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Studies on the Effect of Mordants on Dyeing Behaviour of Bamboo Fibres using Natural Dyes

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Abstract – **Dyeing of bamboo fibres using two natural dyes derived from Turmeric (rhizomes of Curcuma longa) and Tea (Camellia sinensis) have been investigated. The dyeing process was carried out on premordanted bamboo fibres (retted, scoured & bleached), using pre-extracted dye stock employing 100% water as extraction medium. The effect of mordants on the dyeing outcome in terms of shade output as well as fastness properties of the dyeing has been rationalized. The results of fastness properties of the dyed fibres were fair to good. Exhaustion profile of dyeing with respect to time was examined by taking out samples at different intervals of time.**

Keywords – **Tea, Turmeric, Bamboo Fibres, Mordanting, Exhaustion, Fastness Properties.**

I. Introduction

The plant empire provides a good resource of natural dyes which can be produced from many plant parts e.g., seeds, leaves, flowers, fruits, roots and barks. Conventionally, the natural dyes are obtained from leaves, roots, flowers or bark of some plant genus generally by boiling, powdering and extracting by different organic solvents *viz*. acetone, methanol and acetonitrile to find the desired color. It is worthwhile to mention that till now either plant extracts have been used for dyeing or color components isolated from the plants are employed for this purpose. Though, the above mentioned techniques for extraction are linked with certain limitations like variability in tinting strength due to type of extraction, environmental & seasonal variations and storage conditions of extracted dye. For reproducibility of shades, the optimization of application procedures and the test of compatibility of natural dyes are important for textile industry.

Turmeric is a natural substantive dye derived from rhizomes of *Curcuma longa* which can be applied either directly on textile substrates without any supporting chemicals or in the presence of mordants [1]. The powdered rhizome of this plant is used as a yellow dye especially in coloring the food items. The main active substance of turmeric is curcumin [2] is an active ingredient in turmeric (*Curcuma longa L.*), a rich source of producing a bright yellow color [3]. It is also known as C.I. Natural Yellow 3 and chemically known as 1, 7-bis (4-hydroxy-3-methoxyphenyl)-1, 6-heptadiene-3,5-dione [2].

Tea (*Camellia sinensis*) is the most used beverage which contains many compounds like caffeine, polyphenols, fluoride, amino and organic acids [4]. The degree of fermentation and the oxidation of the polyphenols present in the tea during its processing method categorizes it into six classes/colours such as green, yellow, dark, white, oolong and black [5]. Of the polyphenols, catechins [6] like epicatechin, epicatechin gallate, epigallocatechin and epigallocatechin gallate are the principle colorants. Tsujimura determined the chemical structures of catechins (natural phenol antioxidant plant).

In the context of above discussion, the present work was planned to deal with the following objectives: (1) extract color rich fraction from the source (2) examine the dyeing characteristics on bamboo fibres. For this work we have selected (A) Turmeric (rhizomes of *Curcuma longa*) and (B) Tea leaves (*Camellia sinensis*) as the plants of selection because these are abundantly available throughout the northern and western Himalayan region of India.

II. Materials and Experiments

A. Materials

Raw culm of *Bambosa vulgasis* was harvested from Botanical Garden of Guru Nanak Dev University, Amritsar. It was further processed with CAN retting technique followed by scouring with 15 g/L Na_2CO_3 and finally treated with NaOH (0.3N) followed by peracetic acid bleaching. All the chemicals used in this investigation were of AR grade and were purchased from Merck Ltd., Hi-media Labs, Bombay (India).

Turmeric (rhizomes of *Curcuma longa*) was grown and cultivated in Guru Nanak Dev University Campus, Amritsar and rhizomes were collected, hot water washed and air-dried in dust-free environment. The rhizomes were grounded to the dry powder and were used without any particular purification.

Tea (*Camellia sinensis*) leaves were kindly provided by Dr. Arvind Gulati, Chief Scientist and Head, Hill Area Tea Science Division CSIR-IHBT, Palampur.

B. Experimental Methods

The experimental methods were categorized as follows

(a) The retted, scoured and bleached bamboo fibres were divided into three parts. Pretreatmenting of one part was done with 5% Alum (owf) and second one with 5% Tannic acid (T Acid) (owf) and the rest was treated first with 2.5% (owf) Alum followed by intermediate drying and then further treated with 2.5% T Acid (owf).

(b) Extraction of dye from the source.

(c) The premordanted bamboo fibres were dyed with Turmeric & Tea stock solution extracted from the source, through exhaustion application method.
(d) Exhaustion profile of dyeing with respect to time was examined by taking out samples at different intervals of time.
(e) The effects of mordants on the dye strength were investigated through the measurement of K/S.
(e) Fastness properties (washing & light fastness) of the dyed samples were measured and compared.

C. Pre-mordanting of bamboo fibres

1. Premordanting with Alum & T Acid

The following conditions were used for premordanting.
Concentration of mordant used = 5% (owf)
Temperature = Boil
Time = 45 min

The mordanted samples were dried and then taken for dyeing.

2. Premordanting with Alum followed by T Acid (Alum → Tannic acid)

The following conditions were used:
Concentration of mordant used = 2.5% (owf) Alum
2.5% (owf) Tannic acid
Temperature = Boil
Time = 90 min

In this retted, scoured & bleached bamboo fibres were first treated with Alum for 45 min and then treated with T Acid for 45 min.

3. Extraction of dye from the source

2 g source was treated with 1000 ml of water at 70 ^{o}C for 1 hour**.** The solution was then filtered and stored at 4 ^{o}C. The extract was again transferred at 70 ^{o}C in 1000 ml for another 1 hour and filtered, and then this filtrate was mixed with first filtrate. This filtrate was used for dyeing without any addition of water. The optical density of the filtrate was measured using absorbent type UV-vis spectrophotometer. The optical density of the filtrate was found to be 0.7969 (Tea extract) & 2.235 (Turmeric extract).

4. Dyeing of premordanted bamboo fibres

Dyeing conditions used:
Dyes used = Turmeric & Tea
NaCl = 5 % (owf)
Dye concentration = The extracted liquor was used keeping the M :: L ratio at 1 : 30
Temperature = 70 ^{o}C
Time = 60 min

III. TESTING

1. Examination of exhaustion profile with respect to time

The dyeing at each sample was started at 70 ^{o}C simultaneously in seven baths keeping same liquor ratio in all the baths. The baths were removed after 10 min., 15 min., 20 min., 30 min., 60 min., 90 min. & 120 min. The optical densities (OD) of these baths were measured using spectrophotometer.

As OD (optical density *i.e.* absorbency) is proportional to concentration of dye solution, so by using the following relationship, percentage dye exhaustion was calculated.

$$E = \frac{A_0 - A_1}{A_0} \times 100$$

Where, E= Dye exhaustion (%)
A_0 = Absorbance of dye liquor before dyeing (OD_0)
A_1 = Absorbance of dye liquor after dyeing (OD_1)
All these measurement was done in an absorbent type UV-vis spectrophotometer (PERKIN ELMER, Lambda 25).

2. Colour measurement

The K/S values [7] of the dyed samples were measured (D_{65} illuminant, 10^{o} observer) using spectra flash 600 Spectrophotometer interphased with computer colour matching system. Colour strength is expressed as K/S value and can be expressed by the Kubelba – Munk equation given below:

$$\frac{K}{S} = \frac{(1 - R_{\lambda \max})^2}{2R_{\lambda \max}}$$

Where, K = Coefficient of absorption
S = Coefficient of scattering
R = Reflectance of the substance at wavelength
λ_{max} = Maximum absorbance wavelength

3. Washing fastness

Color fastness to washing was evaluated using standard ISO C06 C2S test [8]. ECE reference detergent (4gL^{-1}) and sodium perborate (1gL^{-1}) at pH 10.5 and steel balls (25) were employed in the test. A 10 x 4 cm dyed fibres in the form of strip attached through the short ends to SDC's multifibre test fabric, was washed on a Washtec (RBE) at 60 ^{0}C for 30 min, the samples were rinsed with cold water, air dried and analyzed for the color change and staining.

4. Light fastness

Light fastness was determined using ISO: 105 B02: 2000 test [8] using xenon arc lamp and blue wool reference standards.

IV. RESULTS AND DISCUSSION

In this section, the effects of various mordants on exhaustion and fastness properties have been reported. Mordanting is used to impart better fixation of a dye having less affinity to textile material. The mordants act as a link between the dye and the fibre. (Figure 1).

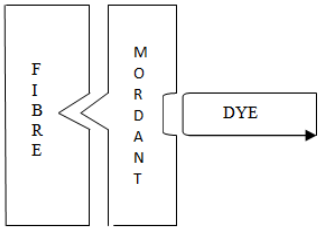

Fig.1. Mordanting: The dye can only bind strongly to the fibre when the mordant act as a link between the two.

In this study, three types of premordanting chemicals have been employed *viz.,* Alum, Tannic acid (T acid) &

Alum → Tannic acid (first with Alum, then dry and then treatment with Tannic acid). The results of these variations have been reported here.

1. Effect of different mordants on dyeing behavior of bamboo fibres using Turmeric as natural dye

The exhaustion profile and K/S values of Turmeric extract on bamboo fibres using different mordants *viz.* alum, tannic acid and alum followed by tannic acid are given in Table 1, Table 2, Table 3, Figure 2 and Figure 3. It is seen from Table 1 & Figure 2 that percent exhaustion gradually increased up to 60 min and after that a slow rise in exhaustion was observed up to 90 min. After 90 min, it found that there was certain drop in exhaustion %. This was because, after 60 min, the material was gradually reaching the equilibrium and slight dye desorption was occurring. Turmeric is a substantive dye and it forms some complex with alum and gets fixed on the material. Reasonable good percent exhaustion was obtained using alum as mordant i.e., 64.4 %. The absorbance values in Table 1 show that the samples with the lower colour yields had higher dyebath exhaustions. A possible explanation may be that a major part of the superficially held mordant-dye complex was washed out during soaping.

Table 1: Exhaustion profile of Turmeric extract using Alum as mordant

Dyeing Time (min)	Exhaustion %	K/S (after washing)
0	0	*0.9
10	49.0	4.6
15	50.9	4.9
20	52.5	4.9
30	55.9	5.3
60	64.4	5.8
90	64.5	5.9
120	64.1	4.3

K/S value of mordanted sample = 0.9

Table 2 & Figure 3 show that the percent exhaustion gradually increased with increase in time, but there was certain drop in percent exhaustion after 60 min. This was so because dye desorption was occurring and the material was gradually reaching the equilibrium. The percent dye exhaustion in Table 2 increased from 0% to 41% but the K/S value was unchanged. During this interval of dyeing, dye molecules seems to get absorbed superficially but removed after washing.

Table 2: Exhaustion profile of Turmeric extract using Tannic acid as mordant

Dyeing Time (min)	Exhaustion %	K/S (after washing)
0	0	*2.9
10	41.0	2.9
15	44.1	3.5
20	47.0	3.7
30	50.3	4.0
60	55.9	4.4
90	54.7	4.1
120	53.0	4.0

K/S value of mordanted sample = 2.9

Table 3: Exhaustion profile of Turmeric extract using Alum → Tannic acid as mordant

Dyeing Time (min)	Exhaustion %	K/S (after washing)
0	0	*1.9
10	54.5	4.2
15	57.7	5.1
20	60.2	6.1
30	65.5	7.3
60	70.2	7.7
90	69.3	7.6
120	68.1	7.7

K/S value of mordanted sample = 1.9

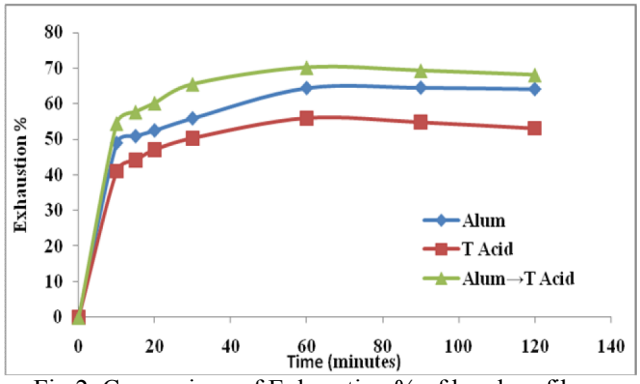

Fig.2. Comparison of Exhaustion % of bamboo fibres dyed with Turmeric extract using different mordants

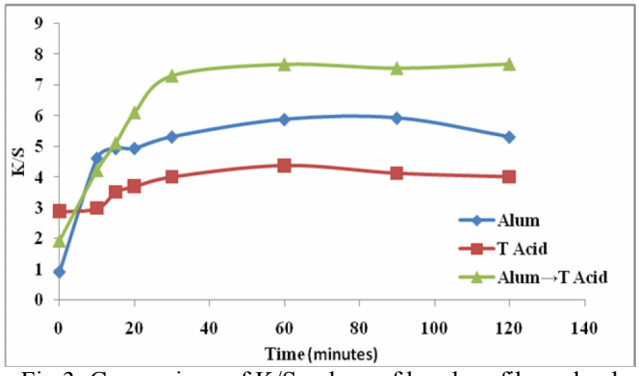

Fig.3. Comparison of K/S values of bamboo fibres dyed with Turmeric extract using different mordants

By comparing Table 1 & Table 2, we found that percent exhaustion using Tannic acid as mordant is poorer than using Alum. We also found that the initial K/S values of Tannic acid treated samples were sufficiently higher than K/S values of Alum treated samples. Also it can be seen from Table 3 & Figure 4 that percent exhaustion gradually increased with increase in time, but there was a certain drop in percent exhaustion after 60 min. Best results were obtained in this combination of Alum & Tannic acid i.e. 70.2 %. The decrease in color strength for 90 and 120 min of dyeing treatment may be credited to desorption of the dye molecules as result of prolong dyeing [1].

There was certain drop in percent exhaustion in all the three mordanting systems after 60 min, therefore, for further examination of properties, this sample (treated for

60 min) was chosen as standard sample for further fastness properties.

2. Effect of different mordants on dyeing behavior of bamboo fibres using Tea as natural dye

The exhaustion profile and K/S values of Tea extract on bamboo fibres using different mordants *viz.* alum, tannic acid and alum followed by tannic acid are given in Table 4, Table 5, Table 6, Figure 4 and Figure 5. It is seen from Table 4 that the percent exhaustion gradually increases with the increase in time, but after 60 min, there is slightly drop in exhaustion %. This was because, after 60 min, the material was gradually reaching the equilibrium and slight dye desorption was occurring. Tea forms some complex with Alum (Figure 1). Tea extract contains polyphenols which form coordinate links with Alum. The resulting dye complex formed during this process is similar that in the metal- complex dyes. Therefore dyes were fixed on the material. Reasonable good percent exhaustion was obtained using Alum as mordant i.e.57.2 %

It has been found in Table 5, Figure 4 & Figure 5 that the percent exhaustion gradually increased with increase in time, but there was also a certain drop in percent exhaustion after 60 min, same as in case of Turmeric. This was so because dye desorption was occurring and the material has been gradually reaching the equilibrium. By comparing Table 4, Table 5, & Figure 4, it has been concluded that percent exhaustion using Tannic acid as mordant is poorer than using Alum. It has also been found that the initial K/S values of Alum →Tannic acid treated samples were sufficiently higher than K/S values of Alum treated samples. It was observed from Table 6 & Figure 5 that percent exhaustion has been gradually increased with increase in time, but there was certain drop in percent exhaustion after 60 min. Thus the highest exhaustion % (68.3 %) was obtained in this combination i.e. Alum →Tannic acid (60 min).

Table 4: Exhaustion profile of Tea extract using Alum as mordant

Dyeing Time (min)	Exhaustion %	K/S (after washing)
0	0	*0.9
10	48..0	3.2
15	49.5	4.6
20	50.6	5.9
30	54.0	8.2
60	57.2	9.6
90	57..0	9.3
120	56.3	9.1

K/S value of mordanted sample = 0.9

Table 5: Exhaustion profile of Tea extract using Tannic acid as mordant

Dyeing Time (min)	Exhaustion %	K/S (after washing)
0	0	*2.9
10	36.0	4.4
15	42.4	5.0
20	44.7	5.6
30	51.0	7.5
60	54.1	8.7
90	53.0	8.6
120	53.5	8.6

K/S value of mordanted sample = 2.9

Table 6: Exhaustion profile of Tea extract using Alum → Tannic acid as mordant

Dyeing Time (min)	Exhaustion %	K/S (after washing)
0	0	*1.9
10	53.0	6.3
15	56.1	7.5
20	57.8	8.1
30	59.9	9.0
60	68.3	10.7
90	68.0	11.0
120	67.7	10.5

K/S value of mordanted sample = 1.9

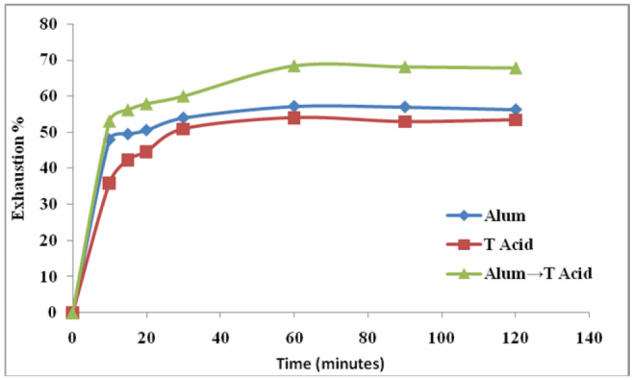

Fig.4. % Exhaustion Tea extract on bamboo fibres for different mordant systems

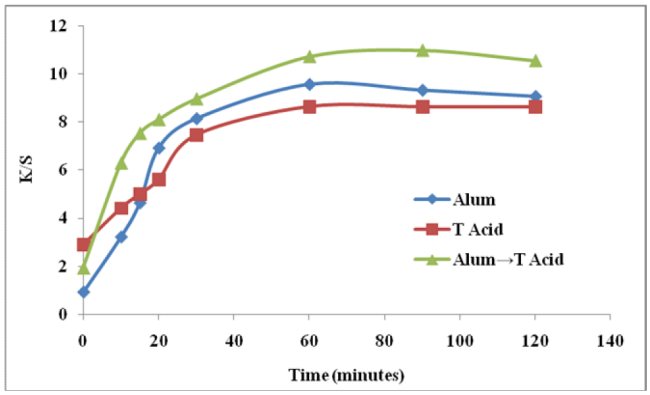

Fig.5. Comparison of K/S values of bamboo fibres dyed with Tea extract using different mordant systems

On further examination of properties like wash fastness, colour staining and light fastness, the samples dyed with Tea extract and Turmeric extract for 60 min were chosen as standard samples. Three different mordants used in the dyeing of bamboo fibres and their codes are given in Table 7. The wash and light fastness properties of the Turmeric & Tea extract on bamboo fibres were determined on fibres treated for time period of 60 min.

Table 8 shows fastness properties of various samples dyed with extract of Turmeric & Tea. The fastness properties are quite satisfactory for practical dyeing with many cases, particularly with the (Alum → Tannic acid) mordanted samples. Out of the two color fastness tests conducted, color fastness rating of the wash fastness test was most satisfactory, the color fastness ratings however were not very good.

Table 7: Various mordants used before dyeing of bamboo fibres with Turmeric & Tea

Sample Code	Dye used	Mordant used
1.	Turmeric	Alum
2.	Turmeric	Tannic acid
3.	Turmeric	Alum → Tannic acid
4.	Tea	Alum
5.	Tea	Tannic acid
6.	Tea	Alum→ Tannic acid

Table 8: Color fastness data[a] of the Turmeric & Tea extract on bamboo fibres

Sample[b]	Wash fastness	Color staining[c]						Light fastness
		SCA	BUC	N	P	A	WW	
1	4	4	4-5	4	5	4-5	4	4
2	3-4	4-5	4-5	4-5	4-5	4-5	4	3
3	3-4	4-5	4	4-5	5	4-5	4	4
4	4	4-5	4	4	4-5	4-5	4	4
5	3-4	5	4-5	4	5	4-5	5	3-4
6	3-4	4-5	4-5	4-5	5	4-5	4-5	4

[a]Grey scale ratings [b]see Table 1 for dyeing methods [c]Colour staining rating: SCA, secondary cellulose acetate; BUC, bleached unmercerised cotton; N, nylon 66; P, polyester; A, acrylic; WW, worsted wool

From above discussion, it is clear that the exhaustions were satisfactory for all the three mordant systems used viz. Alum, Tannic acid and Alum→ Tannic acid for dyeing of bamboo fibres with Turmeric and Tea as natural dyes. Among the three mordant systems, (Alum→ Tannic acid) system produces highest colour uptake on bamboo fibres. Among the two dyes used, Tea extract produced higher colour yield compared to Turmeric extract for the identical dyeing conditions. It was found that the exhaustion % increased in all the cases up to the time period of 60 min. From Figure 2, 3, 4 & 5, it is seen that the premordanting with alum followed by tannic acid has enhanced the dye uptake in comparison to pre-mordanting with alum and tannic acid individually.

V. Conclusion

1. In all the cases, exhaustion of dyes (Turmeric and Tea extracts) has been increased with increase in time. After 60 min, there was slight decrease in percent exhaustion. Therefore, ultimately, the premordanted samples dyed for 60 min were taken as standard for the measurement of other properties.
2. In case of Turmeric & Tea extracts, by using Tannic acid as mordant showed lower percent exhaustion than Alum. The best percent exhaustion has been found with Alum → Tannic acid (i.e., first mordanting with Alum, then drying and then treatment with Tannic acid) combination.
3. The maximum dye uptake in case of two steps mordanting (i.e. alum followed by tannic acid) was found to be 70.2% (in case of Turmeric extract) and 68.3% (in case of Tea extract).
4. In case of Alum & Alum→ Tannic acid mordanting of bamboo fibres with Turmeric & Tea extracts, by which a dye-metal complex was formed, characteristically imparted the enhanced fastness to light as well as washing and also the resulted in a dulling of the shade. However, improvement in the washing fastness of the dyeings with other mordant i.e. tannic acid was not evident.

After critically analyzing the results obtained in this study, it can be said that among the three types of mordants, Alum and Tannic acid combination gave best result as the percent exhaustion with acceptable fastness properties.

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[8] *CIELAB 1976* colour space (D_{65} illuminant, 10° observer).

AUTHOR'S PROFILE

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Prof. Chattopadhyay, the Head of the Department of Textile Chemistry, The M.S. University of Baroda started his career in teaching & research initially with TITS, Bhiwani, a premiere institute of Textile. Before joining teaching he served the textile industry in various positioned. He did his Ph.D under the supervision of Prof. R B Chavan of IIT, Delhi. He joined the department of textile chemistry of the M S University of Baroda in 2001. He has published about 80 papers to his credit in different journals of National and International repute including conferences and coordinated various government sponsored projects. He was awarded the best oral presentation award from faculty category in UGC sponsored national level conference in 2010.

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Prof. Satindar Kaur, the former Head of the Department of Department of Applied Chemical Sciences & Technology, Guru Nanak Dev University, Amritsar started her carrier in teaching & research initially with Guru Nanak Dev University, Amritsar. She has published about 50 papers in national and international journals. She has attended more than 57 national and international conferences in her carrier. She got second prize for oral presentation from Punjab Sciences Congress 2012.

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