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DYEING OF WOOLEN FIBERS WITH *RHODODENDRON ARBOREUM* FLOWERS USING *SYMPLOCOS RACEMOSA* LEAVES AS MORDANT UNDER OPTIMUM CONDITIONS

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Abstract: Out of the multiple applications of the diverse natural products of Uttarakhand dyeing of fabrics with the help of various plant parts has always stood out. In the present investigation, an attempt has been made to extract natural dye from *Rhododendron arboretum* flowers and to optimize conditions of concentration and time for dyeing woolen sample from the extracted dye. The results showed that the colour strength (% Absorbance) increases with the increase in concentration of dye up to 0.03 g/ml concentration. Also, dyeing procedure has been optimized by using *Symplocos racemosa* leaves as mordant. The best results were obtained using simultaneous mordanting method with *Symplocos racemosa* leaves at 0.03 g/ml concentration.

Keywords: Natural dye, *Rhododendron arboreum*, Absorbance, *Symplocos racemosa*, Natural mordant.

Introduction

The co-existence of humanity and colours dates back to the advent of human civilization itself. Apart from this , colour was used on the walls of caves, baskets and many other items of day-to-day use (Gulrajani, 2001). Across the globe some specific plants and animals have been used as sources of specific colours like red, yellow, blue and black (Gulrajani, 1999; Gulrajani, *et al*., 2003; Mukherji, 1999). Various researchers have reported important natural dyes holding promise for the future (Naz and Bhatti, 2011; Samantha and Agarwal, 2007; Gulrajani and Deepti 2011; Singh and Kalirajan 2012). Natural dyes and mordants made from flora and fauna are considered to be safe due to their non-toxic, non-carcinogenic and biodegradable nature (Geetha and Sumathy 2013; Han and Yang 2005; Khan *et al*., 2012). The hill state of Uttarakhand also shows ample references of dyeing with indigenous plant species which are mainly confined to the native Bhotiya and Shauka tribes of high altitudes of the border districts of *Pithoragarh, Chamoli* and Uttarkashi. It was Atkinson in 1882 who for the first time gave a systematic list of the dye-yielding plants of Uttarakhand (Atkinson, 1882; Negi, 2006). The old manuscripts got their name 'Pandulipi'from yellow colour (Pandu = yellow) itself. Scholars like Gaur, (2008) and Sharma *et al*., (2012) have given a detailed list of the dye-yielding plants of Uttarakhand. Kundal & Purohit (2014) optimize some new dyeing procedure on cotton and wool fibers. Purohit et.al (2018) explore some plants leaves as a source of natural dye as well as natural mordants.

Rhododendron arboretum is one of the prominent species of the genus *Rhododendron*. The plant bears graceful flowers in the months of March-April which show a great variety in

colour ranging from a deep scarlet to red with white markings, crimson to pale pink or white (Gaur, 1999). The flowers are showy, red in dense globose, cymes (Chauhan, 1999). The dried flowers of *R. arboretum* are supposed to be highly efficient in checking diarrhoea and blood dysentery (Laloo*et al*., 2006). The flowers which are sweet and sour in taste are used in preparing squash, jam, jellies and local brew. The acid-sweet fresh and dried corolla is prescribed when fish bones get struck in the gullet (Pradhan and Lachungpa, 1990). Recently dye from the flowers of *R. arboretum* has been reported by Purohit *et al*., (2008). *Symplocos racemosa* roxb.is an important member of the family Symplocaceae with vital medicinal applications.Talking of its dyeing application, in Indo-China, *Symplocos racemosa* roxb. is used as a mordant while in India its leaves and bark are used to prepare a yellow dye**.** Purohit *et al*., (2015) have reported the leaves of *Symplocos racemosa* as a source of mordant. Use of leaves of the plant as mordant in combination with lemon juice has also been reported (Joshi *et al*., 2013). Colour fastness properties of dye extracted from *Symplocos* species have been evaluated on wool fibers (Singh and Purohit, 2018).

Keeping in mind the need of reviving natural dyes so as to be environment-friendly the present study was undertaken which shows the use of *Rhododendron arboreum* flowers as natural dyes for dyeing woolen fiber. The shade produced was enhanced by employing *Symplocos racemosa* leaves as natural mordant. The entire process was optimized as well.

Figure 1 a) *Rhododendron arboretum*

Figure 1(b) *Symplocos racemosa*

Materials and methods

Collection of plant material: The flowers of *Rhododendron arboreum* were collected in early April from the hills of Khandyusain, Pauri (1700m) while the collection of *Symplocos racemosa* leaves was done from Phalati (Mandakini Valley), Rudraprayag at the elevation of 1100m.

Textile Preparation for Dyeing: Wool has been taken as the dyeing fibre for the present study. To avoid tangling and to allow even penetration of the dye into the fabric the skein of wool was made by wounding around the arm from the hand and over the elbow. The skein was then soaked in the tap water for about 12 h to get rid of the water soluble impurities and finally it was scoured by cleaning with hot water and neutral soap. Scouring also allows uniform dyeing and removes the grease - '*lanolin*' from the fibre.

Determination of Rate of Exhaustion or Percentage Absorbance: The O.Ds (Optical Densities) or % Absorbances of the dye bath before and after dyeing were determined with the help of the UV-Visible Shimadzu spectrophotometer (Model UV-1601). Distilled water was used as the blank to calibrate the instrument to zero absorbance in each case and the wavelength was set to the maximum wavelength (i.e. wavelength of maximum absorbance λ_{max}) of the dye used at that point.

The Rate of Exhaustion or Percentage Absorbance was calculated as follows:

Rate of Exhaustion (%) = (D1-D2)/D1 * 100

Where D1 = O.D. before dyeing; D2 = O.D. after dyeing;

D1-D2 = amount of dye transferred into the fabric after dyeing.

Optimization of Dyeing Procedure:

For the present study, three parameters were taken into consideration-

(1) **Optimum concentration of dye:** After soaking 10g of *R. arboretum* flowers in varying volumes of hot water (200 ml, 400 ml and 600 ml each for 1 h) and heating for a fixed time period (30 min) the solution was filtered and Optical Density (O.D.) of the filtrate was measured. Then the woolen fiber was dipped in the solution for 30 min (Dyeing time = 30 min) , taken out of the solution, squeezed and spread to dry followed by the measurement of the O.D. of the remaining solution. Percentage absorbance or rate of exhaustion was derived in each case with the help of the given formula. The concentration of dye corresponding to maximum percentage absorbance was finalized as the optimum concentration for each sample.

(2) **Optimum time:** The wool samples of optimum concentration (as determined in the previous step) were heated for 15, 30, 45 and 60 minutes each and filtered. The O.D. of the filtrate was determined in each case. Thereafter, the woolen fibre was soaked in the solution for 30 min, taken out, squeezed and spread to dry. Then the O.D. of the left solution was determined and percentage absorbance or rate of exhaustion was derived in each case. The value of time corresponding to maximum percentage absorbance corresponded to the optimum time for each sample.

(3) **Mordanting method:** After getting the optimum concentration and time of the dye, the woolen fabric was subjected to all the three types of mordanting (Pre-, Post- and Simultaneous).

Pre-mordanting: The fiber was first dipped and heated for 10 min in the mordant solution and then dipped in the dye solution and heated further for another 10 min. It was then taken out and spread to dry. The O.D was measured before and after dyeing.

Post-mordanting: The fiber was first soaked and heated in the dye solution and then in an equal volume of mordant solution for 10 min each. It was then taken out and spread to dry. The O.D. was determined before and after dyeing.

Simultaneous mordanting: Equal volumes of the dye and mordant solutions were mixed together and the fiber was dipped in this. The solution was heated for 20 min. The fiber was taken out and spread to dry. O.D. was measured before and after dyeing.

Table 1: Determination of optimum concentration (Weight of sample (flowers) =W=10 g; Volume of hot water taken =V ml; Concentration of the dye (C) = W/V g ml^{-1}; λ_{max}: 238 nm)

V(ml)	C(10/V)(g/ml)	D1	D2	D1 - D2	% Abs.
200	0.05	3.980	2.341	1.639	41.181
400	**0.03**	**3.311**	**1.643**	**1.668**	**50.377**
600	0.02	3.931	2.729	1.202	30.577

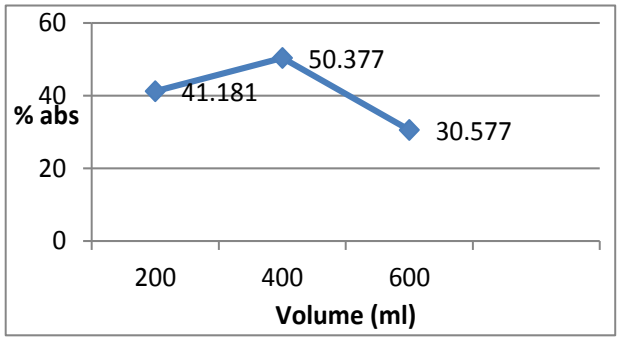

Figure 2: Volume vs % absorbance

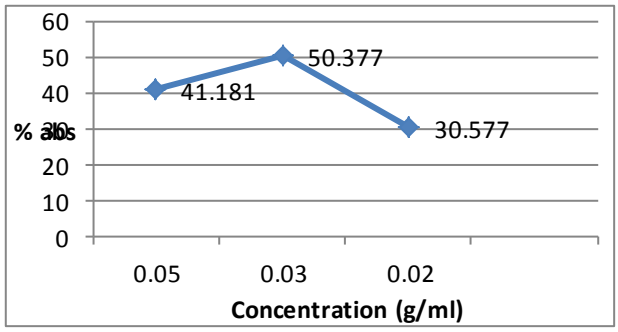

Figure 3: Concentration vs % absorbance

Table 2: Determination of optimum time λ_{max} = 238 nm

Time(min)	D1	D2	D1-D2	% Abs.
15	0.649	0.843	-0.194	-29.890
30	**3.311**	**1.643**	**1.668**	**50.377**
45	3.215	2.642	0.573	17.820
60	2.482	1.689	0.793	31.950

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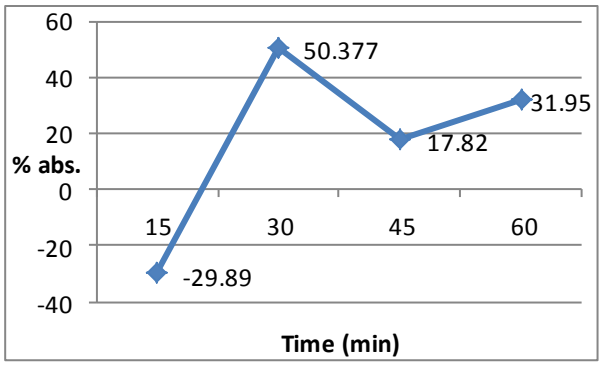


Figure 4: Time vs % absorbance

Table 3: Mordanting of wool with *S. racemosa* leaves (Fibre: Wool Dye: R.*arboreum* flowers Mordant: S. *racemosa* leaves λ_{max} = 416 nm)

Type of Mordanting	D1	D2	D1-D2	% Abs.	Shade obtained
Pre	3.612	2.642	0.97	26.85	Light yellow
Post	3.612	2.341	1.271	35.19	Pale yellow
Simultaneous	3.612	1.689	1.923	53.24	Dark yellow

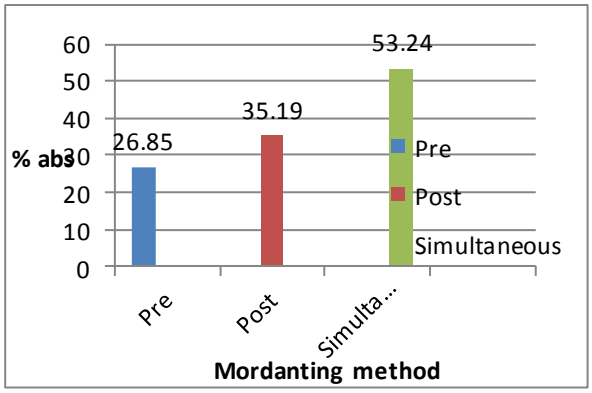
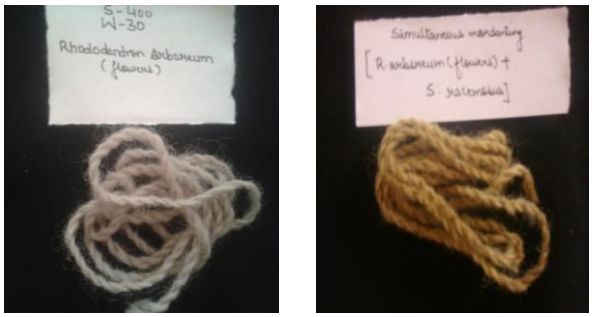


Figure 5: Mordanting method vs % absorbance



(a) Creamish pink (b) Dark yellow

Figure 6: Image of extracted dyes (a-b): *R. arboreum* flowers (400 ml, 30 min) (a); *R. arboreum* (flowers)+S. *racemosa* (leaves): Simultaneous mordanting (b).

Results and discussion

The wool sample corresponding to volume of 400 ml gave maximum percentage absorbance (Table 1) implying that the optimum concentration of R. *arboreum* flowers for dyeing wool is 10g per 400 ml hot water. Table 2 makes it clear that the sample corresponding to 30 minutes showed maximum percentage absorbance. Thus, for dyeing wool 10g of flowers per 400 ml hot water extracted for 30 minutes gave the best result.

The simultaneous mordanting method (Fig. 6) showed the best result when wool was mordanted with S. *racemosa* leaves (Table 3).

Therefore, in accordance with the maximum values of % absorbance in tables 1, 2 and 3 it is clear that:

- Optimum concentration = 10 g *R. arboreum* flowers in 400 ml hot water = 0.03 g ml^{-1}
- Optimum time for dyeing = 30 min
- Optimum mordanting method = Simultaneous mordanting

The woolen fibre dyed with *R. arboretum* flowers only was creamish pink in colour while dark yellow shade was obtained when wool dyed with R. *arobreum* flowers was mordanted simultaneously with S. r*acemosa* leaves. Thus, we see that R. *arboreum* flowers on their own are good sources of natural dyes and using them in combination with S. *racemosa* leaves as natural mordant further produces different shades.

The excellent dye ability exhibited by woolen fiber is attributed to its high values of polarity (2.1) and porosity (Joshi *et al*., 2013).This is because the dye ability increases with the increase of porosity (Rekaby *et al*., 2009; Selvam *et al*., 2015; Hou*et* al., 2013). Also the hygroscopic nature of wool fabric makes it absorb more water due to the presence of several amorphous regions in its molecular chains leading to the swelling of the fibre, thereby increasing the diffusion of the dye solution considerably (Cristea and Vilarem 2016; Mohamed*et al*., 2015; Uddin, 2015). Another reason for the excellent dye ability shown by wool is that the wool molecules are present in the form of flexible chain and bind together with

natural cross-linkages and salt bridges that help the attraction of more dye molecules to the vicinity of the fiber micelles, hence, increasing the dye ability (Lewis, 1992; Gies *et al*., 2017).

Conclusion

The present investigation was planned in search of environmentally benign alternative to the synthetic dyes and mordants, which has been made with the use of *Rhododendron arboretum* flowers as dye and *Symplocos racemosa* leaves as mordant. It was shown that colour yields were found to be enhanced by the addition of mordant at an optimum concentration. Thus, the method used for dyeing the fiber using natural dye and natural mordant instead of synthetic dye and mordant proves to be advantageous as this method uses minimum raw material, water as a solvent which can be reusable in multiple dyeing session and is less toxic, cost effective and eco-friendly. Thus, it proves to be a safe option for the cause of saving environment. Using various parts of plants as source of natural dyes and natural mordants will certainly pave a way in this direction.

Conflict of interest

The authors declare that there is no conflict of interests regarding the publication of this article.

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