

DYEING OF WOOL AND SILK WITH TEA

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ABSTRACT

Silk and wool fabrics were coloured employing aqueous tea extract in absence and presence of magnesium sulphate, aluminium sulphate and ferrous sulphate as the mordanting agents. Colour uptake for wool was found to be more than that for silk under all conditions studied. Colouring biochemical components of tea revealed highest affinity for both the protein fibres at pH 2 to 4 in presence and absence of such mordanting agents. Use of ferrous sulphate and aluminium sulphate produced significant improvement in colour uptake following a pre- or post mordanting methods. Coloured protein fibres in general produced a light-fastness rating of 5 on a scale of 1- 8 and a wash-fastness rating of 4 on a scale of 1-5. Ferrous sulphate and aluminium sulphate improved colour retention on washing and the fastness properties further. Coloured protein fibres became blackish, when ferrous sulphate was employed as mordanting agent.

Keywords: India; wool; silk; natural dye; mordant

INTRODUCTION

Growing consciousness about environmental pollution and toxic and allergic effects associated with the use of synthetic dyes have recently renewed interest for application of natural dyes on textile. Considerable research work is being undertaken around the world on the application of natural dyes (Gulrajani et.al, 1999a-c). It is imperative in such case that the natural dye and the associated process for application of such dye on textile substrate should be free from any hazards. Application of tea (*Camellia sinensis*) as natural dye on natural fibres has been reported elsewhere (Nicholas, 1996; Deo et al, 1999; Teli et.al; 2002). Tea contains flavonol glycosides, theaflavin, thearubigins, phaeophorbide and carotene as the chief colouring biochemical compounds. The colour of the tea liquor is entirely due to the theaflavins and thearubigins (Sarma, 1999). All the above compounds are capable of forming complexes with magnesium, aluminium and iron as they contain ortho-dihydroxy benzene

in their structures. Moreover, phaeophorbide has the potential to combine with magnesium with the formation of a complex known as chlorophyll-a, that shows variable colour from green to yellowish-brown depending on pH of reaction medium (Finar, 1975). *S. epidermiosis*, *S. aureus* and *Coryneforms* are the odour-causing bacteria found in different parts of human skin (Marples, 1965; Kloos, 1975). Antibacterial activity of tea, when applied as dye on textile, has also been reported (Ishikawa, 2005). We, therefore, selected aqueous extract of tea as natural dye for application on protein fibres, such as wool and silk, in view of wide availability and cost effectiveness of the former, and vast popularity and wide acceptance of naturally dyed, elegant, value-added and niche products made from those fibres. This research work is aimed at establishing optimum conditions for application of tea as a dye on protein fibres and evaluating colour-fastness properties of such fibres when dyed with tea.

MATERIALS AND METHODS

A commercial sample of tea obtained from Tata Tea was used for this study. Plain weave bleached wool and loom state silk fabrics having 236 ends/dm and

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430 ends/dm, and having 204 picks/dm and 212 picks/dm with 150 g/m² and 50 g/m² in weight were used. Acrylic yarn made of copolymer having acrylonitrile 91%, vinyl acetate 8.5% and sodium styrene sulphonate 0.5% was also used. All chemicals used in this study were of laboratory reagent grade.

Extraction of Tea

Aqueous extract of tea was prepared by adding 20 g of tea to one liter of water. The mixture was stirred, heated and kept at boiling point for 30 minutes, allowed to stand for 15 minutes and finally filtered. The filtrate was used for dyeing wool and silk.

De-gumming and Bleaching of Silk

The loom state silk fabric was de-gummed at 90^o C for 1.5 hours in an aqueous solution containing soap (6.0 g/l) and sodium carbonate (2.0 g/l). The de-gummed silk sample was bleached at 85^o C for 1 hour in a solution containing hydrogen peroxide (0.9%), non-silicate stabilizer (0.15%) and sodium carbonate (0.1%). Material to liquor ratio for both de-gumming and bleaching operations was maintained at 1:20. Bleached sample was washed at 70^o C for 10 minutes, cold washed and finally dried.

Dyeing of Substrates with Tea Extract

a. In absence of mordant: Silk, wool and acrylic fabrics were dyed using aqueous tea extract at a material to liquor ratio of 1:50. The dye bath temperature was kept at 90^o C for 1 hour. The pH of the dye bath was set at different specified levels ranging from 2 to 10. Hydrochloric acid, acetic acid and sodium carbonate were employed for adjustment of pH at such specified levels.

b. In presence of mordant: Dyeing of silk and wool with aqueous tea extract was carried out in presence of different specified mordants following three different methods like pre-mordanting, post-mordanting and simultaneous mordanting; 5g/l mordant concentration was employed in all the cases. In the pre-mordanting method, the fabrics were first immersed in an aqueous solution of mordants for 45 minutes at 70^o C and rinsed thoroughly with water. The mordanted fabrics were then dyed at 90^o C for 1 hour with a material to liquor ratio of 1:50. For simultaneous mordanting, the fabrics were immersed in a bath containing mordant and dye extract. The dyeing temperature was kept at 90^o C and dyeing was carried out for 1 hour with a material to liquor ratio of 1:50. In case of post mordanting, dyeing was carried out in absence of mordants at 90^o C for 1 hour and a cold wash was given thereafter. Dyed fabrics were then mordanted in a separate bath at 70^o C for 45 minutes. Soaping of all the dyed fabric samples was done employing 2 g/l non-ionic detergent at 60^o C for 10 minutes. Finally the fabric samples were cold washed and dried. Dyeing of the above substrates, following pre mordanting method as described, was done at different specified pH ranging from 2 to 10.

Determination of Wavelength of Maximum Absorption

The wavelength of maximum absorption of aqueous extract of tea at different specified pH was determined employing Hitachi (U-2000), UV-visible absorbance spectrophotometer. Prior to such determination aqueous solution of tea extract was diluted to an appropriate level.

Estimation of Exhaustion (Das et.al, 1997)

The exhaustion of tea extract to different specified substrates was determined from the difference of

initial concentration of tea extract taken in the dye bath and final concentration remaining in the dye bath (including that of wash liquor) after exhaustion. The difference was expressed as percent of initial concentration in each case. The concentration of colouring component was estimated colorimetrically employing Hitachi (U-2000), UV-visible absorbance spectrophotometer.

Assessment of Dye Receptivity in Terms of K/S

In order to determine dye receptivity, the K/S value of fabric samples dyed using aqueous tea extract under the specified conditions was examined in a Mcbeth 2020+ reflectance spectrophotometer interfaced with a computer. The reflectance values (R) of the dyed fabric at wavelength of maximum absorption were converted to the corresponding K/S value using the Kubelka-Munk equation, i.e. $K/S = (1-R)^2 / [2R]$, where K is the absorption coefficient and S is the scattering coefficient (Billmeyer, 1981). A higher K/S value indicates better dye receptivity of the substrates. The result shown here is the average of seven scans over a wavelength range of 400-700 nm for each sample.

Determination of Colour-fastness Properties

Color-fastness to washing and light were determined following a standard procedure as described in IS: 3361-1984 (ISO- II) and IS: 2454-1984, respectively. For determination of colour-fastness to washing following ISO-II method, the specified fabric samples were washed at 50° C using 5 gram per liter soap solution at a material to liquor ratio of 1:50 for 45 minutes in a Launder-ometer. Assessment of colour-fastness to light was done on 1-8 scales against standard Blue Wool reference and assessment of colour-fastness to washing was done on 1-5 scale.

RESULTS AND DISCUSSION

Wavelength of Maximum Absorption of Tea Extract

Table 1 shows wavelength of maximum absorption of tea extract in absence and presence of acid, alkali and mordanting agents. Tea extracts as experimentally obtained showed a pH ~ 5.8. The wavelength of maximum absorption of tea extract remains unaltered for lowering of its pH to a value of 1.5 and 3.0 when hydrochloric acid and acetic acid were added respectively. Addition of alkali, such as sodium carbonate, alters the wavelength of maximum absorption value of tea extract from 381 to 405. Among the mordanting agents employed in this study, magnesium sulphate causes insignificant shifting of wavelength of maximum absorption value. The coloured tea extract became almost colourless upon addition of aluminium and ferrous sulphate separately in consequent to instant and immediate precipitation of the complex formed, when such metals came in contact with the colouring component of tea at normal temperature. Among Mg^{+2} , Al^{+3} and Fe^{+2} , the intensity of formation of complex as described elsewhere (Lee, 1996) follows the order $Mg^{+2} < Al^{+3} < Fe^{+2}$. The results obtained in this case with the tea extract are in tune with the order described above.

Table 1. Wavelength of maximum absorption of tea extract

Tea extract treated with	pH	Maximum absorbance (nm)	Colour observed
None	5.8	380.50	Yellowish- brown
Hydrochloric acid	1.50	381.0	Yellowish- brown
Acetic acid	3.0	381.0	Yellowish- brown
Sodium carbonate	9.0	405	Brownish-red
Magnesium sulphate	5.8	386.0	Yellowish- brown
Aluminium sulphate	2.60	Lake formed	Reddish- brown
Ferrous sulphate	5.10	Lake Formed	Blackish-brown

Effect of Variation of pH

The effect of variation of pH on the exhaustion of dye bath in absence of any mordant, and also that on depth of shade of wool and silk substrates in absence and presence of mordants for application of the tea extracts is shown in Figs. 1, 2 and 3 respectively. Insignificant exhaustion of tea extract to acrylic at different dye bath pH, as shown in Fig. 1, indicates that water soluble colouring component of tea extract has no affinity for acrylic fibre having anionic ($-\text{SO}_3\text{H}$) sites. Exhaustion of tea extract to wool and silk substrates, however, was observed to be significant at all the dye bath pH studied (Fig.1). Exhaustion of tea extract to wool and silk substrates and also the K/S value of those substrates follow a common increasing trend with lowering of dye bath pH (Figs. 1-3). Protonation of both the fibres commonly resulted in an increase in dye uptake showing maximum dye uptake in the dye bath pH range of 2 to 4. In view of absence of any other functional group in the colouring component of tea, it is assumed that the dye fibre attraction in absence of mordants depends on (i) ionic attraction between phenoxide ions of polyphenols of tea and $-\text{NH}_3^+$ group of protein fibre and on (ii) non-polar Vander-waals' interaction between hydrophobic part of dye anion having benzene ring and hydrophobic parts of protein fibres adjacent to the amino groups. Colouring component of tea appears to play the role of an acid dye as it shows increased affinity for protonated fibre; the said affinity appears to be comparable even when dyeing was carried out at near neutral pH (pH 6 and 8) as shown in Figs. 1, 2 and 3. The affinity of dye at near neutral pH is due to the presence of benzenoid rings in the colouring component of tea extract that imparts affinity for wool and silk fibres to the tea extract. Keratin in wool and hydrocarbon chain of silk fibre having hydrophobic regions lead

to such hydrophobic dye-fibre interaction in near neutral pH in a similar manner as observed for application of acid dye on protein fibres (Bird, 1972; Gulrajani, 1993d). Dye uptake as assessed in terms of K/S value appears to be higher in case of wool than that observed in case of silk for all the pH considered in this study (Figs. 2 and 3). This may be viewed as the consequence of presence of more amino groups and higher amorphous region of wool in comparison to those for silk (Bird, 1972). Use of magnesium, aluminium and ferrous sulphate led to improved dye uptake for silk and wool substrates at all the pH considered for application of tea extract in this study. Application of tea extract is recommended to be commenced at pH 4 to 4.5 both in presence and absence of mordanting agents in view of appreciable hydrolytic degradation of protein fibres at elevated temperature of dyeing for a length of time at dye bath pH around or less than 3.5 (Bird, 1972) and insignificant improvement in K/S of all the substrates for lowering dye bath pH from 4 to 2.

Fig.1. Plots of exhaustion of tea extract on 1) acrylic, 2) silk and 3) wool substrates against dye bath pH.

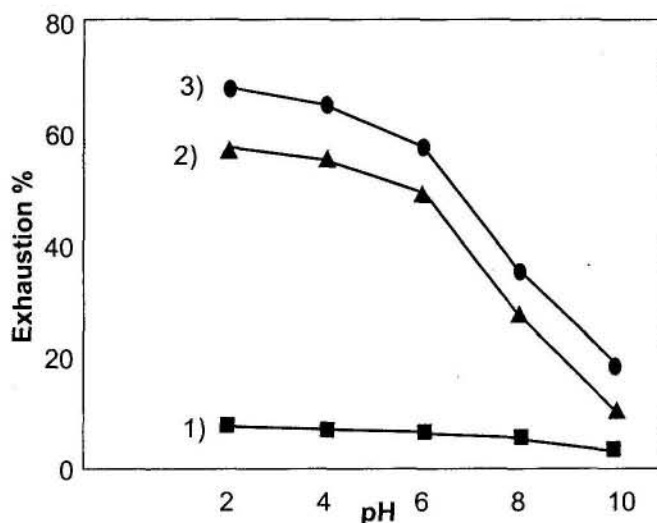


Fig. 2. Plots of K/S against dye bath pH for wool dyed with tea extract in presence of 1) MgSO_4 , 2) $\text{Al}_2(\text{SO}_4)_3$, 3) FeSO_4 as mordanting agents and 4) no mordanting agents.

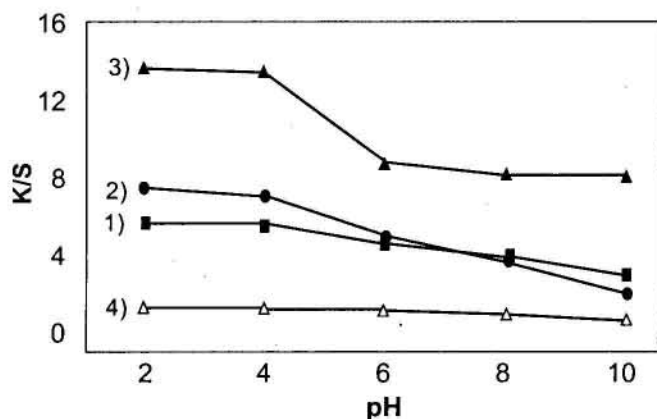
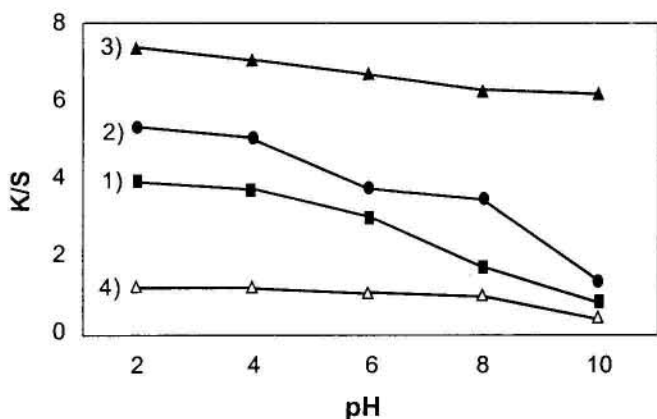


Fig. 3. Plots of K/S against dye bath pH for silk dyed with tea extract in presence of 1) MgSO_4 , 2) $\text{Al}_2(\text{SO}_4)_3$, 3) FeSO_4 as mordanting agents and 4) no mordanting agent



Effect of Variation of Mordanting Methods

Application of tea extract was made on silk and wool substrates employing aluminium, magnesium and ferrous sulphate as the mordanting agents following a pre, post and simultaneous mordanting methods. Results of such studies assessed in terms of K/S (measured at wavelength of maximum absorption) are shown in Table 2.

Table 2. Effect of variation of mordanting method.

Fibre	Mordanting agent used	Mordanting method		
		Pre [(K/S) _{420 nm}]	Post [(K/S) _{420 nm}]	Simultaneous [(K/S) _{420 nm}]
Silk	Magnesium sulphate	3.75 (25.01)	3.90 (24.82)	3.77 (27.16)
	Aluminium sulphate	5.13 (24.27)	4.79 (23.20)	3.37 (24.61)
	Ferrous sulphate	7.96 (1.45)	7.62 (5.55)	5.42 (1.48)
Wool	Magnesium sulphate	5.82 (27.05)	5.73 (26.24)	3.84 (29.20)
	Aluminium sulphate	7.39 (23.84)	7.09 (25.35)	3.62 (23.58)
	Ferrous sulphate	13.65 (3.96)	13.15 (3.86)	5.61 (1.06)

Data given in the parenthesis refer to chroma

For application of tea extract on silk and wool following pre or post mordanting methods, the ability of the mordanting agents to anchor the colour on both the substrates follow a common order $\text{FeSO}_4 \gg \text{Al}_2(\text{SO}_4)_3 > \text{MgSO}_4$; however, that for simultaneous mordanting method it appears to be $\text{FeSO}_4 > \text{MgSO}_4 > \text{Al}_2(\text{SO}_4)_3$. K/S of the dyed substrates is comparable for following a pre and post mordanted methods for all the three mordanting agents employed. Simultaneous mordanting method on the other hand leads to a commonly inferior level of dye uptake to pre or post mordanting methods. Above fact may be viewed as the consequence of relative ability of metal ions to form complex with the colouring component of tea extract. Iron and aluminium (with their superior complex forming ability with the tea extract) formed complex more readily prior to transportation of colouring matter onto the substrates than magnesium, thus giving poor K/S value of the dyed substrates for following simultaneous mordanting method. For following pre or post mordanting method, however, superior

complex forming ability of aluminium and ferrous sulphate could be realized in the fibre as evident from the K/S of the respective samples shown in Table 2. Notably less chroma values for ferrous sulphate mordanted dyed silk and wool substrates are the consequence of presence of iron in the dyed substrate that makes the colour much duller and blackish when compared with those for aluminium or magnesium sulphate mordanted samples. Effect of variation of mordanting methods for three different mordanting agents as described above on the silk substrates is found to be similar for wool substrate also (Table 2). On analysis of the results given in Table 2, it appears that application of tea extract on wool and silk is best accomplished following a pre mordanting method for employing ferrous sulphate and aluminium sulphate as the mordanting agents.

Effect of Variation of Dose in Mordanting Agents

Effect of variation of dose level of mordanting agents on observed colour of wool and silk substrates, when dyed with aqueous tea extract maintaining a pH of 4.5, is shown in Table 3. Use of magnesium sulphate as the mordanting agent gives both the substrates a common yellowish-brown colour. Aluminium sulphate when used as mordanting agent produces a reddish-brown colour. Depth of shade of both the above colours is found to follow a common increasing trend with increase in dose levels of respective mordants without alteration of hue of the dyed substrates. Ferrous sulphate, however, gives a good blackish-brown colour when used as mordanting agent up to a dose level of 5 gram per liter. An application of 10 gram per liter of ferrous sulphate alters the hue of the produced shade from blackish-brown to reddish-black.

Table 3. Effect of variation of dose of mordanting agents

Substrate	Mordanting agent used	Dose level (gram per litre)	Colour observed
Silk	Absent Magnesium sulphate	Nil	Yellowish-brown
		1	Yellowish-brown
		5	Yellowish-brown
		10	Yellowish-brown
	Aluminium sulphate	1	Reddish-brown
		5	Reddish-brown
		10	Reddish-brown
	Ferrous sulphate	1	Blackish-brown
		5	Blackish-brown
10		Reddish-black	
Wool	Absent Magnesium sulphate	Nil	Yellowish-brown
		1	Yellowish-brown
		5	Yellowish-brown
		10	Yellowish-brown
	Aluminium sulphate	1	Reddish-brown
		5	Reddish-brown
		10	Reddish-brown
	Ferrous sulphate	1	Blackish-brown
		5	Blackish-brown
10		Reddish-black	

Colourfastness to Light and Wash of Wool and Silk Dyed with Tea Extracts

Table-4 shows data for colour-fastnesses to light and wash for wool and silk, when dyed with and without mordanting agents at pH 4. The rating for colour-fastness to washing for both the above substrates appears to be 4 for change in colour and for staining to the adjacent fabrics, commonly in absence and presence of three mordanting agents used. Different mordanting methods (pre, post and simultaneous) appear to have no effect on the colour-fastness properties of silk and wool fabrics dyed with tea extracts for all the three mordanting agents used. Such high colour-fastness to washing even in absence of any mordants is the consequence of presence of ionic attraction and non-polar vander-waals' interaction between colouring components of tea and protein

fibres as stated earlier. Colour-fastness to light for wool and silk dyed with tea extracts, in absence of any mordanting agent, was improved commonly by 1 point upon application of aluminium sulphate and ferrous sulphate as the mordanting agents for following two different mordanting methods (pre and post); simultaneous mordanting method, however, was incapable of causing such improvements in colour-fastness to light of dyed tea extract (Table-4). Improvement in colour-fastness to light for use of aluminium and ferrous sulphate as mordanting agents is the consequence of enhancement of size of colouring component of tea extract. Aluminium and iron with their good complex forming ability hold two or more of such polyphenols of colouring components of tea together in the fibre structure by forming co-

ordinate bonds, in addition to formation of intermolecular hydrogen bonds between two dye molecules and between dye and fibre. Such enhancement in the rating for colour-fastness to light was absent in case of simultaneous mordanting method, as aggregation of dye molecule in presence of such ferrous and aluminium ions predominantly took place in the solution phase, prior to their transference into fibre phase. Such aggregation of dye molecules is assumed to be absent for use of magnesium sulphate as the mordanting agent, in view of relatively poor complex forming ability of the later. Use of magnesium sulphate as the mordanting agent for application of the tea extract on protein fibres as described above, therefore, shows no improvement in colourfastness to light (Table-4).

Table 4. Colour-fastness to light and wash for silk and wool dyed with tea extract.

Substrate	Dye bath pH	Type of mordant	Mordanting method	Light fastness	Wash fastness		
					Change in colour	Staining on cotton	Staining on silk
Silk	4	None	—	5	4	4 - 5	4 - 5
		Aluminium sulphate	Post mordanting	6	4	4	4
		Magnesium sulphate	Post mordanting	5	3 - 4	4	3-4
		Ferrous sulphate	Post mordanting	6	4	4	4
		Aluminium sulphate	Pre mordanting	6	4	4	4
		Magnesium sulphate	Pre mordanting	5	3-4	4	3-4
		Ferrous sulphate	Pre mordanting	6	4	4	4
		Aluminium sulphate	Simultaneous mordanting	5	4 - 5	4	4
		Magnesium sulphate	Simultaneous mordanting	5	3	4	3-4
		Ferrous sulphate	Simultaneous mordanting	5	4	4	4
Wool	4	None	—	5	4 - 5	4 - 5	4 - 5
		Aluminium sulphate	Post mordanting	6	4	4	4
		Magnesium sulphate	Post mordanting	5	4	4	4
		Ferrous sulphate	Post mordanting	6	3 - 4	4	4
		Aluminium sulphate	Pre mordanting	6	4	4	4
		Magnesium sulphate	Pre mordanting	5	4	4	4
		Ferrous sulphate	Pre mordanting	6	4 - 5	4	4
		Aluminium sulphate	Simultaneous mordanting	5	4	4	4
		Magnesium sulphate	Simultaneous mordanting	5	3-4	4	3-4
		Ferrous sulphate	Simultaneous mordanting	5	4	4	4

Effect of Progressive Wash on Colour Retention

Colour retention of dyed wool and silk expressed in terms of K/S retained for different wash cycles (up to 5) are shown in Fig. 4 and Fig. 5 respectively. The retention of colour of the dyed substrates for the use of three mordanting agents was found to follow the order ferrous sulphate > aluminium sulphate >> magnesium sulphate. The above trend can be appreciated in view of already established similar trend for co-ordinate bond forming ability of the above three metals (Lee, 1996). Aggregation of colouring component and co-ordinate bond formation involving dye and fibre *in-situ* within fibre, caused by above three metals were found to play significant role in holding the dye molecules in the substrates when repeated number of washing at 50° C temperatures was done.

Fig. 4. Plots of K/S against wash cycle for wool dyed with tea extract in presence of 1) MgSO_4 , 2) $\text{Al}_2(\text{SO}_4)_3$, 3) FeSO_4 as mordanting agents and 4) no mordanting agent.

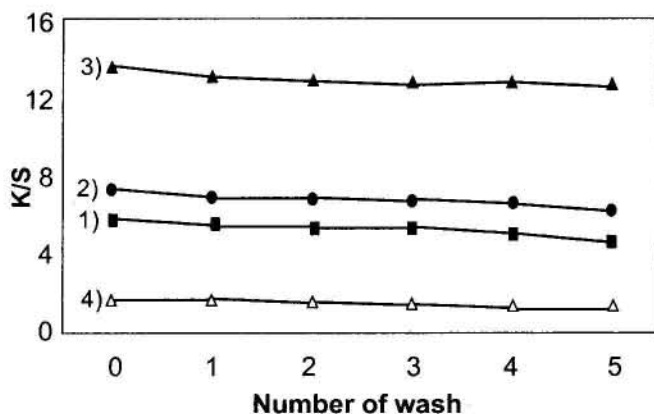
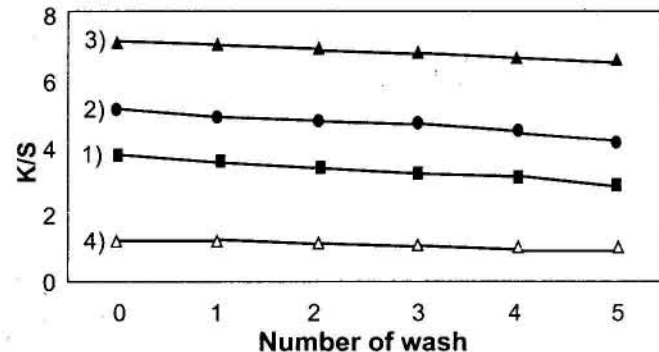


Fig.5. Plots of K/S against wash cycle for silk dyed with tea extract in presence of 1) MgSO_4 , 2) $\text{Al}_2(\text{SO}_4)_3$, 3) FeSO_4 as mordanting agents and 4) no mordanting agent.



CONCLUSIONS

The colouration of silk and wool with tea extracts is shown to be effectively accomplished at pH 4.0 to 4.5. Pre and post mordanting employing ferrous sulphate and aluminium sulphate produced most balanced improvements in colour uptake, light fastness and colour retention on repeated washing. Use of such mordants, however, produced no improvements in washing fastness of dyed substrates. Colour-fastness to light and washing of silk and wool dyed with tea extracts appears to be around 5-6 and 4 respectively. Use of ferrous sulphate as the mordanting agent alters the chroma of tea extracts towards black.

Thus one aims at producing reddish-brown and blackish-brown to reddish-black shade with tea on silk and wool substrate a pre or post mordanting method would be suitable employing aluminium sulphate and ferrous sulphate mordants respectively. Tea also gives a yellowish-brown colour on protein fibres with good fastness properties when applied in absence of any mordanting agent. Therefore, methods for application of tea as natural dye reported here has

the potential to be selectively utilized commercially depending on the desired colour to be produced on silk and wool fibres.

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