Dyeing of jute and cotton fabrics using Jackfruit wood extract: Part I — Effects of mordanting and dyeing process variables on colour yield and colour fastness properties

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Conventionally bleached jute and cotton fabrics have been subjected to pre-mordanting with selective single and double mordants using myrobolan (harda) and other mordants (metallic salts) followed by dyeing with aqueous extract of jackfruit wood and then studied for their mechanical and dyeing properties. It is observed that the application of 10-20% myrobolan followed by 10-20% of $Al_2(SO_4)_3$ or $FeSO_4$ in sequence have been identified as two most prospective mordanting systems. The study on the effect of dyeing process variables on surface colour strength indicates that the 90 min dyeing time, $70-90^{\circ}C$ dyeing temperature, $11.0 \ p$ H, 1:30 material-to-liquor ratio, 20-30% mordants concentration, 30-40% dye concentration, and 15 gpl common salt are the optimum values with minor differences among the different fibre-mordant systems studied. Colour fastness to washing, rubbing and exposure to sunlight, in general, and dyeing-pH sensitivity, in particular, for selective fibre-mordants-dye systems have also been assessed and compared. Dyeing at pH 11.0 for both the double pre-mordanting systems offers overall good colour fastness properties.

Keywords: Cotton, Dyeing, Jackfruit wood, Jute, Mordanting, Myrobolan, Natural dyes IPC Code: D06P

1 Introduction

Worldwide growing consciousness for use of ecofriendly products in daily life has generated renewed interest of consumers towards use of textiles from natural fibres dyed with ecofriendly natural dyes. However, all the natural dyes are not essentially non-toxic, though most of them are proved to be nontoxic and ecofriendly.¹ Jute is a ligno-cellulosic and multicellular bast fibre while cotton is a unicellular and pure cellulosic seed hair fibre. Cotton is mainly used for apparels while jute is usually used for sacking and wrapping fabric, besides its additional uses as decorative and home textiles. Both cotton and jute are agro-renewable and biodegradable natural fibres. Easy availability, low price, high tensile strength, initial modulus, moisture regain, good sound and heat insulation, dimensional stability and good dye acceptability are the major advantages for jute.² availability, agro-renewability, biodegrad-Easy

ability, good moisture regain, good dye receptivity, high comfort and excellent fineness and softness are the major advantages of cotton fibre.^{3,4} Chemically jute contains cellulose, hemicellulose and lignin as major constituents, while cotton is purely cellulosic. Hence, the dye-receptivity, dye-affinity and dyeabsorption characters of any natural dye are different in jute and cotton for their varying chemical composition, chemical functionality pattern as well as due to their differences in gross and fine structure.

Besides ecofriendliness, natural dyes have many more technical advantages^{5,6}, including their uncommon and soothing shades. However, the common drawbacks of natural dyes are their nonreproducible and non-uniform shades, poor to moderate colour fastness and lack of scientific information on the chemistry of dyeing and standardised dyeing methods.^{5,6} Many reports are available on application of natural dyes on silk⁶⁻⁸, wool ^{9,10} and a few on cotton^{11,12}, while reports on jute are rather scanty and sporadic.¹³ In the present work, an attempt has been made to study the effects of

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selective single and double mordanting and dyeing process variables on mechanical and dyeing properties of jute and cotton fabrics, dyed with aqueous extract of natural dye obtained from jackfruit wood.

2 Materials and Methods

2.1 Materials

Conventionally H_2O_2 (3%) bleached plain weave, fine hessian jute fabric of decorative variety (176 tex warp, 164 tex weft, 67 ends/dm, 67 picks/dm, 225 g/m² fabric area density and 0.70 mm fabric thickness), obtained from M/s Champdany Industries, Rishra, Hooghly, was used for the study.

Conventionally desized, scoured and H_2O_2 (1%) bleached plain weave cotton fabric (15 tex warp, 19 tex weft, 220 ends/dm, 180 picks/dm, 120 g/m² fabric area density and 0.28 mm fabric thickness), obtained from National Textile Corporation Ware-house, West Bengal, was used for the study.

Laboratory reagents (LR) grade ferrous sulphate, aluminium sulphate, ethylenediaminetetraacetic acid (EDTA), aluminium potassium sulphate and stannous chloride; and commercial grade acetic acid, common salt, sodium carbonate, sodium hydroxide, and nonionic soap obtained from local suppliers were used. A natural mordant myrobolan (haritaki or harda, *Terminalia chebula*) powder was also used for the study.

Jackfruit wood or wood of Kanthal tree (*Artocarpus heterophyllus* Lam) was used to get yellow colour aqueous extract for dyeing of fabrics. Perkin⁶ first identified this colour component as morol (Fig. 1), a typical flavanol (hydroxy-flavone), also found in fustic.

Depending on the mordant used, the colour obtained on textiles from jackfruit wood extract may vary from yellow to golden brown. In such hydroxy-flavone colour component, it is the -CO-C(OH)-group at C₃ and C₄ positions in the pyrone ring which accounts for the ability of these compounds to form complex with metal salts. Also, two -OH groups at $-C_3$ and $-C_4$ positions of the benzene ring may account

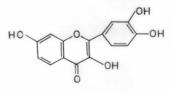


Fig. 1 — Structure of morol

for mordanting with metal salts. However, there is immense possibility of hydrogen bonding with cellulosic -OH groups, utilizing the said -OH groups of the colour component of jackfruit wood.

2.2 Methods

2.2.1 Extraction of Colour Component

For optimizing the extraction method, the aqueous extraction of dye liquor was carried out under varying conditions, such as time of extraction, temperature of extraction bath, *p*H of extraction liquor, concentration of colour-source material (jackfruit wood) and material-to-liquor ratio. In each case, the optical density or absorbance value at a particular maximum absorbance wavelength (λ_{628} nm) for the aqueous extract of the jackfruit wood was estimated using Hitachi-U-2000 UV-VIS absorbance spectrophotometer.

Various conditions used for the aqueous extraction of colour component from jackfruit wood and the respective absorbance values are given below:

Extraction variable	Absorbance of colour component at $\lambda_{628 \text{ nm}}$
Time, min	
5	2.50
10	2.62
15	2.71
30	2.77
60	2.68
120	2.60
Temperature, °C	
30	0.72
60	1.55
90	2.63
100	2.77
Colour source material	
(w/w), %	
10	2.69
20	2.74
40	2.67
80	2.61
Material-to-liquor ratio	
1:25	2.66
1:5	2.69
1:10	2.71
1:15	2.54
1:20	2.23
pH (Acidity/alkalinity)	
4.0	1.49
5.0	1.60
6.0	1.99
8.0	2.01
10.0	2.69
11.0	2.77
12.0	2.73

The bold values indicate the optimum conditions for the extraction of colour component from jackfruit wood. The material-to-liquor ratio (1:10) is finally reduced to 1:2.5 by further boiling and evaporation after the extraction. Unless otherwise mentioned, in usual cases the aqueous extract of dye liquor from jackfruit wood was prepared following the above optimized conditions of extraction.

2.2.2 First Mordanting of Jute and Cotton Fabrics with Myrobolan

The myrobolan (harda) powder was soaked in water (1:10 volume) for overnight (12h) at room temperature to obtain the swelled myrobolan gel. This gel was then mixed with a known volume of water and heated at 80°C for 30 min. The solution was then cooled and filtered in a 60 mesh nylon cloth and the filtrate was used as final mordant solution (10-40%) for mordanting, using MLR of 1:20. Pre-wetted conventional H₂O₂ bleached jute and cotton fabrics were separately treated with the harda solution in separate bath initially at 40-50°C and then the temperature was raised to 80°C. The mordanting was continued for 30 min. After the harda mordanting, fabric samples were dried in air without washing to make them ready for either subsequent dveing or for second mordanting.

2.2.3 First/Second Mordanting of Jute and Cotton Fabrics with Metallic Salts

Conventionally bleached (H_2O_2) jute and cotton fabrics with or without initial Ist mordanting were further mordanted prior to dyeing using 10-40% of any one of the chemical mordants, such as aluminium sulphate, aluminium potassium sulphate, ferrous sulphate, stannous chloride and EDTA, at 80°C for 30 min with material-to-liquor ratio of 1:20. After mordanting, the fabric samples were finally dried in air without washing to make them ready for subsequent dyeing.

2.2.4 Dyeing of Pre-mordanted Jute and Cotton Fabrics

Bleached and differently mordanted (single or double) jute and cotton fabrics were dyed using the aqueous colouring extract of jackfruit wood under specific and/ or varying condition of dyeing. For general study of dyeing behaviour using different mordants, a prefixed normal dyeing condition (aq. extract of jackfruit wood, 20%; mordant, 20%; MLR, 1:20; common salt, 10gpl; pH 11.0 with requisite amount of NaOH; dyeing temperature, 100°C and dyeing time, 60 min) was used.

For studying the effect of dyeing process variables on colour yield to optimize the dyeing conditions, the dyeing conditions were varied as follows: dyeing time 30-120 min, dyeing temperature 70-100°C, materialto-liquor ratio 1:10 - 1:50, concentration of mordants 10-40% (myrobolan) and 20-40% (other chemical mordants), concentration of jackfruit wood extract 10-40%, common salt concentration 5-20gpl and pH 4.5-12.0. The selective mordant systems used for the study of dyeing process variables are: (i) double pre-mordanting with 20% harda (Ist mordant) followed by 20 % aluminium sulphate (2nd mordant) applied in sequence on jute (A) and on cotton (A')double pre-mordanting with 20% harda (ii) (Ist mordant) followed by 20% ferrous sulphate (2nd mordant) applied in sequence on jute (B) and on cotton (B'), expect in the cases of study on variation in mordant concentrations (10-40%).

In each case, after the dyeing is over, the dyed samples were repeatedly washed with hot and cold water and then dried in air. Finally, the dyed samples were subjected to soaping with 2 gpl soap solution at 60°C for 15 min, followed by repeated water wash and drying under sun.

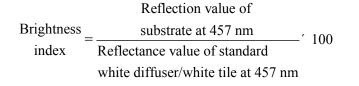
2.2.5 Determination of K/S Value and Brightness Index

The *K/S* value (surface colour strength) of the undyed and dyed jute and cotton fabrics was determined by measuring surface reflectance of the samples using a computer-aided Macbeth 2020 plus reflectance spectrophotometer, using the following Kubelka Munk equation¹⁴ with the help of relevant software:

$$K/S = \frac{(1 - R\lambda_{\max})^2}{2R\lambda_{\max}} = \alpha C_{\rm d}$$

where *K* is the coefficient of absorption; *S*, the coefficient of scattering; C_d , the concentration of the dye; and $R\lambda_{max}$, the surface reflectance value of the sample at a particular wavelength, where maximum absorption occurs for a particular dye/colour component.

Brightness index was calculated as per the standard (ISO-2470-1977) method¹⁵ using the following relationships after measuring the reflectance value of the corresponding sample by the same computeraided Macbeth 2020 plus reflectance spectrophotometer:



2.2.6 Measurement of Breaking Tenacity and Breaking Extension

Warp-way breaking tenacity (cN/tex) and breaking extension (%) values of the selective mordanted fabrics were comparatively measured after prior conditioning of the samples at $65 \pm 2\%$ RH and $27 \pm 2^{\circ}$ C temperature for 48 h as per the IS : 6359-1971 method¹⁶) according to IS:1969:1968 ravelled strip method¹⁶ on an Instron (Model 1445) CRT-Universal tensile tester with a traverse speed of 100 mm/min and a pretension of 0.5 N. The final gauze length (sample size) of the fabric sample maintained was 50 mm × 20 mm after raveling.

2.2.7 Measurement of Fabric Stiffness

Stiffness of the selective mordanted fabric, as expressed by the warp-way bending length, was measured as per IS: 6490-1971 (Cantilever Test) method¹⁶, using Sasmira fabric stiffness tester for the fabric samples of the size 200 mm \times 25 mm.

2.2.8 Evaluation of Colour Fastness

Colour fastness to washing¹⁶ of the dyed fabric samples was determined as per IS: 764-1984 method using a Sasmira launder-O-meter following IS-3 wash fastness method. The wash fastness rating was assessed using grey scale as per ISO -05-AO2 (loss of shade depth) and ISO -105-AO3 (extent of staining) and the same was cross-checked by measuring the loss of depth of colour and staining using Macbeth 2020 plus computer-aided colour measurement system attached with relevant software.

Colour fastness to rubbing (dry and wet) ¹⁶ was assessed as per IS: 766-1984 method using a manually operated crockmeter and grey scale as per ISO-105-AO3 (extent of staining).

Colour fastness to exposure to light¹⁶ was determined as per IS: 2454-1984 method. The half of the samples (10cm \times 1cm) was exposed to UV light in a Shirley MBTF Microsal fade-O-meter (having 500 watt Philips mercury bulb tungsten filament lamp simulating day light) along with the eight blue wool standards (BS 1006: BOI: 1978). The fading of each sample was observed against the fading of blue wool standards (1–8).

3 Results and Discussion

3.1 Effect of Single and Double Mordanting on Jute and Cotton Fabrics

The bleached jute and cotton fabrics have been mordanted using different single and double mordants and the resultant changes in mechanical properties (breaking tenacity, breaking extension and bending length), surface colour strength (K/S value) and total colour differences (dE) of the mordanted fabrics have been assessed and shown in Table 1 for single mordanting and Table 2 for sequential double mordanting. It is observed that when bleached jute and cotton fabrics are mordanted with stannous chloride alone or double mordanted with harda + stannous chloride, there is a considerable loss in fabric strength. Bleached jute and cotton fabrics when mordanted with aluminium sulphate or ferrous sulphate either alone or double mordanted with harda + aluminium sulphate/ferrous sulphate, they show much lower loss in fabric strength as compared to that observed with stannous chloride alone and in combination with harda. In general, on treatment with all the selective mordants, there is some loss in warpway fabric tenacity along with some increase in warpway breaking elongation of bleached jute and cotton fabrics, irrespective of the mordants concentrations applied. However, with the increase in amount of myrobolan or all other chemical mordants used for single and double mordanting, the strength loss increases noticeably. The strength loss is found to be minimum for EDTA on both jute and cotton fabrics, irrespective of its percentage of application and type of mordanting (single or double). The degree of loss in fabric strength and associated low increase in breaking extension owing to mordanting treatment may be viewed as an effect of limited acidic degradation and disorientation caused in the polymeric chains of the fibre substrate by the tannic acid component of myrobolan and acid generated in aqueous solution of most of the other chemical mordants used, except EDTA. For mordanting with $FeSO_4$ and $Al_2(SO_4)_3$ alone, the bending length value is found to increase and for mordanting with EDTA alone, it is found to decrease to a small extent: however, it remains almost unaltered in all other cases including the cases of double mordanting.

It is interesting to note that harda, being a natural light yellow mordantable dye, shows a steady increase in K/S value as well gradual increase in colour difference values with the increase in percentage of

Mordant conc. %		y tenacity / tex	Warp-way breaking elongation, %			y bending h, cm	Selective colour strength and colour difference				
	Jute	Cotton	Jute	Cotton	Jute	Cotton	Jute		Cotto	n	
							K/S value at λ_{max}	dE	K/S value at λ_{max}	dE	
Nil (Control	6.09	5.41	3.47	5.36	4.2	2.1	0.80		0.01		
bleached fabric)											
Myrobolan											
5	5.56	5.21	5.60	5.25	4.1	2.1	1.40	0.50	0.07	7.10	
10	4.89	5.03	5.73	5.19	4.2	2.1	1.57	0.54	0.15	8.27	
15	3.71	4.48	5.88	5.14	4.3	2.2	1.74	0.64	0.25	9.36	
20	3.50	4.78	5.97	5.08	4.4	2.2	1.96	0.73	0.36	10.49	
$Al_2(SO_4)_3$											
5	5.96	4.89	4.27	1.51	4.3	2.1	0.96	0.74	0.01	1.05	
10	5.85	4.67	4.31	1.63	4.4	2.1	1.05	0.87	0.03	1.27	
15	5.27	4.51	4.45	1.89	4.5	2.1	1.10	0.99	0.04	1.41	
20	4.88	4.26	4.47	1.97	4.6	2.2	1.20	1.10	0.07	1.50	
30	4.37	4.01	4.51	2.09	4.6	2.2	1.33	1.89	0.08	1.78	
40	4.07	3.97	4.61	2.19	4.7	2.3	1.34	1.94	0.09	2.01	
FeSO ₄											
5	5.77	5.01	4.32	3.51	4.4	2.1	3.79	4.21	0.03	19.54	
10	5.06	4.99	4.64	3.69	4.4	2.3	4.63	4.87	0.04	20.05	
15	4.89	4.80	4.72	3.99	4.5	2.3	4.79	5.19	0.05	20.87	
20	4.45	4.72	4.84	4.09	4.5	2.4	5.03	5.97	0.06	21.02	
30	4.12	4.63	4.95	4.47	4.7	2.4	6.12	6.21	0.08	22.01	
40	3.99	4.54	5.01	4.97	4.8	2.5	6.54	6.84	0.09	23.33	
$KAl(S0_4)_2$	5.77	1.0 1	0.01	1.57	1.0	2.0	0.51	0.01	0.09	20.00	
5	3.47	4.01	4.79	3.98	4.0	2.1	0.95	1.01	0.02	5.31	
10	3.30	4.12	4.91	4.12	4.0	2.2	0.99	1.37	0.02	5.51	
15	3.12	4.27	5.01	4.23	4.0	2.2	1.10	1.64	0.02	8.61	
20	3.00	4.34	5.11	4.57	4.1	2.5	1.15	1.77	0.03	8.88	
SnCl ₂	5.00	т.5т	5.11	4.57	7.1	2.5	1.15	1.//	0.04	0.00	
5	3.54	4.89	4.54	3.20	4.2	2.1	0.97	1.79	0.01	3.67	
10	3.32	4.27	4.71	3.89	4.2	2.1	1.01	1.89	0.01	3.78	
15	3.11	3.84	4.87	4.65	4.2	2.3	1.01	2.14	0.01	5.54	
20	2.98	3.84	5.09	4.98	4.3	2.5	1.12	2.14	0.02	5.93	
EDTA	2.98	5.21	5.09	4.90	4.5	2.5	1.12	2.29	0.02	5.95	
5	4.51	4.03	4.01	4.02	3.8	2.1	0.89	1.01	0.02	4.51	
10	4.31	4.05	4.01	4.02	3.8 3.8	2.1	0.89	1.01	0.02	4.31 5.41	
10 15	4.32	4.35 4.71	4.15	4.34 4.65	3.8 3.8	2.1 2.2	0.89	1.07	0.03	5.41 6.47	
13 20	4.12 4.04	4.71	4.32 4.57		3.8 4.0	2.2	0.90		0.04 0.04		
20	4.04	4.98	4.37	4.70	4.0	2.2	0.91	1.19	0.04	7.43	

Table 1 — Tensile properties, bending length, surface colour strength and colour differences of jute and cotton fabrics treated with selective single mordants (without dyeing)

harda on both bleached jute and cotton fabrics. In between jute and cotton, the increase in colour difference is much higher for cotton than that for jute.

Among all the mordants used, the increase in K/S value and colour differences is found to be the highest for FeSO₄ mordant due to the inherent colour of FeSO₄ salt. This is more predominant on cotton than on jute and shows an increasing trend with the increase in percentage of FeSO₄ on both the fabrics. The increase in colour difference values after single mordanting (without dyeing) with selective mordants on both jute and cotton fabrics is in the following order:

For cotton : $FeSO_4 \gg Harda > KAl(SO_4)_2 > EDTA >$ $SnCl_2 > Al_2(SO_4)_3$ For jute : $FeSO_4 \gg SnCl_2 > EDTA > KAl(SO_4)_2 >$

 $Al_2(SO_4)_3 > Harda$

However, in case of double mordanting (without dyeing) with selected double mordanting systems, the increase in colour difference values is in the following order:

For cotton: Harda+FeSO₄ >> Harda+Al₂(SO₄)₃> Harda+KAl(SO₄)₂>Harda+SnCl₂> Harda+EDTA

Mordant conc. %		y tenacity / tex	Warp-way breaking elongation, %			ay bending th, cm	Surface colour strength and colour difference				
	Jute	Cotton	Jute	Cotton	Jute	Cotton	Jute		Cotto	n	
							$\frac{K/S}{\text{at }\lambda_{\text{max}}}$	dE	K/S value at λ_{max}	dE	
Nil (Control bleached fabric) Myrobolan: Al ₂ (SO ₄	6.09	5.41	3.47	5.36	4.2	2.1	0.80	_	0.01	—	
5:5	5.02	5.15	5.57	5.76	4.1	2.2	1.61	2.54	0.13	9.87	
10:10	4.62	4.79	5.57	6.56	4.1	2.2	2.23	2.87	0.13	10.04	
15:15	4.31	4.05	5.56	7.89	4.1	2.2	2.23	2.99	0.27	11.27	
20:20	3.94	3.40	5.58	8.57	4.1	2.4	2.64	3.55	0.57	11.55	
30:30	3.16	3.27	5.67	8.60	4.2	2.4	2.88	3.87	0.60	11.97	
40:40	2.73	3.15	5.80	8.87	4.3	2.5	2.87	4.27	0.78	12.07	
Myrobolan : FeSO ₄	2.75	5.10	0.00	0.07		2.0	2.07	,	0.70	12:07	
5:5	4.01	5.18	5.09	5.18	4.2	2.2	4.63	5.97	1.05	12.97	
10:10	3.75	4.99	5.18	5.54	4.2	2.2	4.90	6.87	1.45	13.04	
15:15	3.29	4.13	5.69	5.65	4.6	2.4	8.86	7.21	1.58	13.29	
20:20	2.70	4.08	4.10	5.98	4.5	2.6	8.67	8.47	1.72	14.28	
30:30	2.36	3.98	4.55	6.51	4.5	2.6	8.94	8.97	2.20	15.47	
40:40	1.79	3.74	4.80	6.91	4.6	2.7	8.97	9.54	2.47	16.97	
Myrobolan : KAl(S0	$()_4)_2$										
5:5	3.59	4.50	5.39	4.31	4.1	2.3	0.99	3.27	0.45	6.54	
10:10	3.30	4.26	5.50	4.55	4.3	2.4	1.70	3.48	0.50	8.69	
15:15	3.84	4.06	5.64	4.75	4.3	2.6	2.24	3.89	0.56	9.87	
20:20	2.71	3.89	5.79	4.92	4.5	2.8	2.60	4.34	0.64	10.61	
Myrobolan : SnCl ₂											
5:5	3.45	4.87	5.40	5.29	4.3	2.2	1.47	3.27	0.02	6.89	
10:10	3.20	4.08	4.01	5.04	4.4	2.4	1.54	3.43	0.03	7.01	
15:15	3.04	3.32	4.39	4.86	4.6	2.6	1.89	3.54	0.04	7.21	
20:20	2.79	3.02	4.69	4.48	4.8	2.8	1.98	3.63	0.06	7.39	
Myrobolan : EDTA											
5:5	5.27	4.71	5.61	6.98	4.2	2.1	0.94	2.01	0.02	5.77	
10:10	4.80	4.44	5.97	7.65	4.2	2.2	1.10	2.09	0.03	5.87	
15:15	4.52	4.21	6.21	8.54	4.3	2.3	1.15	2.21	0.04	6.01	
20:20	4.30	4.03	6.41	9.77	4.3	2.3	1.50	2.34	0.05	6.11	

Table 2 — Tensile properties, bending length, colour strength and colour differences of jute and cotton fabrics treated with selective double mordants (without dyeing)

For jute: Harda+FeSO₄>>Harda+KAl(SO₄)₂> Harda+SnCl₂>>Harda+Al₂(SO₄)₃> Harda+EDTA

This increase in K/S value and colour difference value to a different extent after selective mordanting may be due to the changes in scattering because of the chemical interactions between fibres and harda or metallic salts along with the additional inherent colour input of the corresponding mordants. In case of double mordanting, due to the complimentary or opposite colour interactions, the dE values reduce as in the case of harda + FeSO₄ double mordanting on bluish-white cotton. However, the dE value increases as in case of harda + FeSO₄ double mordanting on creamish-yellow jute due to the supportive and additive colour interaction.

Bleached jute and cotton fabrics differently mordanted with varying concentration of mordants have been subsequently dyed with varying concentration of extracted dye liquor (JFW), following a prefixed normal dyeing conditions as reported in section 2.2.4. All the dyed fabrics have been assessed for their surface colour strength (K/S value), brightness index, total colour difference (dE)and colour fastness behaviour to washing, rubbing and exposure to light and the results are given in Table 3 for single and Table 4 for double mordanted jute and cotton fabrics. It is observed that among differently mordanted bleached jute subsequently dyed with 20% aqueous extract of jackfruit wood, the sequential double mordanting with 20% myrobolan and 20% ferrous sulphate renders the fabric relatively higher K/S value (~ -12.21) as compared to other

% % KS value at λ_{max} dE Nil (Control bleached fabric) Jute 0 — 0.80 — 38.20 Nil (Control fabric) Cotton 0 — 0.01 — 92.06 Myrobolan	Mordant conc.	Fabric	Dye conc.	Shade obtained	Surface colour strength and c	olour difference	BI
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$			2		¥		
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$		Jute	0	—	0.80	—	38.20
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	Nil (Control fabric)	Cotton	0		0.01	—	92.06
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	Myrobolan						
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	20	Jute			1.96	0.73	25.72
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	20	Jute	20	Creamish yellow	2.30	1.36	21.06
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$				_			
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	20	Cotton	20	Creamish yellow	0.55	6.23	45.13
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	$Al_2(SO_4)_3$						
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	20	Jute			0.99		
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	20	Jute	20	Light green			
$\begin{array}{cccccccccccccccccccccccccccccccccccc$		Cotton		—	0.07		
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	20	Cotton	20	Creamish yellow	0.16	6.58	68.28
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	FeSO ₄						
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$				—			13.46
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$		Jute		Golden brown			
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	20	Cotton		—			
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	20	Cotton	20	Golden yellow	1.09	7.97	32.72
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	$KAl(S0_4)_2$						
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	20	Jute	0	—	1.15		
20 Cotton 20 Creamish yellow 0.10 9.47 68.09 SnCl ₂ 20 Jute 0 — 1.12 2.29 35.59 20 Jute 20 Light yellow 1.61 5.95 28.15 20 Cotton 0 — 0.02 5.93 75.93 20 Cotton 20 Cream 0.14 6.68 65.26 EDTA 20 Jute 0 — 0.80 1.19 37.56 20 Jute 20 Cream 1.03 1.68 33.40	20	Jute	20	Light green	1.48		
SnCl2 Jute 0 — 1.12 2.29 35.59 20 Jute 20 Light yellow 1.61 5.95 28.15 20 Cotton 0 — 0.02 5.93 75.93 20 Cotton 20 Cream 0.14 6.68 65.26 EDTA				—		8.88	
20 Jute 0 — 1.12 2.29 35.59 20 Jute 20 Light yellow 1.61 5.95 28.15 20 Cotton 0 — 0.02 5.93 75.93 20 Cotton 20 Cream 0.14 6.68 65.26 EDTA 20 Jute 0 — 0.80 1.19 37.56 20 Jute 20 Cream 1.03 1.68 33.40	20	Cotton	20	Creamish yellow	0.10	9.47	68.09
20 Jute 20 Light yellow 1.61 5.95 28.15 20 Cotton 0 — 0.02 5.93 75.93 20 Cotton 20 Cream 0.14 6.68 65.26 EDTA 20 Jute 0 — 0.80 1.19 37.56 20 Jute 20 Cream 1.03 1.68 33.40							
20 Cotton 0 — 0.02 5.93 75.93 20 Cotton 20 Cream 0.14 6.68 65.26 EDTA	20	Jute		—	1.12	2.29	
20 Cotton 20 Cream 0.14 6.68 65.26 EDTA 20 Jute 0 — 0.80 1.19 37.56 20 Jute 20 Cream 1.03 1.68 33.40	20	Jute	20	Light yellow	1.61		
EDTA 20 Jute 0 — 0.80 1.19 37.56 20 Jute 20 Cream 1.03 1.68 33.40		Cotton		—			
20Jute0-0.801.1937.5620Jute20Cream1.031.6833.40	20	Cotton	20	Cream	0.14	6.68	65.26
20 Jute 20 Cream 1.03 1.68 33.40	EDTA						
		Jute		—	0.80		
				Cream			
	20	Cotton	0	—	0.04	7.43	87.43
20Cotton20Cream0.078.1079.10	20	Cotton	20	Cream	0.07	8.10	79.10

_	
	Table 3 — Brightness index (BI), surface colour strength, colour differences and colour fastness of dyed jute and cotton fabrics after
	single mordanting with selective mordants

mordanting system at a comparable dose level. The use of 20% harda and 20% aluminium sulphate mordanting technique followed by further dyeing with comparable dose of jackfruit wood colour (20% JFW) shows the K/S value of 3.35 (Table 4) and thus is considered as next good performer. In all the cases of dyeing with different percentages of aqueous extract of jackfruit wood using different mordants, the brightness index values are found to be reduced to a noticeable extent from the corresponding value obtained after respective mordanting. When the jute fabric is double mordanted with myrobolan and ferrous sulphate in sequence, the brightness index decreases with the increase in K/S value. Hence,

considering the dyeing results, the sequential mordanting systems using 20% myrobolan + 20% aluminium sulphate and 20% myrobolan + 20% ferrous sulphate are found to be more prospective, rendering a higher degree of increase in surface colour strength. These two systems of mordanting have therefore been chosen for further study of dyeing process variables for both jute and cotton fabrics. However, the use of ferrous sulphate in any case always renders both jute and cotton fabrics a deep brownish/grey colour owing to the inherent colour of this transition metal salt anchored to the corresponding fibres, besides the improvement in *K/S* value due to the natural dye component.

Mordant conc.	Fabric	Dye conc.	Shade	Surface colour	strength	BI		Colo	our fastnes	s to	
%		%	obtained	and colour dif			Was	hing	Light	Rub	bing
				K/S value at λ_{max}	dE		LOD	ST		Dry	Wet
Myrobolan : A	$l_2(SO_4)_3$										
20:20	Jute	0	_	2.54	3.55	23.56	_	_	_		
20:20	Jute	20	Greenish yellow	3.35	5.65	19.77	4	5	3	5	5
20:20	Cotton	0	_	0.08	11.5	49.71					
20:20	Cotton	20	Light greenish yellow	0.55	7.45	49.66	5	4-5	3	5	4-5
Myrobolan :]	FeSO₄										
20:20	Jute	0	_	8.67	8.41	5.38	_		_		
20:20	Jute	20	Blackish brown	12.21	35.2	3.79	3	5	3	4-5	3-4
20:20	Cotton	0	_	2.29	14.28	17.11			_		
20:20	Cotton	20	Grey	3.99	5.11	12.51	1	3-4	1	4-5	3-5
Myrobolan : K.	$A1(S0_4)_2$										
20:20	Jute	0		2.60	4.34	24.17					
20:20	Jute	20	Greenish brown	3.37	5.76	19.94	4	5	3	5	5
20:20	Cotton	0	_	0.10	10.6	48.70	_		_		
20:20	Cotton	20	Light yellow	1.05	7.72	36.20	5	4-5	3	5	4-5
Myrobolan :	SnCl ₂										
20:20	Jute	0	_	2.51	3.63	25.57					
20:20	Jute	20	Light ocher yellow	3.13	5.93	22.29	5	4-5	1	4-5	4-5
20:20	Cotton	0		0.06	7.39	58.88					
20:20	Cotton	20	Light yellow	0.55	10.3	50.62	5	4-5	3	5	4-5
Myrobolan :]	EDTA										
20:20	Jute	0	_	1.50	2.34	27.71			_		
20:20	Jute	20	Cream	1.36	0.33	29.11	4	5	2	5	5
20:20	Cotton	0	_	0.05	6.11	65.65					
20:20	Cotton	20	Cream	0.18	8.47	62.61	5	4-5	3	5	4-5

Table 4 — Brightness index, surface colour strength, colour differences and colour fastness of dyed jute and cotton fabrics after sequential double mordanting with selective mordants

dE-Colour difference, BI-Brightness index, LOD- Loss of depth of shade and ST- Staining on cotton.

3.2 Effect of Dyeing Process Variables for Optimizing the Dyeing Conditions

Figure 2 shows the effect of dyeing process variables, such as dyeing time, dyeing temperature, material-to-liquor ratio, pH (concentration of acid/alkali added), mordants concentration, dye concentration and common salt concentration respectively, on the surface colour strength (*K/S* values) of bleached and dyed jute and cotton fabrics after mordanting. The selective fibre-mordanting systems used for the study of dyeing process variables are shown in section **2.2.4**.

Figure 2a shows that keeping all other variables prefixed and unaltered, with the increase in time of dyeing from 15 min to 120 min, the K/S value slowly

increases upto 90 min and then either levels off or slowly decreases for all the combinations of fibremordanting systems, such as (A), (B), (A') and (B'). Among these mordanting systems, the increase in surface colour strength is much higher for fibremordanting system (B). For other fibre-mordanting systems (A), (A'), and (B'), the effect of increase in dyeing time is much subdued; the optimum dyeing time is found to be 90 min. Rate of dyeing for fibremordanting system (B) is, however, found to be sharp for 60-90 min period of dyeing. This higher rate of dyeing above 60 min and up to 90 min in case of fibre-mordanting system (B) may be due to the possible reduction in activation energy required for absorption and fixation of dyes on fibre surface by

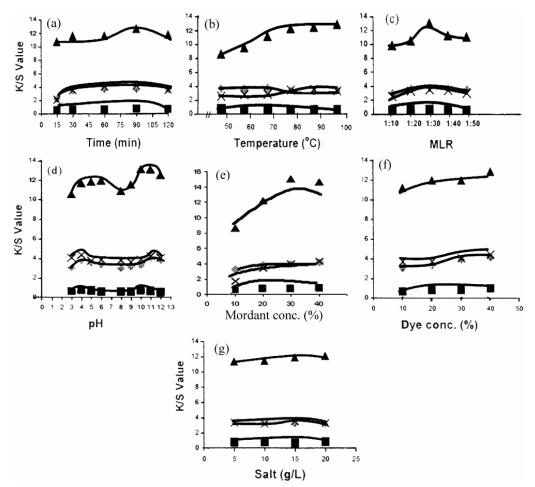


Fig. 2 — Effects of dyeing process variables on colour strength of jute and cotton fabrics dyed with jackfruit wood extract after double pre-mordanting with selective mordants [\blacklozenge Jute-Harda + Al₂(SO₄)₃(**A**), \blacktriangle Jute-Harda + FeSO₄(**B**), \blacksquare Cotton-Harda+Al₂(SO₄)₃(**A**'), and × Cotton-Harda+FeSO₄(**B**']

jute-hemicellu-COO⁻...Fe-ion complex formation, which is not possible in cotton.

Keeping other dyeing variables constant, with the increase in dyeing temperature from 50°C to 100°C (Fig.2b), the surface colour strength increases measurably up to 80°C for double fibre-mordanting systems (B) and (B') and then almost levels off. However, the rate of dyeing and the rate of increase in K/S value are found to be much higher for fibremordanting system (B) than for fibre-mordanting system (B'). In case of fibre-mordanting system (A) and (A'), the K/S value increases slowly at the temperature range 50°-70°C and then drops down. The increase in time or temperature of dyeing inevitably supplies more energy, usually facilitating higher rate of dye sorption for all the fibremordanting systems, in general, up to a certain limit of dyeing temperature, before desorption starts. However, in case of application of ferrous sulphate as

2nd mordant on jute substrate, the effect is much predominant and higher than those obtained by other mordanting systems applied on both jute and cotton substrates due to the possible jute-hemicellu -COO⁻....Fe-ion complexing in case of fibremordanting system (B).

For the variation in material-to-liquor ratio (MLR) from 1:10 to 1:50 (Fig. 2c), initially the *K/S* value sharply increases upto MLR of 1:30 and then gradually drops down with the further increase in MLR for both the fibre-mordanting systems (B) and (B'); while the effect is much subdued and marginal in cases of fibre-mordanting systems (A) and (A'). However, for all these fibre-mordants systems, the optimum MLR is found to be 1:30.

Figure 2d interestingly shows varying degree of crests and troughs in the trends of K/S value with the variation in pH values between 3 and 12. However, for fibre-mordanting system (B) on jute sample,

Pre-mordanting	pН					Colour	fastness to				
treatment of dye		Washing				Li	ight		Rub	bing	
	liquor	Ju	Jute Cotton		ton	Jute	Cotton	Ju	ıte	Co	tton
		LOD	ST	LOD	ST			Dry	Wet	Dry	Wet
20% myrobolan	3.0	5	4	4	5	2	3	3-4	3	4	3-4
+ 20% Al ₂ (SO ₄) ₃	4.0	5	4	4	5	3	3	3-4	3	4	3-4
	5.0	4	4-5	4	5	3	3	3-4	3	4	3-4
	6.0	4	4-5	5	5	3	3	3-4	3	4	3-4
	8.0	4	4-5	5	5	4	4	4	3-4	4-5	4
	9.0	5	4	4	5	4	4	4	3-4	4-5	4
	10.0	4	5	4	5	4	4	4	3-4	4-5	4
	11.0	4	4-5	4	4-5	4	4	4	3-4	4-5	4
	12.0	4	4-5	4	5	4	5	4	3-4	4-5	4
20% myrobolan	3.0	3	4-5	1	4	1	1	4	3-4	3-4	3
+ 20 % FeSO ₄	4.0	3	4-5	1	4-5	1	1	4	3-4	3-4	3
	5.0	3	5	1	4-5	1	1	4	3-4	3-4	3
	6.0	3	4-5	1	4-5	1	1	4	3-4	3-4	3
	8.0	3	4-5	1	4-5	2	2	4	3-4	4	3-4
	9.0	4	4-5	1	4-5	2	2	4	3-4	4	3-4
	10.0	4	5	1	4-5	2	2	4	3-4	4	3-4
	11.0	4	4-5	1	4	2	2	4	3-4	4	3-4
	12.0	4	4-5	1	4	2	2	4	3-4	4	3-4

Table 5 — Effect of pH of the dye liquor on colour fastness properties of jute and cotton fabrics

relatively two broader crests and one trough are observed, showing higher K/S value at pH 5.0 and 11.0; among the two pH values, the highest K/S value being obtained at pH 11.0. For other fibre-mordanting systems (A), (A'), and (B'), the K/S values are found to be much lower than that obtained for fibre-mordanting system (B). But all these three fibre-mordanting systems, except (B), show corresponding maximum K/S values either at pH 4.0 or at pH 11.0 which are also much closer. However, considering the surface colour strength as well as colour fastness behaviour (Table 5) together, pH 11.0 may be taken as optimum for all the fibre-mordanting systems studied, as the colour fastness properties are found to be relatively better at *p*H 11.0 than that at *p*H 4.0-5.0.

Figure 2e shows a slow increase in K/S value with the increase in mordant concentration upto 20% before showing the leveling off trend for fibremordanting systems (A) and (A'), while there is an increase in K/S value with the increase in mordant concentration up to 30% for fibre-mordanting system (B') before leveling off. The increase in K/S value is much sharper and predominant in case of fibremordanting system (B) up to 30% mordant concentration, above which it marginally drops down. Thus, for all the fibre-mordanting systems studied, the optimum mordant concentration may be considered to be around 20-30%. However, there is noticeable loss in tensile strength after mordanting bleached jute and cotton fabrics with more than 20% mordants. Hence, the use of more than 20% mordant concentration for mordant systems (A), (A') and (B') is not recommended. But despite some strength loss, the use of 30% mordant concentration may be allowed for obtaining much higher K/S value for fibre-mordanting system (B), as the increase in K/S value for this double mordanted jute fabrics is found to be exceptionally higher. So, for fibre-mordanting system (B), the optimum concentration of mordant should be within 20-30%.

Figure 2f shows slow increase in K/S values with the increase in concentration of extracted dye liquor up to 30% (on the basis of % solid jackfruit wood), above which it almost levels off reaching almost the saturation level. However, the K/S value obtained is always found to be much higher for fibre-mordanting system (B) than that for other fibre-mordanting systems (A), (A') and (B'). The higher dye uptake in case of fibre-mordanting system (B) indicates and confirms that there is more attraction / substantivity of colour component of jackfruit wood towards such mordanted jute, causing jute-hemicellu-COO⁻....Feion complex formation.

Figure 2g indicates that the optimum concentration of common salt for all the four fibre-mordanting systems is 15gpl, as the K/S values are found to be maximum at this concentration. However, the observed much lower K/S value in the cases of fibremordanting systems (A) and (A') on both bleached jute and cotton substrates is due to the suppression of the action of harda by blocking its functional coordinating groups by aluminium metal ion $[Al_2(SO_4)_3]$ to a certain degree. The observed highest K/S value in case of fibre-mordanting system (B) on bleached jute substrate, indicating the synergistic intensification of colour yield, is assumed to be due to higher absorption and fixation of the dye by the complex formed between the Fe-salts and the -COO⁻ group of jute hemicellulose, which is not possible in cotton due to the absence of -COO⁻ group. The observed slow increase in K/S value in cotton treated with same mordants is only due to the additive colour yield for the additional incorporation of the inherent colour of FeSO₄ itself. The addition of an electrolyte (common salt) to the dyeing liquor in the dyebath in case of dyeing with a mordantable anionic dye like jackfruit wood extract expectedly increases the exhaustion of the dye on the cellulosic or lignocellulosic fibres. They dissociate completely in the aqueous dye liquor at different temperatures of dyeing and increase the force of repulsion between the dye molecules and the water so that the dye molecules are more attracted to the cellulosic or ligno-cellulosic fibres. But higher amount of salt/electrolyte above a certain limit causes retardation effect in the dye adsorption.

4 Conclusions

4.1 Among the different fibre-mordanting systems studied, the use of 10-20% (owf) of both myrobolan and ferrous sulphate applied in sequence as double mordant for subsequent dyeing with jackfruit wood extract (30%) and 10-20% (owf) of both myrobolan and $Al_2(S0_4)_3$ applied in sequence as double mordant for subsequent dyeing with 40% of jackfruit wood extract show maximum *K/S* values as compared to other selective single and double mordanting systems.

4.2 The optimized conditions of dyeing of both bleached jute and cotton substrates are: 90 min dyeing time for all the mordanting systems; 70°C dyeing temperature for fibre-mordant systems (A), (B) and

(B') and 90°C for fibre-mordant (B); 1:30 MLR, for all the fibre-mordant systems; 11.0 pH for all the fibre-mordant systems; 20% (owf) mordant concentration for the fibre-mordant systems (A), (A') and (B'), and 20-30% for fibre-mordant system (B); 30-40% dye (JFW) concentration for all the fibre-mordant systems; and 15 gpl common salt for all the fibre-mordant systems.

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