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Dyeing of Angora wool with natural dye from bark of beefwood (*Casurina equisettifolia*)

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ABSTRACT

The method of dyeing Angora wool with beefwood (Jor Tor) bark (*Casurina equisettifolia*) was standardised by determining the optimum dyeing conditions, like dye material concentration, dye material extraction time, dyeing time, mordant concentration and mordanting method. The mordants used were Alum, Chrome and Copper Sulphate. The dye material concentration and dyeing time giving the maximum dyeing absorption were taken as optimum values. The extraction time giving the maximum optical density of the dye extract was taken as the optimum value, whereas, the mordant concentration and mordanting method giving the highest washing fastness were taken as optimum concentration and best method, respectively. The washing, rubbing and light fastness of Angora wool, dyed with different mordants were higher than those of Angora wool dyed without mordants. Per cent dye absorption and washing, rubbing and light fastness of dyed Angora wool increased with dye material concentration and dyeing time upto their optimum values, after which, they decreased. Similarly, the optical density of dye extract increased with increase in dye extraction time, upto the optimum value, after which, it decreased.

INTRODUCTION

Dyeing is an ancient art. Dyeing is the art of imparting colour to yarns, fabric and other materials in a variety of hues and tints employing any kind of colouring matter. Colouring matter can be obtained from natural as well as synthetic sources. Earlier dyeing was done by sticking plants to fabric or rubbing crushed pigments into cloth (Annapoorani and Sundarraj, 2014). Natural dyes have been used since prehistoric times for dyeing of various things such as, body, food, cave walls, textiles, leather and objects of daily use. A large number of plant, animal, insect or mineral sources have been identified for extraction of dyes and pigments. The art of dyeing is as old as human civilization. Dyed textiles found during archaeological excavations at different places all over the world give confirmation to the practice of dyeing in ancient civilizations (Krížová, 2016).

India has always been a leader in the trade of dyed textiles and the entire world has always appreciated its exquisite fabrics with excellent colours and designs. The Indian dyeing has been famous for its vivid and diverse colours, with all their mellow and rich dyes. India has a very rich tradition of using natural dyes. The art and craft of producing natural dyed textile has been practiced since ages in many villages by traditional expert crafts-persons in the country (Chakrabarti and Vignesh, 2014). Indian craft has always been far ahead of textile crafts of the rest of the world. Most commonly used dyes upto invention of synthetic dyes in 1850's were indigo and woad for blue, madder, kermes, safflower for red, cutch for brown, turmeric for yellow and myrobalan for black. Indian dyers could produce beautiful colour harmonies with intelligent management of so many colours (Chavan, 1995). In addition to plant dyes animal dyes such as Cochineal-red derived from bodies of cochineal insects and Tyrian purple and crimson-from the bodies of marine snails. Mineral dyes derived from colored clays and earth oxides such as Chrome green, Chrome red, Chrome yellow and Prussian blue were also used (Alemayehu and Teklemariam, 2014). Natural dyes were used not only in clothing, but also in cosmetic industry (Henna, Catechu), pharmaceutical industry (Saffron, Rhubarb) and in food industry (Annatto, Curcumin and Cochineal) (Shahid and Mohammad, 2015 and Melo, 2009).

In 1856, cheaper and easily available synthetic dyes were introduced, which resulted in a dire turn down in the usage of natural dyes. Synthetic dyes were rapidly accepted because they had certain advantages over natural dyes. The synthetic dyes were easy to handle, fast to light and washing, easily available, commercially in standardized form and cheaper due to mass production. Slowly the industry started preferring synthetic dyes over natural ones. As a result, the use of conventional dyes shrunk to very small parts of the country and that too to a very limited extent (Verma and Gupta, 1994).

Almost all synthetic dyes have their origin in coal tar. The synthetic dyes and other chemicals used in their manufacturing are very harmful. The effluents from dye houses pose severe threat to human health as these are carcinogenic and to the environment by causing pollution. On the other hand, natural dyes are very safe to use (Gulrajani *et al.,* 1992). Also, synthetic dyes such as azo dyes are reported to cause allergic reactions (Samanta and Agarwal, 2009). Germany was the first to put ban on production and use of several specific azo dyes. Netherlands, India and some other countries also followed the ban (Patel, 2011).

Natural dyes produce very exceptional, calming and soft shades as compared to synthetic dyes. Natural dyes are biodegradable, non-toxic and non-allergic. In addition to environment friendliness and aesthetic appeal natural dyes provide opportunities for employment generation and utilization of wasteland. Fabric dyed with natural dyes show higher UV absorption which results in reduction of melanoma. Many natural dyes have antibacterial properties. Natural dyes are mostly renewable as most of them are plant based whereas synthetic dyes are petroleum-based that is a non-renewable source of energy. The waste generated can be used as bio-fertilizers in some cases such as dyeing with indigo and as a result there is no waste disposal problem. Despite this, use of natural dyes for dyeing textiles has been limited mainly to artisan/craftsman, small scale/ cottage level dyers and printers as well as to small scale exporters and producers dealing with high-valued eco-friendly textile production and sales. There are more than a few limitations of natural dyes also, such as same shade is difficult to reproduce and mordant is needed to fix the dye onto the fabric. Moreover, skilled man power is required. Natural colourants are expensive and their colour and light fastness is low (Arora *et al.,* 2017).

India has a rich biodiversity and there are more than 450 plant varieties yielding dyes and pigments. However, many of these plant dyes are not yet fully explored for their prospect in dyeing textiles. In addition, there is critical need to work out a suitable, scientific technique for extraction of dyes and their application on textile materials without compromising the quality of the fabric. Majority of the plants used for extraction of dyes are classified as medicinal plants and some of these have recently been shown to exhibit antimicrobial effect (Mahesh *et al.,* 2011). The antibacterial activity of some of these dyes is due to the presence of phenol, tannin and quinine in their extracts. When these dyes were applied to textiles, the antimicrobial properties of these plant dyes contribute to the longer life of the textile materials (Calis *et al.,* 2009).

Natural dyes are moth proof and can replace synthetic dyes in kids garment and food stuff for safety. Despite several limitations, there has been a trend to revive the art of natural dyeing in recent years (Arora *et al.,* 2017). This shift in pattern in favour of natural dyes is also credited to the strict environmental standards imposed by many countries in response to toxic and allergic reactions related with synthetic dyes (Grover and Patni, 2011). Therefore, if natural dyes have to be commercialized, they need to conform to the same stringent standards of performance that are applied to synthetic dyes. It thus follows that a lot of research and developmental effort is needed in this area. The traditional practices may have to be replaced by modern, more scientific practices in order to overcome some of the so-called disadvantages of this dye (Jothi, 2008).

Textile dyeing industry at present uses excessive amount of synthetic dyes to meet the required coloration of global consumption of textiles due to cheaper prices, wider ranges of bright shades, and considerably improved fastness properties in comparison to natural dyes (Uddin, 2015). During the coloration process a large percentage of the synthetic dye does not bind and is lost to the waste stream (Ratna and Padhi, 2012). The effluent from textile industries thus carries a large number of dyes and other additives which are added during the colouring process. However, even after a century, the use of natural dyes never erodes completely and they are still being used. Recently a number of commercial dyers and small textile export houses have started looking at the possibilities of using natural dyes for regular basis dyeing and printing of textiles to overcome environmental pollution caused by the synthetic dyes (Glover and Pierce, 1993).

A new international interest has arisen in natural dyes due to increased consciousness of the environmental and health hazards associated with the synthesis, processing and use of synthetic dyes. Textile processing industry is one of the major environmental polluters as the effluent from these industries contains a heavy load of chemicals including dyes used during textile processing. There are two main ways to limit the environmental impact of textile processing. One is to put up adequately large and highly efficient effluent treatment plants, and the other way is to make use of dyes and chemicals that are environment friendly. Natural dyes, when used alone have many limitations of fastness and brilliancy of shade. However, when used along with metallic mordants, they produce bright and fast colours. Mordants are metal salts which produce an affinity between the fabrics and the dye. Alum, chrome, stannous chloride, copper sulphate, ferrous sulphate etc. are the commonly used mordants (Samanta and Agarwal, 2009). Metal ions of mordants act as electron acceptors for electron donors to form co-ordination bonds with the dye molecule, making them insoluble in water (Mongkholrattanasit *et al.*, 2011). Therefore, instead of using unsustainable technology for producing colours, one can use mild chemistry to achieve almost similar results (Kanchana *et al.*, 2013).

Angora fibres are extremely soft and lustrous fibres. The fibres are produced in small quantities and are found in limited regions around the world. This is the reason the angora products very expensive. Angora fibres are also called "Luxury Fibres". Angora rabbit fibre is one of the world's finest luxury fibres with the values of 10 to 12 micron in diameter and 40 to 70 mm in length. Despite this fineness, the angora fibre has cavities in which air is trapped. Due to these air entrapments, angora has high thermal insulation and is extremely light in weight. These fibres are used in the production of inner and outer garments (hats, sweaters, blankets, etc.) either alone, or by blending with other fibres (generally fine wool) (Riza, 2014). In this paper, standard procedure of dyeing of Angora wool with natural dyes was studied. The effect of different parameters of dyeing on dye ability of Angora wool with Beefwood dye as well as washing, light and rubbing fastness was studied.

MATERIAL AND METHODS

The bark of beefwood (*Casurina equisettifolia*) was collected from Ludhiana. Pure Angora wool was purchased from Kullu, Himachal Pradesh. Alum, chrome and copper sulphate were used as mordants.

Dyeing was carried out at seven concentrations, six extraction times and six dyeing times to determine optimum concentration of dye material, optimum time for extraction of dye material, and optimum time for dyeing, respectively. The dyeing was carried out at four mordant concentrations each employing the premordanting method and optimum dye material concentration, optimum extraction time and dyeing time to determine the optimum concentration of three mordants namely alum, chrome and copper sulphate. Then dyeing with three different methods of mordanting, *i.e.*, Premordanting, simultaneous mordanting and post mordanting in case of each mordant was carried out to determine best mordanting method. Each set of experiments were repeated for reliability.

OBSERVATIONS AND ANALYSIS

The results obtained from the present investigation as well as relevant discussion have been summarized under following heads :

Optimum dye material concentration :

The dye absorption increased with the concentration of the dye material. It was highest *i.e.*, 32.63% at the concentration 2.0 g/g of wool. The dye absorption decreased beyond this concentration of dye material.

Washing, rubbing and light fastness of dyed woollen samples was found to be highest at the concentration of 2.0 g/g of Angora wool (Table 1).

Optimum dye material extraction time :

Optical density increased with extraction time of the dye material, it being maximum *i.e.*, 0.233 at the extraction time of 90 minutes. Depth of shade also increased with extraction time but it was not deepest at optimum extraction time. Washing, rubbing and light fastness were also maximum at 90 minutes of extraction (Table 2).

Optimum dyeing time:

The dye absorption increased with increase in dyeing time of dye material, it being maximum, *i.e.*, 24.57% at the dyeing time 90 minutes. The dye absorption decreased beyond it. Washing, rubbing and light fastness were highest at optimum dyeing time (Table 3).

Optimum mordant concentration:

The washing, rubbing and light fastness grades of woollen samples, premordanted at different concentrations of each mordant, namely alum, chrome and copper sulphate, employing concentration of dye material 2.0 g/g of wool, dye material extraction time 90 minutes and dyeing time 90 minutes was tested.The optimum concentration of alum mordant on the basis of washing, rubbing and light fastness was found to be 27.5%. In case of chrome mordant the optimum concentration was 4.0 per cent. The optimum concentration for copper sulphate mordant was found to be 2.0 per cent (Table 4).

Best mordanting method:

Three processes of mordanting were used-pre mordanting, simultaneous mordanting and post mordanting. After dyeing, the dyed material was washed with cold water and dried at room temperature (Pruthi *et al.*, 2008; Jothi, 2008 and Suitcharit *et al.*, 2010). The washing, rubbing and light fastness grades of woollen samples mordanted with different mordanting methods, employing Beefwood dye material concentration 2.0 g/g of wool, extraction time 90 minutes, dyeing time 90 minutes, concentration of alum, chrome and copper sulphate being 27.5 per cent, 4.0 per cent and 2.0 per cent, respectively in each mordanting method were determined.

Table 1 : Optimization of Beefwood (Jor-Tor) dye material concentration for Angora wool (Dye material extraction time 1 hr., dyeing time 1 hr., wavelength 370 nm)

Sr. No.	Conc. of dye material g/g of wool	Optical density before dyeing	Optical density after dyeing	% dye absorption	Washing fastness grade: Colour change	Washing fastness grade: Staining Wool	Washing fastness grade: Staining Cotton	Rubbing fastness grade: dry	Rubbing fastness grade: wet	Light fastness grade
1.	1.0	0.103	0.083	19.41	3	4	3-4	3	2-3	2
2.	2.0	0.144	0.097	32.63	4	4-5	4-5	4	3-4	2-3
3.	3.0	0.185	0.150	18.91	4	4	4	3-4	3	2
4.	4.0	0.229	0.199	13.10	3-4	4	3-4	3	2-3	2
5.	5.0	0.240	0.223	7.08	3-4	3-4	3-4	3	2-3	1-2
6.	6.0	0.257	0.247	3.89	3	3-4	3	3	2-3	1-2
7.	7.0	0.274	0.267	2.55	3	3	2-3	2-3	2	1-2

Table 2 : Optimization of beefwood dye extraction time for Angora wool (dye material concentration 2g/g of wool, dyeing time 1 hr. wavelength 370 nm)

Sr. No.	Extraction time (min)	Optical density	Washing fastness grade: Colour change	Washing fastness grade: Staining Wool	Washing fastness grade: Staining Cotton	Rubbing fastness grade: Dry	Rubbing fastness grade: Wet	Light fastness grade
1.	30	0.124	3	3-4	3	3	2-3	2-3
2.	60	0.191	3	3-4	3-4	3-4	3	2-3
3.	90	0.233	4	4	4	4	3-4	3
4.	120	0.189	3-4	3-4	3-4	3-4	2-3	2-3
5.	150	0.180	3	3-4	3-4	2-3	2	2
6.	180	0.131	3	3	3	2-3	1-2	2

The best method of mordanting for alum and copper sulphate was post mordanting. In case of chrome it was premordanting (Table 5).

Conclusion :

The whole process of extraction and dyeing is ecologically safe (Kulkarni *et al.*, 2011). The present study was conducted to develop the standard methods of dyeing with beefwood dye on Angora wool and to determine their washing, rubbing and light fastness and the effect of different conditions of dyeing on washing, rubbing and light fastness. It was found that the highest

Table 3: Optimization of beefwood dyeing time for Angora wool (dye material conc. 2g/g of wool, extraction time 90 min., wavelength 370 nm)

Sr. No.	Dyeing time (min)	Optical density before dyeing	Optical density after dyeing	% dye absorption	Washing fastness grade			Rubbing fastness grade		Light fastness grade
					Colour change	Staining		dry	wet	
						Wool	Cotton			
1.	30	0.129	0.118	8.52	3	3	3	3	2-3	2-3
2.	60	0.120	0.101	15.83	3	3-4	3	3-4	3	2-3
3.	90	0.118	0.089	24.57	4	4	4	4	3	3
4.	120	0.122	0.104	14.75	3-4	3-4	3-4	3-4	2-3	2-3
5.	150	0.119	0.109	8.40	3-4	3-4	3	3	2-3	2
6.	180	0.118	0.112	5.08	3	3	2-3	3	2	2

Table 4 : Optimization of mordant concentration (Dye material conc. 2g/g of wool, extraction time 90 min, dyeing time 90 min)

Sr. No.	Type of mordant	Per cent mordant conc.	Washing fastness grade			Rubbing fastness grade		Light fastness grade
			Colour change	Staining		Dry	Wet	
				Wool	Cotton			
1.	Alum	22.5	3	2	1-2	3	1-2	2
		25.0	3	2	2	3	2	2
		27.5	3-4	3	2-3	3-4	2-3	2-3
		30.0	2-3	1-2	1	2-3	1-2	2
2.	Chrome	2.0	4	3-4	3	3	2-3	1-2
		3.0	4	3-4	3-4	3	3	2
		4.0	4	4-5	4	4	3-4	3
		5.0	4	4	3-4	3	3	2-3
3.	Copper sulphate	2.0	4	3-4	3-4	3-4	3	2-3
		3.0	3-4	3-4	3	3	2-3	2
		4.0	3-4	3	3	2-3	2-3	2
		5.0	3	3	2-3	2	1-2	1-2

Table 5 : Optimization of mordanting method (dye material conc. 2g/g of wool, extraction time 90 min, dyeing time 90 min, conc. of alum 27.5 %, conc. of chrome 4.0%, conc. of copper sulphate 2.0%)

Sr. No.	Method of mordanting	Type of mordant	Washing fastness grade			Rubbing fastness grade		Light fastness grade
			Colour change	Staining		Dry	Wet	
				Wool	Cotton			
1.	Pre mordanting	Alum	3-4	3	2-3	3-4	2-3	2-3
		Chrome	4	4-5	4	4	3-4	3
		Copper sulphate	4	3-4	3-4	3-4	3	2-3
2.	Simultaneous mordanting	Alum	4	3-4	3	3-4	3-4	2
		Chrome	4	4	3-4	3-4	3	2-3
		Copper sulphate	4	4	4	3	3	2-3
3.	Post mordanting	Alum	4	3-4	3-4	4	3-4	3
		Chrome	4	4	4	3-4	3-4	2
		Copper sulphate	4-5	4-5	4-5	3-4	3-4	3-4

fastness to washing, rubbing and light was achieved when Angora wool was dyed with beefwood dye at a concentration of 2g/g of wool, extraction time of 90 minutes, dyeing time of 90 minutes. The optimum concentration of alum, chrome and copper sulphate was 27.5%, 4.0% and 2.0%, respectively. Best mordanting method for alum was post mordanting, for chrome it was pre mordanting and in case of copper sulphate it was post mordanting.

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