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Profitable management of noxious weed *Chromolaena odorata* through extraction of natural dye for silk fabric

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Abstract
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 Uttarakhand. 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. But, this weed can be successfully used as a quickly renewable and profitable source of natural dye which can be contributed to the additional income of the farm families as well as will lead to the effective management of this deleterious weed. The process of silk dyeing with twigs of Bitter Bush was optimized to produce varying colour shades on silk fabric by using selected natural mordants. Colour fastness of dyed and mordanted samples to light, washing, crocking and perspiration was also evaluated in present study.

Keywords: *Chromolaena odorata,* weed management, natural dyes, doubling the farmers' income

Introduction
As the world is slowly awaken to the damaging effects of some of the chemicals that were synthesized by humans including some carcinogenic chemical dyes. Pollution control, environment conservation, consumer health and safety have come among the emerging issues to be handled on priority. Now-a-days consumer world wide look increasingly for eco-friendly textiles, which are finished with replenish able products obtained from nature. Hence, there is huge demand for natural dyes which are obtained from quickly renewable sources. The Bitter bush weed plant locally known as *kalabasa* or *banmara*, is easily available and found in abundance, mainly in hilly regions of Uttarakhand and southern part of India. It commences a serious threat to the cultivation as its invasion is very difficult from agricultural fields. 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 natural mordants.

Experimental details
Materials and Methods
Bitter bush twigs were collected from hillsides of District- Tehri Garhwal (Uttarakhand) and dried under shade and made into coarse powder. Natural mordants, *harad* (*Myrobolan*), *bahera* (*Terminalia bellirica*), *amla* (*Phyllanthus emblica*), tea leaves (*Camellia sinensis*) and walnut bark (*Juglans regia*) were procured from local market. All the natural mordants except tea leaves were broken into small pieces and ground into fine powder form. The silk fabric used in this study was purchased from Gandhi Ashram. 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.
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 50^0C, 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 80ºC 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 natural mordants (*Harad*, *bahera*, tea leaves, *amla* and walnut bark) were taken at constant concentration of 1 g. These were soaked separately in 50 ml of water and kept overnight, boiled for fifteen minutes and filtered. The dye liquors were prepared with optimized conditions. These liquors were mixed with mordant extract and dyeing of pre soaked samples (1g each) was carried out separately on 80ºC at optimized dyeing time using material and liquor ratio 1:100. Simultaneous mordanting and dyeing technique was used to select the mordant. After drying and ironing, visual evaluation of samples was done to select the three mordants giving best results with the dye.

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. Pre soaked 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 pre soaked 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 $\pm$ 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 mordant was taken in five beakers in concentration of 1g, 2g, 3g, 4g and 5g /50 ml of water, soaked overnight and filtered after heating for fifteen minutes. The dye liquors were prepared with optimized conditions. These liquors were mixed with mordant extract and simultaneous mordanting and dyeing was done to dye 1 gram of pre soaked silk carried out separately on 80ºC at optimized dyeing time. M: L ratio was kept 1:100. After dyeing samples were rinsed in tap water, dried in shade and ironed when half wet. Visual evaluation was done to select best concentration of each selected mordants.

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 $\pm$ 0.1g were prepared from the selected plant dye source with optimized dyeing conditions and optimum concentration of mordant and method of mordanting.

Assessment of colour fastness: 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 colour fastness 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 X_1, X_2 … X_k have known weights w_1, w_2 … w_k, respectively, then the weighted mean is:

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

Results obtained

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 natural mordants *i.e. harad*, *bahera*, tea leaves, *amla*, and walnut bark with Bitter bush dye. On the basis of visual evaluation tea leaves, *amla*, walnut bark 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 3 per cent concentration of tea leaves and 4 per cent concentration of *amla* and walnut bark were found to produce best results with Bitter bush dye on silk samples. Difference in percentage of marks for different methods of mordanting with natural mordants for Bitter bush dye is shown in Fig. 4. Colours developed through different methods of mordanting with selected natural mordants using Bitter bush dye are shown in Sample Sheet. Fig. 4. Reveals that pre-mordanting method was found best with natural mordant *amla*, whereas post mordanting method produced best results with tea leaves and walnut bark.

The blank sample dyed with Bitter bush dye showed Tata mimosa colour, whereas natural mordants *amla*, tea leaves, walnut bark gave Mustard, Golden brown, and Yellow oxide colour respectively.

Results of colour fastness 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 all mordanted samples. 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. Samples mordanted with *amla*, walnut bark and tea leaves showed poor to fair (2-3), fair to fairly good (3-4), fairly good (4) fastness to light respectively. It can be said that natural 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 natural and metallic mordants for colour fastness indicated that *amla* was found best as it occupied first rank. Tea leaves obtained second while walnut bark mordant got lowest rank in overall ranking of mordants for colour fastness.

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

Table 2: Overall ranking of final mordanted samples against different types of color 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.	Tea leaves	III	5	4-5	5	3-4	4-5	5	4-5	4-5*	4-5	4-5	3-4*	5	4-5	4	17.10	II
2.	Walnut bark	III	5	5	4-5	3-4	4-5*	4-5	4	5	4	4	2-3*	3	2-3	3-4	15.65	III
3.	Amla	I	5	5	5	4-5	4-5*	4-5	4-5	5	5	3	4*	4-5	4	2-3	17.30	I
	Blank	-	5	5	5	5	4-5	5	5	4-5*	5	5	4-5*	5	5	2	-	-

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

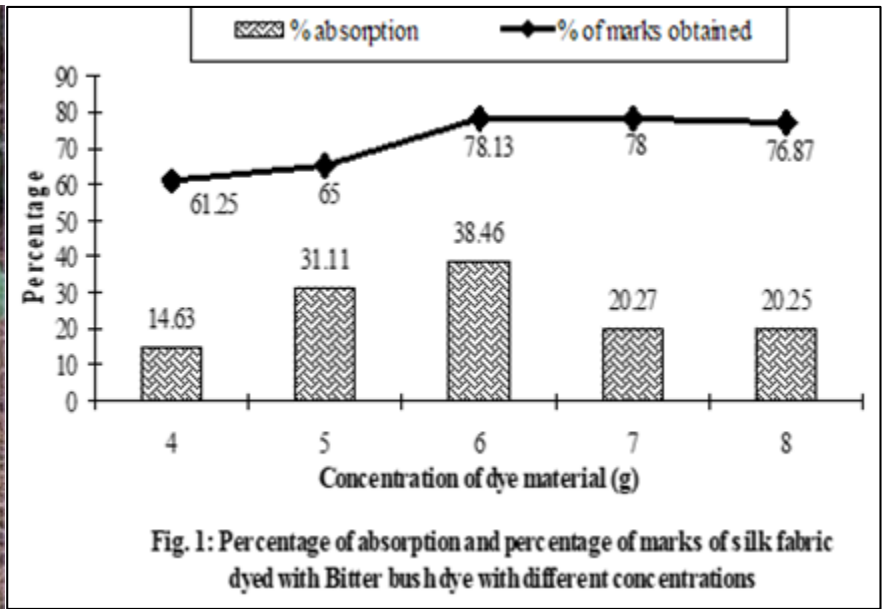
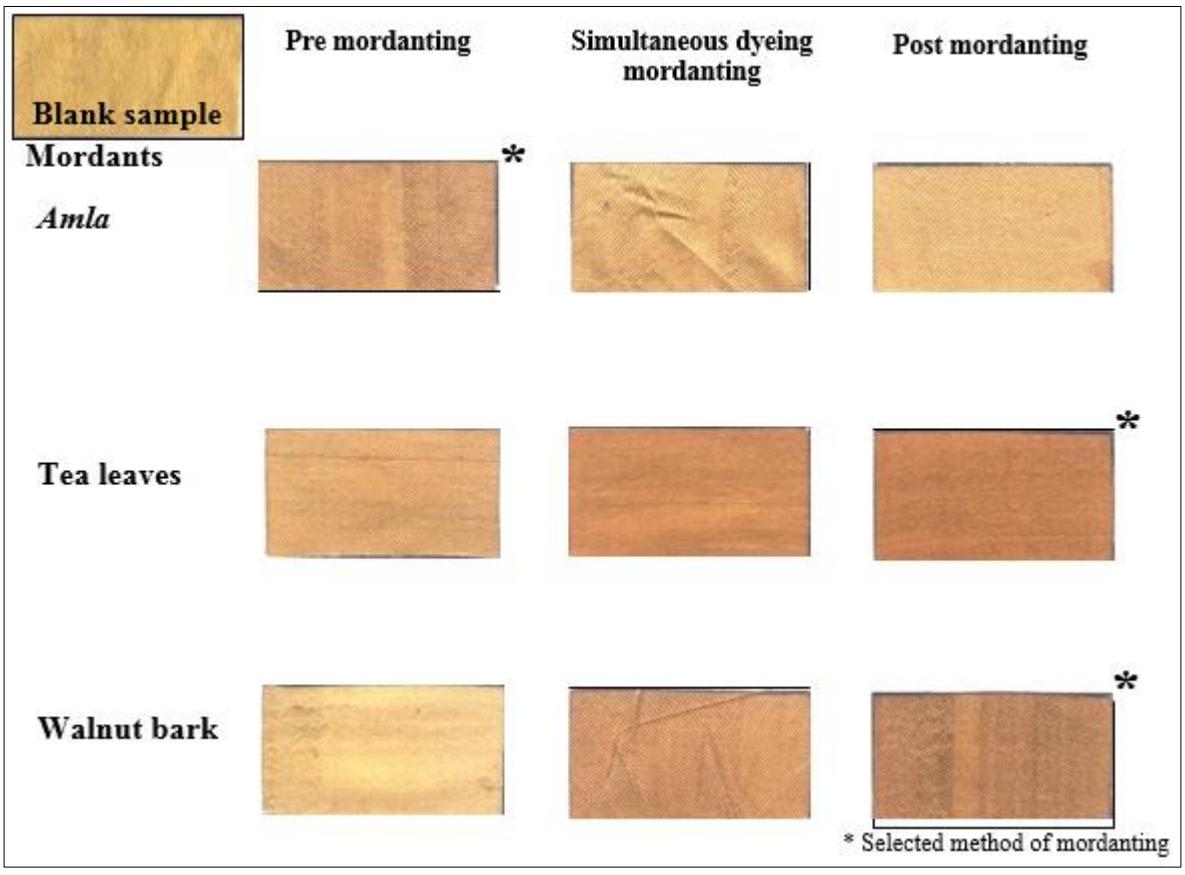


Fig. 1: Percentage of absorption and percentage of marks of silk fabric dyed with Bitter bush dye with different concentrations

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



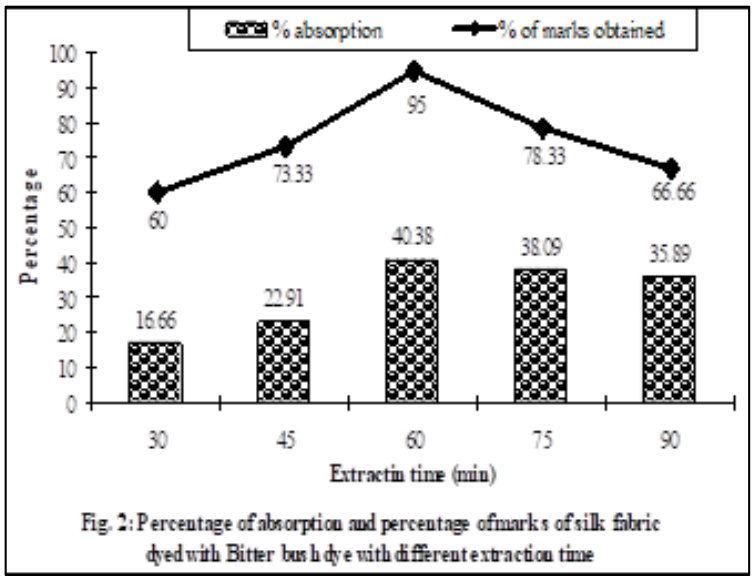


Fig. 2: Percentage of absorption and percentage of marks of silk fabric dyed with Bitter bush dye with different extraction time

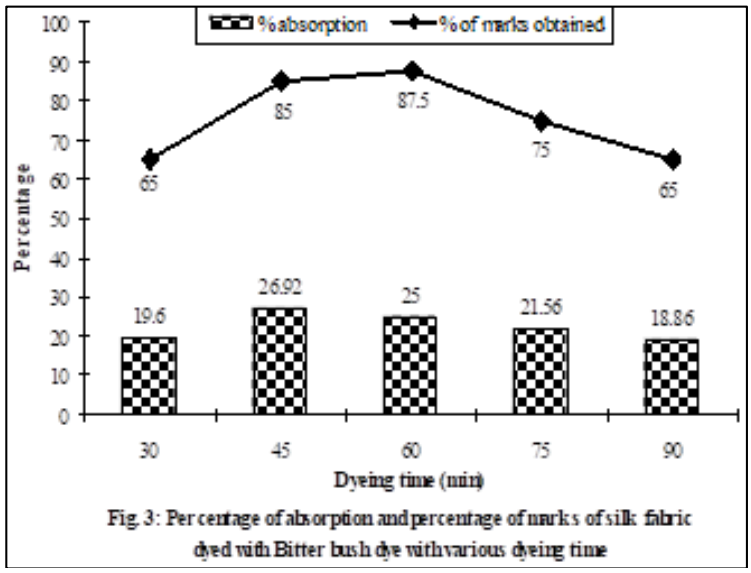


Fig. 3: Percentage of absorption and percentage of marks of silk fabric dyed with Bitter bush dye with various dyeing time

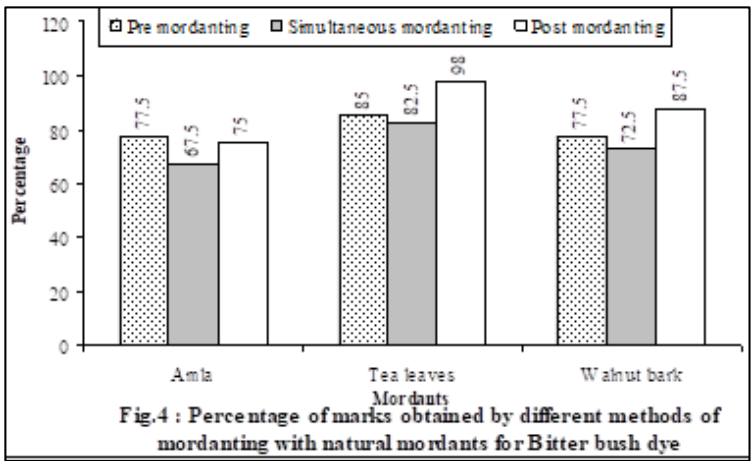


Fig.4 : Percentage of marks obtained by different methods of mordanting with natural mordants for Bitter bush dye

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) [2] also reported that the hue of silk sample dyed with Nargis leaf got darkened on washing. Natural mordants

obtained lower ratings for colour fastness to washing. The reason may be presence of tannins in natural mordants which are large molecules and retained only on the surface of the fabric and easily washed off as reported by Gulrajani (1999) [1].

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. The optimized process of dyeing is easy and suitable for adoption at cottage and household level. The colour fastness of the dye is satisfactory so it can be used in dyeing of fabrics for apparels, upholsteries and other household articles.

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