Research Paper :

Dyeing of cotton and silk fabrics using stem of *Achras sapota* extract: Effects of mordanting and dyeing process variables on colour yield and colour fastness properties

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ABSTRACT

Eco-friendliness is one of the priority concerned in the textile industry where natural dyes can be used instead of synthetic dyes. The present study deals with an attempt which has been made to obtain dye extract from the stem of *Achras sapota*. Conventionally bleached cotton and silk fabrics have been subjected to pre, post and simultaneous mordanting with selective mordants using myrobolan (harda) and other mordants (metallic salts) followed by dyeing with ethanol extract of stem of *Achras sapota*. It is observed that the application of 3 per cent of $Al_2(SO_4)_3$ and 3 per cent of $FeSO_4$ have been identified as two better prospective mordanting system. The study on the effect of dyeing process variables on surface colour strength indicates that the 60 min. dyeing time, 60°C dyeing temperature, 1:20 material-to-liquor ratio, 3 per cent mordant concentration, 5 per cent dye concentration and 5g l^{-1} common salt are the optimum values with minor differences among the different fibre- mordant system studied. This study also includes the comparison of mordanting techniques as well as dying properties and visualizes the effect of myrobolan and metallic mordants. Colour fastnesses to washing, rubbing, light fastness and perspiration of cotton and silk fabrics dyed with and without mordants have also been studied.

KEY WORDS : Achras sapota, Cotton, Light fastness, Mordant, Myrobolan, Perspiration, Rubbing, Silk

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Recently, interest in the use of natural dyes has been growing rapidly due to the result of stringent environmental standards imposed by many countries in response to toxic and allergic reactions associated with synthetic dyes[1]. Until about 150 years ago all dyes were natural substances, derived mainly from plants and animals. The natural dyes present in plants and animals are pigmentary molecules[2] which impart colour to the materials. With the world becoming more conscious towards ecology and environment, there is greater need today to revive the tradition of natural dye and dyeing techniques as an alternative of hazardous synthetic dyes is an extremely crude.

There are several plants/plant parts that provide natural dyes which are used in the textile industry. However, the common drawbacks of natural dyes are their non-reproducible and non-uniform shades, poor to moderate colour fastness and lack of scientific information on the chemistry of dyeing and standardised dyeing methods [3, 4]. Many reports are available on application of natural dyes on silk[5,6] and cotton[7,8].The present investigation deals with the extraction of natural dyes from the stem extract of the plant *Achras sapota* that grows in all warm and damp parts of India and is considered to be one of the most useful trees in the world.

The aim of present work is to prepare eco-friendly natural dyes from the stem of *Achras sapota* for dying of cotton and silk fabrics. The stem extract of the plant *Achras sapota* produces a brown solution. In the present work an attempt has been made to study the effect of mordanting and dyeing properties[9] of cotton and silk fabrics such as, washing, rubbing, light fastness and perspiration[10] and also to visualize the effect of myrobolan and metallic mordants which have been undertaken.

EXPERIMENTAL METHODOLOGY

Conventionally designed, scoured and H_2O_2(1%) bleached plain weave cotton fabric (220 ends/ dm,180 picks/dm,120 g/m^2) and loom state silk (430 ends/ dm,212 picks/dm,50g/m^2 fabrics obtained from Gandhigram Rural University,Dindugal, were used for the study.

Analytical reagents (AR) grade ferrous sulphate, aluminium sulphate, nickel sulphate, potassium dichromate, stannous chloride, commercial grade acetic acid, common salt, sodium carbonate were used. A natural mordant myrobolan (*Terminalia chebula*) powder was used for the study.

The stem of *Achras sapota* was used to get brown colour ethanol extract for dyeing of fabrics. Depending upon the mordant used, the colour obtained on fabrics from the stem of *Achras sapota* extract may give different shades.

The myrobolan (harda) powder was soaked in water (1:10 volume) for overnight (12h) at room temperature to obtain the swelled myrobolan gel. It was then mixed with a known volume of water and heated at 80^{0}C for 30 min.The resulting solution was cooled and filtered. The filtrate was used as final mordant solution for mordanting[11].

Extraction of colour component:

For optimizing[12] the extraction method the ethanol extraction of dye liquor was carried out under varying conditions, such as time of extraction, temperature of extraction bath and material-to-liquor ratio. In each case, the optical density or absorbance value at a particular maximum absorbance wavelength (λ_{420nm}) for the ethanol extract of the stem of *Achras sapota* was estimated using Hitachi-U-2000 UV-VIS absorbance spectrometer.

Various conditions used for the aqueous extraction of colour component from the stem of *Achras sapota* and the respective absorbance values are given below:

Extraction variable time (min.)	Absorbance of colour component at $\lambda_{420\ nm}$
15	2.61
30	2.70
60	2.82
90	2.72
120	2.61
Temperature ^{0} C	
30	1.70
60	2.20
90	2.00
120	1.90
Material-to-liquor ratio	
1:10	2.24
1:20	2.72
1:30	2.45
1:40	2.56
1:50	2.49

The bold values indicate the optimum conditions for the extraction of colour component from the stem of *Achras sapota.* In usual cases the ethanol extract of dye liquor from the stem of *Achras sapota* was prepared by following the above optimized conditions of extraction.

Dyeing of cotton and silk fabrics with the extract of stem of *Achras sapota:*

The wetted out cotton samples were dipped into the dye baths containing required amount of dye extract and water. After 10 minutes, required amount of sodium carbonate and sodium chloride were added. The dyeing was carried out for one hour at 60°C.

Pre-Mordanting of cotton and silk fabrics with myrobolan and metallic salts:

Conventionally bleached (H_2O_2) cotton and silk fabrics with or without being subjected to pre-mordanting were further mordanted prior to dyeing using 1-3 per cent of any one of the chemical mordants, such as aluminium sulphate, nickel sulphate, potassium dichromate, stannous chloride, copper sulphate and the myrobolan, at 60^{0}C for 30 min with material-to-liquor ratio of 1:20. The samples treated with metal salts were dyed with the dye extract.

Simultaneous -Mordanting of cotton and silk fabrics with myrobolan and metallic salts:

Bleached cotton and silk were treated with both dye extract and metal salts simultaneously, using 1-3 per cent of any one of the chemical mordants, such as aluminium sulphate, nickel sulphate, potassium dichromate, stannous chloride, copper sulphate and the myrobolan, at 60^{0}C for 30 min with material-to-liquor ratio of 1:20.

Post-Mordanting of cotton and silk fabrics with myrobolan and metallic salts:

Bleached cotton and silk were dyed with dye extract. The wetted out cotton and silk samples were dipped into different dye baths containing required amount of dye extract and water. After 10 minutes required amount of sodium sulphate was added. After 20 minutes required amount of sodium chloride was added. The dyeing was carried out for one hour at 50^{0}C. The unwashed dyed samples were taken out, squeezed and used for treatment with metal salts. The dyed cotton and silk samples were treated with different metal salts using 1-3 per cent of any one of the chemical mordants, such as aluminium sulphate, nickel sulphate, potassium dichromate, stannous chloride, copper sulphate and the myrobolan, at 60^{0}C for 30 min with material-to-liquor ratio of 1:20.

In each case, for general study of dyeing behaviour using different mordants, a prefixed normal dyeing condition (ethanol extract of *Achras Sapota*, 5 per cent ; mordant, 1-3 per cent ; MLR, 1:20; common salt 5 g l^{-1} with requisite amount of NaOH; dyeing temperature, 60^0C and dyeing time, 60min) was used.

In all the above three methods, after the dyeing is over, the dyed samples were subjected to soaping with 2 g l^{-1} soap solution at 50^0C for 10 min, followed by repeated water wash and drying under sun.

Determination of surface colour strength[13] (K/S value):

The K/S value of the dyed cotton and silk fabrics was determined by measuring the surface reflectance of the samples using a computer-aided Macbeth 2020 plus reflectance spectrophotometer, using the following Kubelka Munk equation with the help of relevant software:

$$K/S = \frac{(1 - R\lambda_{max})2}{2R\lambda_{max}} = \alpha C_d$$

where K is the coefficient of absorption; S the coefficient of scattering; C_d, the concentration of the due 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.

Evaluation of colour fastness:

Colour fastness to washing 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-A02 (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) was assessed as per IS: 766-1984 method using a manually operated crock meter and grey scale as per ISO-105-AO3 (extent of staining).

Colour fastness[14] to exposure to light was determined as per IS: 2454-1984 method. The sample 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).

Colour fastness to perspiration assessed according to IS 971-1983 composite specimen was prepared by placing the test specimen between two adjacent pieces of fabrics of silk and cotton and stitched all among four sides. The sample was soaked in the test solution (acidic /alkaline) separately with MLR 1:50 for 30 minutes at room temperature. The sample was then placed between two glass plates of perspirometer under load of 4.5kg (10 lbs). The apparatus was kept in the oven for four hours at 37 ± 2^0C. At the end of this period the specimen was removed and dried in air at a temperature not exceeding 60^0C. The test samples were graded for change in colour and staining using grey scales.

EXPERIMENTAL FINDINGS AND ANALYSIS

Bleached silk and cotton fabrics mordanted with varying concentration of mordants have been subsequently dyed by using pre, simultaneous and post-mordanting

Table 1 : Surface colour strength of dyed cotton fabrics after pre, simultaneous and post –mordanting methods by using % mordant concentration (K/S value without mordant: Silk-2.34 and cotton-1.31)

Fabric	Mordant concn.: 1%	K/S(λ=420 nm)		
		Pre-mordanting	Simultaneous mordanting	Post-mordanting
Silk	Nickel sulphate	2.17	2.49	2.39
	Aluminium sulphate	2.45	2.71	2.51
	Potassium dichromate	1.88	2.17	2.08
	Ferrous sulphate	2.48	2.77	2.63
	Stannous chloride	2.39	2.64	2.54
	Myrobolan	1.66	2.08	2.02
Cotton	Nickel sulphate	1.41	2.34	2.07
	Aluminium sulphate	1.71	2.57	2.48
	Potassium dichromate	1.18	1.26	1.32
	Ferrous sulphate	1.77	2.61	2.72
	Stannous chloride	1.62	2.53	2.38
	Myrobolan	0.92	1.22	1.26

Table 2 : Surface colour strength of dyed cotton fabrics after pre, simultaneous and post –mordanting methods by using 2% mordant concentration (K/S value without mordant: Silk-2.34 and cotton-1.31)

Fabric	Mordant concn.: 2%	K/S(λ=420 nm)		
		Pre-mordanting	Simultaneous mordanting	Post-mordanting
Silk	Nickel sulphate	2.20	2.54	2.43
	Aluminium sulphate	2.49	2.76	2.56
	Potassium dichromate	1.91	2.24	2.12
	Ferrous Sulphate	2.52	2.82	2.65
	Stannous Chloride	2.43	2.67	2.57
	Myrobolan	1.70	2.12	2.09
Cotton	Nickel sulphate	1.46	2.37	2.12
	Aluminium sulphate	1.75	2.59	2.53
	Potassium dichromate	1.23	1.29	1.34
	Ferrous sulphate	1.82	2.67	2.76
	Stannous chloride	1.67	2.56	2.43
	Myrobolan	0.98	1.27	1.31

Table 3: Surface colour strength of dyed cotton fabrics after pre, simultaneous and post–mordanting methods by using 3% mordant concentration (K/S value without mordant: Silk-2.34 and cotton-1.31)

Fabric	Mordant concn.: 3%	K/S(λ=420 nm)		
		Pre-mordanting	Simultaneous mordanting	Post-mordanting
Silk	Nickel sulphate	2.23	2.57	2.47
	Aluminium sulphate	2.54	2.81	2.61
	Potassium dichromate	1.95	2.28	2.18
	Ferrous sulphate	2.56	2.86	2.69
	Stannous chloride	2.47	2.70	2.61
	Myrobolan	1.74	2.16	2.14
Cotton	Nickel sulphate	1.51	2.41	2.16
	Aluminium sulphate	1.79	2.64	2.58
	Potassium dichromate	1.27	1.34	1.38
	Ferrous sulphate	1.87	2.71	2.81
	Stannous chloride	1.71	2.62	2.48
	Myrobolan	1.03	1.31	1.35

methods as reported in section 2.2.3, 2.2.4 and 2.2.5. All the dyed fabrics have been assessed for their colour strength (K/ S) value as reported in Table 1, 2 and 3. All the dyed fabrics have been assessed for their colour fastness behaviour to washing, rubbing and exposed to light and perspiration and the results are given in Table 4.

All the treated samples subjected to light which show fairly good (4) to light fastness and excellent grade to washing fastness and all the treated samples no colour staining to washing fastness. The colour change to dry and wet rubbing for all the treated samples was excellent (5). There was slight colour staining except for simultaneous mordanting method where it was negligible staining (4-5).

The perspiration fastness grades ranged between 4 and 5 for all samples in both acidic and alkaline media. There was no colour staining (5) for all the treated samples in both acidic and alkaline media.

It is interesting to note that harda, being a natural light yellow mordantable dye, shows a steady increase in K/S value on both silk and cotton fabrics.

It is observed from the Fig. 1, 2, 3, 4, 5 and 6 that among the three mordanting techniques, simultaneous mordanting gave excellent results (K/S value) as compared to other mordanting system.

It is observed that among differently mordanted bleached silk subsequently dyed with 5 per cent ethanol extract of stem of *Achras sapota* with 3 per cent ferrous sulphate by simultaneous mordanting technique, renders the fabric relatively higher K/S value (~2.86) as compared to other mordanting system .The use of 3 per cent aluminium sulphate by simultaneous mordanting technique followed by further dyeing with comparable dose of 5 per cent ethanol extract of stem of *Achras sapota* colour

Table 4 : Colourfastness of dyed cotton and silk fabrics with selective mordants using pre, simultaneous and post–mordanting methods

Mordants	Method of mordanting	Mordant concentration (%)	Washing			Rubbing				Sun light	Perspiration					
				CS		Dry		Wet			Acidic			Alkaline		
			CC	S	C	CC	CS	CC	CS		CC	CS		CC	CS	
												S	C		S	C
Nickel sulphate	Pre-mordanting	1	5	4	4	5	5	5	5	4	5	5	5	5	5	5
		2	5	4	4	5	5	5	5	4	5	5	5	5	5	5
		3	5	5	5	5	5	5	5	4	4	5	5	4	5	5
	Simultaneous mordanting	1	5	4	4 -5	5	5	5	5	4	5	5	5	5	5	5
		2	5	4	4 -5	5	4-5	5	5	4	5	5	5	5	5	5
		3	5	4	4	5	5	5	5	4	4	5	5	4	5	5
	Post-mordanting	1	5	4	4 -5	5	5	5	5	4	5	5	5	5	5	5
		2	5	4	4 -5	5	5	5	5	4	5	5	5	5	5	5
		3	5	4	4	5	5	5	5	4	4	5	5	4	5	5
Aluminium sulphate	Pre-mordanting	1	5	4	4	5	5	5	5	4	5	5	5	5	5	5
		2	5	4	4	5	5	5	5	4	5	5	5	5	5	5
		3	5	5	5	5	5	5	5	4	4	5	5	4	5	5
	Simultaneous mordanting	1	5	4	4 -5	5	4	5	4	4	5	5	5	5	5	5
		2	5	4	4 -5	5	4-5	5	4-5	4	5	5	5	5	5	5
		3	5	4	4	5	5	5	5	4	4	5	5	4	5	5
	Post-mordanting	1	5	4	4 -5	5	5	5	5	4	5	5	5	5	5	5
		2	5	4	4 -5	5	5	5	5	4	5	5	5	5	5	5
		3	5	4	4	5	5	5	5	4	4	5	5	4	5	5
Potassium dichromate	Pre-mordanting	1	4	4	4	5	5	5	5	3	4	5	5	4	5	5
		2	4	4	4	5	5	5	5	3	3	5	5	4	5	5
		3	4	4	4	5	5	5	5	4	3	5	5	4	5	5
	Simultaneous mordanting	1	3	4	3	5	4	5	4	3	3	5	5	3	5	5
		2	4	3	3	5	4-5	5	4-5	3	3	5	5	3	5	5
		3	4	4	3	5	5	5	5	4	3	5	5	4	5	5
	Post-mordanting	1	4	4	3	5	5	5	5	3	3	5	5	3	5	5
		2	4	3	4	5	5	5	5	3	3	5	5	3	5	5
		3	4	4	4	5	5	5	5	4	4	5	5	4	5	5
Ferrous sulphate	Pre-mordanting	1	4	4	4 -5	5	5	5	5	4	4	5	5	4	5	5
		2	4	4	4 -5	5	5	5	5	4	4	5	5	4	5	5
		3	4	5	4	5	5	5	5	4	4	5	5	4	5	5
	Simultaneous mordanting	1	5	4 -5	4 -5	5	4	5	5	4	4	5	5	4	5	5
		2	5	4	4	5	4-5	5	5	4	4	5	5	4	5	5
		3	5	5	4	5	5	5	5	4	4	5	5	4	5	5
	Post-mordanting	1	5	4	4 -5	5	5	5	5	4	4	5	5	5	5	5
		2	5	4	4 -5	5	5	5	5	4	4	5	5	5	5	5
		3	5	4	4	5	5	5	5	4	4	5	5	4	5	5
Stannous chloride	Pre-mordanting	1	4	4	4 -5	5	5	5	5	4	4	5	5	4	5	5
		2	4	4	4	5	5	5	5	4	4	5	5	4	5	5
		3	5	5	3 -4	5	5	5	5	4	4	5	5	4	5	5
	Simultaneous mordanting	1	5	4	4 -5	5	4	5	4-5	4	4	5	5	4	5	5
		2	5	4	4	5	4-5	5	4-5	4	4	5	5	4	5	5
		3	5	5	4	5	5	5	5	4	4	5	5	4	5	5
	Post-mordanting	1	4	4	4 -5	5	5	5	5	4	4	5	5	5	5	5
		2	5	5	4 -5	5	5	5	5	4	4	5	5	5	5	5
		3	5	5	4	5	5	5	5	4	4	5	5	4	5	5
Myrobolan	Pre-mordanting	1	3	4	4	5	5	5	5	4	4	5	5	4	5	5
		2	3	4	3 -4	5	5	5	5	4	3	5	5	4	5	5
		3	4	4	4	5	5	5	5	4	4	5	5	4	5	5
	Simultaneous mordanting	1	3	4	4	5	4	5	5	4	4	5	5	4	5	5
		2	3	4	3 -4	5	4	5	5	4	5	5	5	4	5	5
		3	4	4	4	5	5	5	5	4	4	5	5	4	5	5
	Post-mordanting	1	3	4	4	5	5	5	5	4	4	5	5	4	5	5
		2	3	4	3 -4	5	5	5	5	4	5	5	5	4	5	5
		3	4	4	4	5	5	5	5	4	4	5	5	4	5	5
Control	-	-	4 -5	4 -5	4	5	5	5	5	4	4	5	5	4	4	5

C CC – Colour change, CS – Colour staining, S-Silk, C – Cotton

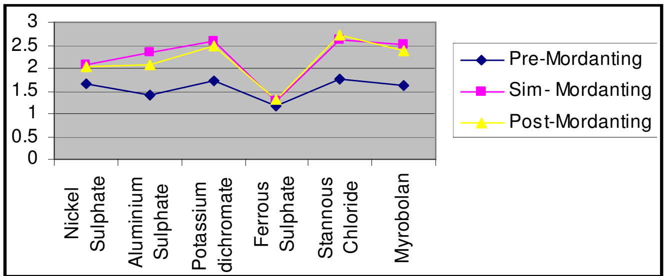

Fig. 1 : Surface colour strength of dyed silk fabrics after pre, simultaneous and post –mordanting methods by using 1 per cent mordant concentration (K/S value without mordant: Silk-2.34 and cotton-1.31)

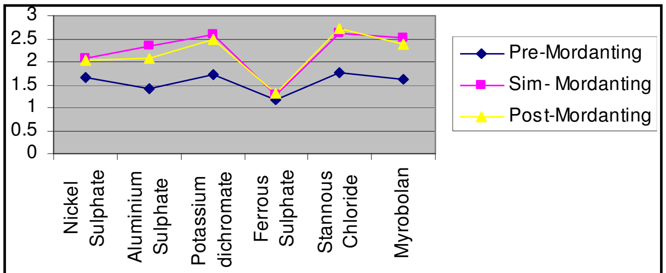

Fig. 2 : Surface colour strength of dyed cotton fabrics after pre, simultaneous and post –mordanting methods by using 2 per cent mordant concentration (K/S value without mordant: Silk-2.34 and cotton-1.31)

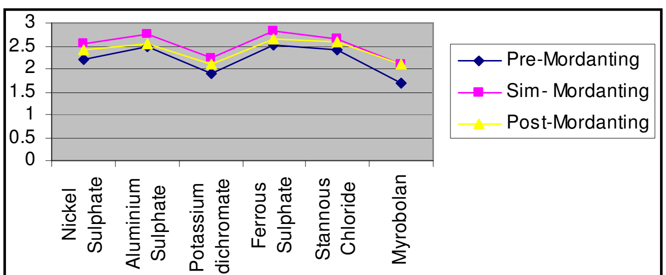

Fig. 3 : Surface colour strength of dyed silk fabrics after pre, simultaneous and post –mordanting methods by using 2 per cent mordant concentration (K/S value without mordant: Silk-2.34 and cotton-1.31)

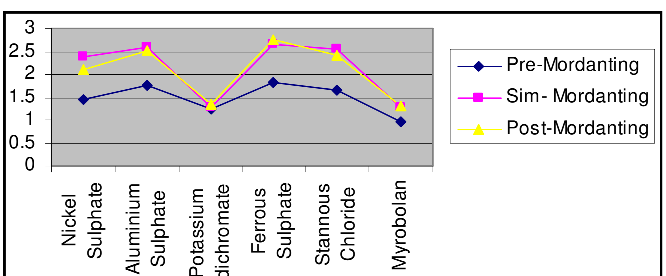

Fig. 4 : Surface colour strength of dyed cotton fabrics after pre, simultaneous and post –mordanting methods by using 2 per cent mordant concentration (K/S value without mordant: Silk-2.34 and cotton-1.31)

shows the K/S value of 2.81 and thus is considered as next good performer (Fig. 5)

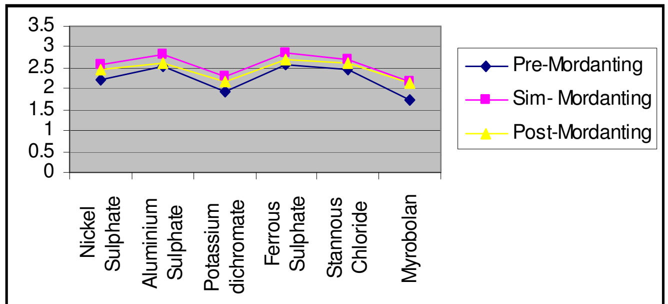

Fig. 5 : Surface colour strength of dyed silk fabrics after pre, simultaneous and post –mordanting methods by using 3 per cent mordant concentration (K/S value without mordant: Silk-2.34 and cotton-1.31)

Similarly for cotton fabric, it is observed that among differently mordanted bleached cotton subsequently dyed with 5 per cent ethanol extract of stem of *Achras sapota* with 3 per cent ferrous sulphate by simultaneous mordanting technique, renders the fabric relatively higher K/S value (~2.71) as compared to other mordanting system .The use of 3 per cent aluminium sulphate by simultaneous mordanting technique followed by further dyeing with comparable dose of 5 per cent ethanol extract of stem of *Achras sapota* colour shows the K/S value of 2.64 and thus is considered as next good performer(Fig. 6).

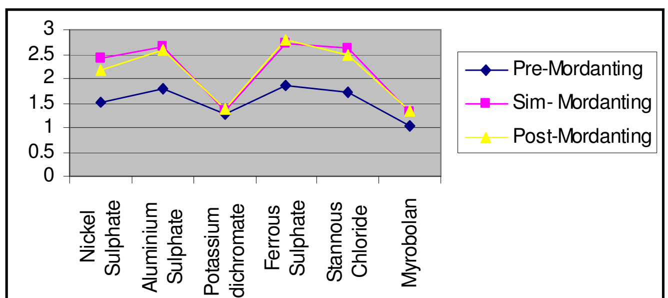

Fig. 6 : Surface colour strength of dyed cotton fabrics after pre, simultaneous and post –mordanting methods by using 3 per cent mordant concentration (K/S value without mordant: Silk-2.34 and cotton-1.31)

Among all the mordants used, the increase in K/S value is found to be the highest for ferrous sulphate mordant due to the inherent colour of ferrous sulphate salt .This is more predominant on silk than on cotton.

The increase in colour strength K/S values after pre, simultaneous and post-mordanting with selective mordants (1-3%) on both silk and cotton fabrics are in the following order:

For silk : FeSO4 >$Al_2(SO_4)_3$> $SnCl_2$> $NiSO_4$> $K_2Cr_2O_7$>Myrobolan

For cotton: FeSO4 >$Al_2(SO_4)_3$> $SnCl_2$> $NiSO_4$> $K_2Cr_2O_7$Myrobolan

This increase in K/S value to a different extent after pre, simultaneous and post-mordanting may be due to the changes in scattering because of the chemical interaction between fibres and harda or metallic salts along with the additional inherent colour input of the corresponding mordants.

Hence ,considering the dyeing results, the sequential mordanting systems using 3 per cent $FeSO_4$+ 5 per cent ethanol extract of stem of *Achras sapota* and (ii) 3 per cent $Al_2(SO_4)_3$ 5 per cent ethanol extract of stem of *Achras sapota* are found to be more prospective, rendering a higher degree of increase in surface colour strength.

These above two systems of mordanting have therefore been chosen for further study of dyeing process variables for both silk and cotton fabrics.

The observed highest K/S value in case of fibre-mordanting system on bleached silk 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 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 $FeSO_4$ itself.

Conclusion:

It was found from the study that stem of *Achras sapota* dye can be successfully used for dyeing of silk and cotton to obtain a wide range of soft, pastel and light colours by using natural and metallic mordants. With regards to colourfastness,test samples exhibited excellent fastness to washing(except pre,simultaneous and post mordanting-$K_2Cr_2O_7$);excellent fastness to rubbing (except pre,simultaneous and post mordanting- $K_2Cr_2O_7$); good to excellent fastness to perspiration in both acidic and alkaline media and fairly good fastness to light.

Among the different fibre-mordanting systems studied, the use of 3 per cent of ferrous sulphate applied by simultaneous mordanting for subsequent dyeing on cotton and silk with 5 per cent ethanol extract of stem of *Achras sapota* and 3 per cent of aluminium sulphate applied by simultaneous mordanting for subsequent dyeing on cotton and silk with 5 per cent ethanol extract of stem of *Achras sapota* show maximum K/S values as compared to other selective pre, simultaneous and post mordanting systems.

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