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Effect of Synthetic and Natural Mordants on Nettle Plant Fiber Dyed with *Rubia Cordifolia*

Rashmi Bala, Manisha Gahlot, Beenu Singh

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ABSTRACT

The major process in the production of textile material is colouration of fibre. In this study, natural dye extracted from the madder (*Rubia cordifolia*) roots was used to dye nettle (*Girardinia diversifolia*) fibres using selected synthetic and natural mordants. The synthetic mordants used in the study were potassium aluminium sulphate (alum) and ferrous sulphate whereas the natural mordant used was gallnut extract. Nettle fibres were dyed using each of the selected mordants with three conventional mordanting techniques; pre-mordanting, simultaneous-mordanting and post-mordanting. A range of soft and light shades were obtained with the use of mordants, also the mordanting techniques influenced the results of the dyeing. Natural mordant gave light shades of brown colour while synthetic mordant such as alum gave pink shades and ferrous sulphate produced grayish and black tones. The colour strength measurement of mordanted dyed fibres was conducted using spectrophotometer. The wash, rub and light fastness of dyed samples have been evaluated, giving fair to excellent fastness grades. Fourier-transform infrared (FTIR) analysis of untreated nettle fibre, gallnut treated nettle fibre, ferrous sulphate treated nettle fibre and alum treated nettle fibre shows presence of hydroxyl groups, COC vibrations of glycoside bonds.

Keywords Mordants, Natural dye, Nettle fibre, *Rubia cordifolia*, *Girardinia diversifolia*.

INTRODUCTION

Natural dyes are known for their use in colouration on food substrates as well as for colouration of textile materials like fibres, yarns and fabrics and are used since prehistoric times (Samanta and Aggarwal, 2009). Dyeing is an ancient art and natural dyes/ colourants have been used historically throughout the world for colouration of textile material. With the discovery of synthetic dyes, the use of natural dyes reduced rapidly contradicting their use till 1900 (Cristea and Vilaren, 2006). Despite of bright colours, ease of application and availability, synthetic dyes also have certain disadvantages. A large number of synthetic dyes ate toxic and carcinogenic in nature and my lead to skin allergies, skin cancer and irritation (Elango *et al*. 2011). In a survey it was found that there was a substantial increase in the number of consumers who are concerned with harmful residues on textiles

Rashmi Bala[1], Dr Manisha Gahlot[2], Dr Beenu Singh*[3]
[1]Dr Beenu Singh, [2]Professor, [3]Senior Research Fellow,
Clothing and Textiles Department, College of Home Science, GBPUAT, Pantnagar Uttarakhand 263153, India

Email: beenuindolia8@gmail.com
*Corresponding author

and the ecological impacts of textile manufacturing. Consumers are becoming more aware about environment and health issue (Henriques and Shankar, 2007).

Dye and textile industries had gained attention towards newer products which compliments fashion trends and are eco-friendly (Caldwell, 2017). Natural dyeing practices are helpful in promoting rural entrepreneurship by creating employment opportunities in rural areas (Srithong, 2007). Natural dyed products can be promoted in domestic and international niche market as ecological safe products among the consumers. The stem of *Rubia cordifolia* (The Indian madder; family: Rubiaceae), a very variable prickly creeper or climber, commonly available throughout India is chosen for the present work. Metal Salts or Metallic Mordants Metal salts of alum and ferrous sulphate are used. Maximum absorption of dyes on fibres was obtained at 3% concentration of dyes obtained from *Rubia cordfolia* (35.350%) Bhuyan and Saikia, (2005). According to Vankar (2000), mordant helps in the absorption and fixation of natural dyes. It also helps in improving the colour fastness properties of the dyed textiles. The natural dyes have limited substantively for the fibre. They require mordant, which enhances the fixation of the natural colourant on the fibre by the formation of the complex with the dye. Alum, potassium dichromate, ferrous sulphate, copper sulphate, zinc sulphate, tannin and tannic acid are some examples of mordants used for the fixation of natural dye to the natural fibre (Maulik and Pradhan, 2005).

MATERIAL AND METHODS

The fibres selected for this study was nettle fiber delignified with three step optimized process involving use of Sodium chlorite, Sodium hydroxide and acetic acid. The dyeing of nettle was carried out at optimized dyeing conditions which were 3% dye concentration, 45 minutes dyeing time and 1:15 dyeing M:L ratio and dyeing temperature was kept constant at 90°C. Two synthetic mordants namely alum and ferrous sulphate and one natural mordant i.e., gallnut was used.

For optimization of mordanting method three methods of mordanting that is pre, post and simultaneous mordanting were used. Selection of mordanting method was done on the basis of colour strength of dyed and mordanted fibres. Mordanting was done with selected method with different concentrations of mordants. For natural mordant gallnut 5, 10, 15, 20 % concentrations (w/v) were taken and for synthetic mordant ferrous sulphate 0.25, 0.5, 0.75, 1.00 %, alum, 3, 5, 7, 9 % concentrations were taken. After the dyeing, samples were rinsed and dried under the shade.

Colour strength

Colour strength of dyed and mordanted fibre samples was determined at 360 nm wave length by measuring surface reflectance of the samples using a SS5100A spectrophotometer and Colour Lab plus software. The concentration of mordant with highest K/S on dyed and mordanted fibers was selected for the further study.

Colour fastness to washing, crocking and light

Colour fastness to washing of dyed and mortdanted fibre samples was determined as per standard method IS:3361-1979, Test no. 2 using a laundrometer. The wash fastness ratings were assigned using grey scales namely ISO-105-A03 for change in colour and ISO-105-A03 for extent of staining. Colour fastness to rubbing (dry and wet) was assessed as per IS:766-1956 standard test method using a manually operated crockmeter and grey scale ISO-105-A03 for extent of staining.

Colour fastness to light was determined as per IS:2454-1985 standard test method. The dyed and mordanted samples along with 8 blue wool standards were exposed to artificial day light using a Digital Light Fastness Tester. It consisted of 500 watt philips mercury bulb tungsten filament lamp which simulated day light. Light fastness ratings were assigned using blue wool standards.

Fourier Transform Infrared (FTIR) testing of dyed and undyed fibers

FTIR is a sensitive technique widely used to identify organic compounds and inorganic compounds by recording infrared spectra. FTIR of treated and untreated nettle fiber was done using Bruker's ALPHA II Compact FTIR spectrometer.

RESULTS AND DISCUSSION

Optimization of mordanting method

Colour strength values (K/S) of dyed nettle fibres using different mordanting method is shown in Table 1. K/S value of control sample was 5.834, which was found to be mordanting of fibres with natural and synthetic mordants. According to the results given in Table 1, it can be observed that nettle fibre mordanted and dyed with gallnut extract showed maximum K/S value of 7.534 for simultaneous mordanting method. The K/S values observed for pre and post mordanting methods were 6.067 and 5.247 respectively. It can also be observed from the table that maximum colour strength was observed for pre mordanting in case of synthetic mordants i.e., ferrous sulphate (10.971) and alum (5.90). Recorded values of K/S were 9.785 and 10.474 for simultaneous and postmordanting method respectively in case of Ferrous sulphate mordant. In case of alum mordant K/S values for simultaneous and postmordanting methods were 5.357 and 4.08 respectively. In most of the cases K/S values were found to be improved after mordanting except for samples mordanted with post and simultaneous mordanting using alum and post mordanting using gallnut. Dyed samples with natural and synthetic mordants using different methods of mordanting have been shown in Plate 1.

Table 1. Colour strength values of dyed nettle fibres with different methods of mordanting for natural and synthetic mordants.

	Mordants	Method of mordanting	K/S
Natural	Gallnut	Pre	6.067
	(Quercusinfectoria)	Simultaneous*	7.534
	(15% w/v)	Post	5.247
Synthetic	Ferrous sulphate	Pre*	10.971
		Simultaneous	9.785
	(0.5% w/v)	Post	10.474
	Alum	Pre*	5.905
	(15% w/v)	Simultaneous	5.357
		Post	4.080
Control sample (dyed without mordant)			5.834

*Selected method of mordanting (Dye conc - 3%, Dyeing Time – 45 minutes, M:L ratio -1:15, Mordanting time - 30 minutes, Mordanting Temp – 90°C).

Optimization of concentration of mordant

Optimized mordanting methods were selected for further experiments. Samples mordanted with gallnut mordant exhibited highest K/S (7.206) by using 5%

Plate 1. Dyed samples with natural and synthetic mordants using different methods of mordanting.

Table 2. Colour strength values of dyed nettle fibres using different concentrations of natural and synthetic mordants.

	Mordants	Concentration of mordant (w/v)	K/S
Natural			
	Gallnut (Simultaneous mordanting)	5*	7.206
		10	5.956
		15	6.418
		20	6.832
Synthetic			
	Ferrous Sulphate (Premordanting)	0.25	12.05
		0.50	15.72
		0.75*	15.74
		1	14.65
	Alum (Premordanting)	3	4.913
		5	5.295
		7*	6.104
		9	3.885
Control sample (dyed without mordant)			5.834

*Selected concentration of mordant (Dye conc -3%, Dyeing Time -45minutes, M:L ratio -1:15, Mordanting time -30 minutes, Mordanting Temp – 90°C).

concentration. In case of Ferrous sulphate and alum 0.75% and 7% concentrations were selected on the basis of highest K/S values i.e., 15.74 and 6.104 respectively. The selected concentration showed higher colour strength values as compared to control shown in Table 2. Dyed samples with natural and synthetic mordants using different concentrations is given in Plate 2.

Assessment of colour fastness properties of the dyed and mordanted nettle Fibres

The dyed nettle fiber samples mordanted with gallnut extract showed very good (6) light fastness property. In case of samples mordanted using synthetic mordants i.e Ferrous sulphate and alum, the light fastness rating was found good (5) shown in Table 3.

The good results of washing fastness were exhibited by the sample mordanted with gallnut mordant with rating of 4 i.e slight change in colour with negligible staining (5) on adjacent wool fabric whereas in case of adjacent cotton fabric noticeable staining (3) was observed. In case of alum mordant noticeable colour change (3) was observed and noticeable staining (3) was found on cotton and slight staining (4) was seen on wool fabric (Table 3).

Plate 2. Dyed samples with natural and synthetic mordants using different concentrations.

FTIR Analysis

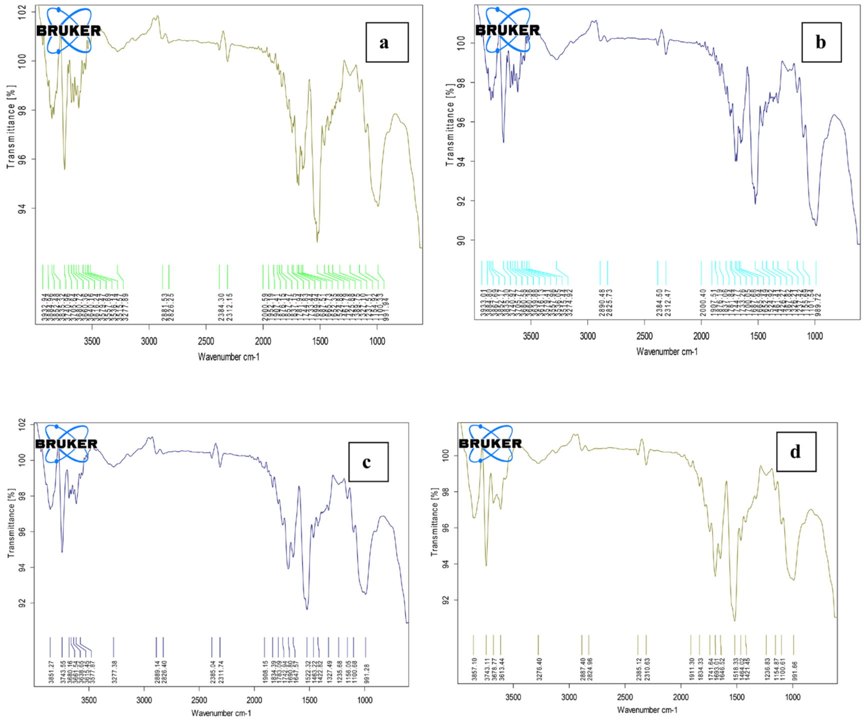

Fig. 3. FTIR Spectra of (a) untreated nettle fibre and (b) gallnut treated nettle fibre (c) ferrous sulphate treated nettle fibre and (d) alum treated nettle fibre.

In case of mordanting with ferrous sulphate considerable change in colour (2) was observed but negligible staining (5) on adjacent wool fabric was reported and noticeable colour staining (3) was observed on adjacent cotton fabric.

The tabulated results reveal that negligible colour change (5) was observed in case of both dry and wet rubbing fastness tests for all three mordants. While analysing the staining, negligible staining was observed for all, in both wet and dry rubbing. It may be due to improvement in fastness properties with the use of mordants.

Table 3. Fastness grades of sunlight, washing, dry and wet rubbing of dyed fibres.

Type of dye	Treatments	Colourfastness grades							
		Light	Washing			Rubbing			
						Dry		Wet	
			CC	CS		CC	CS	CC	CS
				C	W				
Natural % (optimized concentration)	Gallnut	6	3	5	4/5	5	5	5	5
	Alum	6	3	4	4/5	5	5	5	5
	Ferrous sulphate	6	3	5	4/5	5	5	5	5
Control sample		6	3	4	4	5	5	5	5

FTIR Analysis

FTIR spectra were obtained for the untreated (a), gallnut treated (b), ferrous sulphate treated (c) and alum treated (d) nettle fibres is shown in Fig. 3. In the untreated and gallnut treated fibre strong transmittance peaks were observed at 3740cm^{-1} and the increase in peak intensity was found at 3743 cm^{-1} in ferrous and alum treated nettle fibre which indicated the presence of hydroxyl groups. The another peak intensity was found at 1524cm^{-1} in untreated and gallnut treated nettle fibre which was reduced to 1518cm^{-1} in alum treated and 1522cm^{-1} in ferrous treated which showed that there was removal of lignin content from the mordanted fibre. The peak intensity at 991 cm^{-1} in untreated, ferrous and alum treated fibre showed the presence of COC vibrations of glycoside bonds.

CONCLUSION

The work conducted above demonstrated that madder can be used to successfully dye nettle fibre. A wide range of soft and light colours could be derived by using different mordants and mordanting techniques. Synthetic mordants performed better with madder dye in this study as a source of dyestuff than natural mordant. Nonetheless, it was found that as natural mordant gallnut if used under simultaneous mordanting technique; ferrous sulphate and alum under premordanting technique produce good results yielding satisfactory colour fastness.

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