- 4: Noticeable to slight change / staining
2-3: considerable to noticeable change/ staining

Sample Sheet: Final samples dyed with Bitter bush dye using different methods of mordanting



Conclusion

At the end it can be concluded that Bitter bush is profitable source of fast renewable dye which is locally available and found in abundance to the people of Uttarakhand and other parts of the country. The optimized process of dyeing is easy and suitable for adoption at cottage and household level. The colourfastness of the dye is satisfactory so it can be used in dyeing of fabrics for apparels, upholsteries and other household articles.

Acknowledgement

The authors are thankful to teaching and non-teaching staff of department of Clothing & Textiles, department of Plant Pathology and College of Forestry and Hill Agriculture, Ranichauri, Hill-campus: G.B. Pant University of Agriculture and Technology, Pantnagar, District Tehri Garhwal (Uttarakhand).

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