

**OPTIMIZATION OF CONDITIONS OF DYEING POLYESTER  
WITH NATURAL DYES EMPLOYING SYNTHETIC  
MORDANTS**

**Dissertation**

**Submitted to the Punjab Agricultural University  
in partial fulfilment of the requirements  
for the degree of**

**DOCTOR OF PHILOSOPHY  
in  
CLOTHING AND TEXTILES**

**(Minor Subject : Economics)**

**By**

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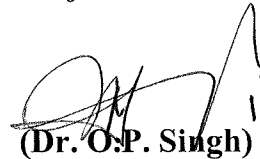
*Dedicated*  
*to my*  
*mother and daughter*

## CERTIFICATE-I

This is to certify that the dissertation entitled, “**Optimization of conditions of dyeing polyester with natural dyes employing synthetic mordants**” submitted for the degree of **Ph.D.** in the subject of **Clothing and Textiles (Minor Subject : Economics)** of the Punjab Agricultural University, Ludhiana is a bonafide research work carried out by **Anita Rani (L-98-HSc-60-D)** under my supervision and that no part of this dissertation has been submitted for any other degree.

The assistance and help received during the course of investigation have been fully acknowledged.

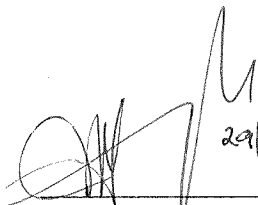
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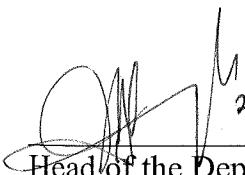
## CERTIFICATE - II

This is to certify that the dissertation entitled, "**Optimization of conditions of dyeing polyester with natural dyes employing synthetic mordants**" submitted by **Anita Rani (L-98-HSc-60-D)** to the Punjab Agricultural University, Ludhiana in partial fulfillment of the requirements for the degree of **Doctor of Philosophy** in the subject of **Clothing & Textiles (Minor subject : Economics)** has been approved by the Student's Advisory Committee after an oral examination on the same, in collaboration with an External Examiner.



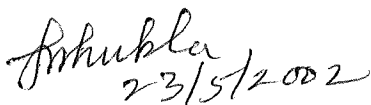
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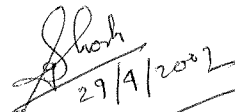
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Ludhiana

*Anita*  
(ANITA RANI)

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## CHAPTER I

# INTRODUCTION

The rainbow, rising and setting sun, blooming flowers, trees, birds, animals, rivers, etc. have always delighted man since times immemorial. As a result, he tried to bring their colours in his own life by using them in his environs and for self-decoration. This instinct of decoration finally led to the colouring of objects used by man in his daily routine.

Colour is a matter of perception and subjective interpretation, which differs from person to person according to his psychology. Colour makes an object appealing and attractive and gives pleasure to its observer. Besides enhancing the outlook of an object colour also serves as a mode for expression of emotions and ideas. The use of colour by man can be traced from stone age through ancient civilizations to the present silicon age in the form of paintings, sculptures, self-decoration and dyeing of textiles. An interesting noteworthy peculiarity in history of colouring is that all the colours were derived from natural sources. The art of dyeing of textiles dates back to pre-historical times. Kermes, henna, saffron and pomegranate are a few of the dyes which were used even before 1500 B.C. in ancient civilization. Evidence supports that in India dyeing of textiles was carried

out since 3500 B.C. during the time of Indus civilization. Besides, the natural dyes in red, yellow, blue, black and white are mentioned in various relics and other books of India (Singh 1985).

This art of using natural dyes from plant, animal or mineral origin continued all over the world until synthesis of the first synthetic dye 'Mauve' by W.H. Perkin in 1856. Ever since the development of first synthetic dye, there is no looking back in this sector. Very soon the use of synthetic dyes brought an abrupt end to the use of natural dyes on large scale due to certain merits of synthetic dyes like availability of bright and varied colours, low cost and ease in their application. Besides, the unique craft of natural dyeing suffered a set back due to absence of systematic records of technical knowledge of extracting and dyeing techniques by the ancient dyers as this craft passed down from one generation to the other only by word of mouth. Also, the natural dyeing being lengthy and tedious lost interest of younger generation.

At present there are many classes of synthetic dyes for each type of fibre available today. However, the excessive use of synthetic dyes has now started proving hazardous from ecological point of view. Some of these dyes themselves and several chemicals used as an aid in their application are creating both environmental pollution as well as health hazards.

With the increasing emphasis now being given to ecology, textile processing sector in general and textile dyeing sector in particular has been forced to adopt safer and green technology. The United Nations Organization organized a conference on environment and development on June 14-15, 1992, at Reo de Janerio popularly called as 'Earth Summit'. In this conference heads of the governments from all over the world agreed that there is direct interrelation between earth environment and human life. Hence, a declaration 'Rio declaration' containing twenty-one points was passed in order to pursue the use of eco-friendly technologies, which in turn will lead to sustainable development. As a result, various eco-bans have come into force which, in turn, has established eco-standards for various harmful chemicals to restrict their usage to the minimum or even to prohibit their use for certain processes. Forced by this on-going global eco-movement, green fashion has become a priority of the consumers. A large number of projects have been initiated in government and private sector organizations to strengthen research on processes for dyeing with natural dyes. Under these projects many new sources of natural dyes are under trial besides various ingredients used in the kitchen (Singh 2000a).

In efforts to create green products, the producers are now adopting natural dyes also as an option and this step has initiated a period of renaissance in use of natural dyes for colouring textiles. The use of natural

dyes in cottage industries has once again shown an upswing due to higher demand of green products in international market. Also the natural dyes have found way in the value addition of traditional textiles through dyeing and printing (Singh 1999).

The synthetic dyes are produced from petrochemicals that make use of high temperature, toxic solvents and various other chemicals. On the other hand, natural dyes can serve as ecological alternative if plants which are renewable sources are cultivated for specific purpose of colour production. The natural dyes now a day are produced not only from plants, animals and minerals but also from microbes, which can be cultured easily, on a small scale in rural areas, without exerting any undue pressure on land. Natural products are not always the best choice. This is also true for natural dyes since the present requirement of natural dye is 10,000 tons which is equivalent to 1 per cent of world synthetic dye consumption. Thus the adoption of natural dye as viable alternative will lead to shift in usage of land globally which is already scarce (Gulrajani 2001). So complete substitution of synthetic dyes by natural ones is both undesirable as well as impossible (Chavan 1995). Besides these, other drawbacks of natural dyes which restrict their use as substitute for synthetic dyes, are high cost, poor colour fastness, limited shade range, difficulty in reproduction and lack of eco toxicity profiles (Glover 1995). Also most of natural dyes are not

substantive in nature and require assistance of mordants to get adhered to fibers (Singh 2000b). In ancient times also, the fastness of natural dyes was enhanced by use of natural mordants as urine, saliva, egg albumin and tannins (Robinson 1969). But the mordants used today include mainly the oils, tannic acids and metallic salts which results in severe effluent problems if not controlled properly.

Since the beginning of the civilized world, the man's quest for comfortable clothing is going on. For centuries until today, the mankind has used the natural fibers like cotton, wool and silk for clothing. For further improving the clothing standards of masses at low cost, it was considered better to switch over to production of man-made fibres. This, on one hand, will reduce the pressure on land for production of fibers and, on the other hand, food needs of growing population will be met more satisfactorily.

Initially man-made fibers, e.g. rayons were produced from available fibrous polymer sources, such as cellulose. Later on the production of fibers, such as nylon, polyester and acrylic, started using basic chemicals. (Talele and Gandhi 1993). Polyester was invented during years of World War II, i.e. 1941, by J.R. Whinfield and J.T. Dixon. Its commercial production started in 1950 by ICI in the UK and Dupont in the USA under trade names terylene and dacron, respectively. In India, its production

started in 1965 under trade name, terene. Polyester showed rapid growth initially and overtook nylon in 1972 (Gupta 1995). In 1980, polyester production had share of 50 per cent in total production of synthetic fibers. Also by this period, the production shifted from developed to developing countries due to high labour cost in the former countries (Sharma and Hora 1995). In 1996, the annual worldwide polyester fibre usage was 27.2 per cent of total fibre production of 47 million tones, next only to that of celluloses, which was 44.7 per cent (Park and Shore 2000).

The basic repeat unit of polyester confers on the resulting semi-crystalline oriented fibre a number of desirable features which make it suitable for a wide range of applications. It is one of the cheapest synthetic fibers to be produced in bulk, which is one of the major reasons of its rapid growth besides the high cost of cotton (Narayanaswamy 1996). Today it is the most popular synthetic polymer for fibre production. Its uses range from apparel and home furnishings to industrial fabrics (Perkins 1996). This versatility in the use of polyester is due to its characteristics like durability, dimensional stability and ease of care although it also suffers from certain drawbacks as static charge accumulation, hydrophobicity, pilling and low moisture regain (Mittal and Bhatt 1983). Despite these disadvantages, polyester is termed as the fibre of the century because of the fact that the inherent drawbacks in it can be corrected to a great extent by

carrying out modifications at spinning stage, by adding certain chemicals in polymer melt and by changing fibre morphology or by blending fibers with other natural or man-made fibres according to end use (Cama 1987). More than 50 per cent of polyester fibres are, therefore, used in the form of blends (Achwal 1994).

The disperse dyes based on azo and anthroquinone are used to dye polyester fibers with the help of various auxiliaries like dispersing agents, levelling agents, carrier, etc. These auxiliaries create a large effluent problem in polyester dyeing units. Also, many of disperse dyes come under the list of dyes banned by Germany and some other European countries. The use of natural dyes for dyeing polyester as an eco-alternative to disperse dyes has been reported only by few workers.

Hence, an attempt has been made in the present study to investigate dyeing of polyester with natural dyes with the following specific objectives:

1. To optimize processes of dyeing polyester with natural dyes by examining the effect of different parameters of dyeing on dyeability of polyester.
2. To investigate the effects of synthetic mordants on dyeing.
3. To evaluate the colour fastness properties of dyed polyester.

## **CHAPTER II**

### **REVIEW OF LITERATURE**

The review of literature revealed that several research studies had been conducted on natural dyes both abroad and in India. Besides research work on dyes extracted from plant, animal and mineral origins, efforts are now also directed to isolate colours from microbes such as fungi and bacteria.

The reviewed literature is presented under following heads :

- 2.1 Mechanism of dyeing synthetic fibres with natural dyes
- 2.2 Dyeing of polyester
- 2.3 Dyeing of polyester with natural dyes
- 2.4 Dyeing of other synthetic fibres with natural dyes
- 2.5 Dyeing of natural fibres with natural dyes used in study
- 2.6 Dyeing of natural fibres with natural dyes
- 2.7 Dyeing with natural dyes from microbes

## 2.1 Mechanism of dyeing synthetic fibres with natural dyes

Gupta (2000) carried out thermodynamic and kinetic studies using dye fibre combinations to see the effect of various isomers present in dye molecules on the dyeing mechanism. The dyeing process can be evaluated by investigating the equilibrium dye distribution between the dye bath (D)<sub>s</sub> and the substrate (D)<sub>f</sub> which could be presented graphically by an isotherm. The important ones could be expressed as-

$$\text{Nernst} \quad : (D)_f = K(D)_s$$

$$\text{Freundlich} \quad : \text{Log } (D)_f = \log K + x \log (D)_s$$

$$\text{Langmuir} \quad : \quad \frac{(D)_f}{(S) - (D)_s} = K(D)_s$$

$$\text{where} \quad K = \text{constant}$$

$$(S) = \text{potential dye sites per kg of fibre.}$$

Linear relationships were obtained with disperse dyes on hydrophobic fibres like acetate, polyester and polyamide. Langmuir isotherms had been obtained with anionic dyes on wool and on polyamide where (S) corresponded in many cases very accurately to the amino group content of these fibres. The thermodynamic parameters taken into consideration are standard affinity ( $-\Delta\mu$ ), enthalpy or heat of dyeing ( $\Delta H$ ) and entropy of dyeing ( $\Delta S$ ). For polyester dyed with lawsone and juglone,

the values of standard affinity were reported to be high indicating a high affinity of natural dyes for polyester and as a result of this saturation dye uptake was found to be very high. The dark shades at higher concentration resulted because of aggregation of dye molecules in the fibre or due to some modification of dye molecule during the process of dyeing. In the case of dyeing of nylon, the isotherm observed was Nernst isotherm similar to one observed for polyester for the dyes which were used on polyester. Further with annatto, which has linear ionic molecule, more than one mechanism of sorption were observed. However, Langmuir mechanism was predominant, though some dye may be getting sorped as disperse dye. In the case of acrylic fibre dyed with berberine, a cationic dye in nature, the dyeing mechanism corresponded to the Langmuir sorption confirming the adherence of the dye to the fibre with formation of ionic bonds between the negatively charged sites on the fibre and the positively charged ones on the dye. The heat or enthalpy of dyeing ( $\Delta H$ ) for all dye – fibre systems studied was found to be positive i.e. the dyeing of synthetic fibres with natural dyes was endothermic in nature. So the dye uptake increased with the rise in dyeing temperature.

Gulrajani (2000) specified that the technology for production of natural dyes can vary from simple aqueous to complicated solvent system and presently to sophisticated supercritical fluid extraction techniques

depending on the product and the purity to be achieved. Purification may require filtration or reverse osmosis or preparatory HPLC and drying of the product may be spray or under vacuum or by a freeze drying technique. The natural dye sector can further draw benefits from the catalytic ability of enzymes to hydrolyse the dye-cellulose bonds as in the case in production of indigo or microbes can be used to generate anthraquinone based colorants the way *Lusiffer lacca* produces the lac dye or biotechnological methods can be used to increase yield of colorants in plants.

## **2.2 Dyeing of polyester**

### **2.2.1 Dyes used for dyeing polyester**

Giles (1974) reported that the normal polyester fibres could be dyed only with disperse dyes for obtaining best results. Besides, it could be dyed with certain unreduced vat dyes which behaved as disperse dyes and with certain azoic dyes. The azoic dyeings can be produced on polyester fibres by applying the diazo component and the coupling component to the fibre by a dyeing process and then simultaneously diazotising the diazo component and coupling it with the coupling component *in situ*. Azoic blacks were the most important azoic combinations on polyester fibres. Recent advances have led to development of polyester fibres having acidic group and thus it can be dyed with basic dyes also.

Trotman (1994) reported that the exhaustion of disperse dyes was quite good but the diffusion into the fibre was extremely slow and required longer time period to reach equilibrium.

Leaver *et al* (1992) reviewed the commercial development of new disperse dyes for 100 per cent polyester and polyester/cellulose blends. The traditional anthraquinone disperse dyes for bright red and blue shades have lower wetfastness. The new disperse dyes based on benzo-difuranone chemistry and thiophene based azo dyes have benefits over existing dyes. The outstanding levels of wet fastness of new disperse dyes will reduce the pollutant from dye house effluent.

Gulrajani (2001) stated that the chemical structures of many natural dyes are similar to those of synthetic disperse dyes in the sense that they do not have solubilizing groups and are only sparingly soluble in water. Hence, they can be dyed on hydrophobic fibres, such as polyester and nylon.

### **2.2.2 Mechanism of dyeing polyester**

Pajgrt and Reichstadter (1979) reported that the mechanism involved in dyeing polyester was as follows :

- (a) when the temperature of dyebath is increased, the dispersed dye particles in the liquor are gradually dissolved

- (b) the dissolved particles migrate towards the fibre liquor interface and are absorbed by the fibre surface
- (c) the absorbed dye particles diffuse into the fibre.

Of these steps, the dye diffusion is the slowest and it determines, therefore, the equilibrium and dyeing conditions. The limited dye diffusion into the fibre is due to relative sizes of dye molecules and pores in the fibre on one hand and the bonds between macromolecular chains in the fibre on the other.

The dye diffusion can be facilitated into fibre by:

- (a) proper selection of dye whose molecules get into fibre easily
- (b) overcoming the energy barrier by either increasing temperature or by addition of chemical agents capable of assisting the dye diffusion into the fibre even at temperatures not exceeding 100°C.

Giles (1974) reported that the dyeing of natural fibres is generally done at the boil at atmospheric pressure in open or closed vessels or machines for an aqueous environment at 90-95°C. When polyester fabrics were introduced these open dyeing machines could not be used for dyeing these fabrics, which do not get swollen by water at 100°C. The pore size of the unswollen polyester fibres is so small that disperse dye molecules

cannot diffuse into the fibre. So different methods of dyeing were adopted as given below-

### **2.2.2.1 Dyeing at 100°C (Carrier dyeing)**

Moncrief (1975) reported that for dyeing polyester at 100°C and atmospheric pressure, carriers are used which modify the structure of fibre and facilitate dyeing, increasing the rate of penetration of dyes into fibres with level dyeing.

#### **2.2.2.1.1 Functions of carriers**

Ashkenazi (1984) reported that the mechanism which underlies the function of the carrier within the dye liquor on the one hand and on the fibre on the other. The functions of the carrier within the liquor are – (i) the carrier and dyestuff form a complex compound which facilitates diffusion into the fibre (ii) carriers increase the solubility of the dyestuff in the liquor and this has the effect of intensifying the depth of the shade, a result of better dye penetration into the fibre. The carrier has the following functions within the fibre (i) carriers promote the swelling of the fibre, which facilitates the diffusion of the dyestuff into the fibres, (ii) carriers increase the water absorption capacity of the fibre which, in turn, has the effect on improving the dyestuff absorption capacity of the synthetic material, (iii) carriers form a co-liquid within the fibre thereby facilitating

the solution of the dyestuff and (iv) carriers have a tendency to relax the fibre structure.

Trotman (1994) stated that the soluble carrier enters into the fine structure of the polyester and pushes adjacent long-chain molecules apart. This loosens up the molecular pattern and facilitates the entry of the larger dyestuff molecules. While the water insoluble carriers appear to form a surface film on the fibre in which the disperse dye is highly soluble. The transfer of dye in such conditions, does not take place between aqueous phase and the fibre but between dyestuff dissolved in carrier and fibre.

Shenai (2000) reported that the carriers are believed to alter the dispersing properties of the dyes and the physical characteristics of the fibre so that more of dye can be transferred from the dye bath to the fibre. Carriers swell the fibre and causes relaxation. They may operate by opening up of the internal fibre structure and allow the dye molecules to diffuse more rapidly. They act as molecular lubricants, reducing inter molecular forces operating in the fibre thereby allowing the dye molecule to force its way in.

#### **2.2.2.2 High temperature high pressure dyeing**

Trotman (1994) stated that the use of carrier in dyeing bath causes effluent problem besides that of carrier residues left on dyed fabrics. HTHP dyeing method eliminates all these problems. The rise in temperature

causes molecules of polyester to move more freely and dyestuff can penetrate well into fibres to give a wide range of medium to deep shades in relatively short periods of dyeing. There is non-perceptible loss of elasticity or tensile strength when dyed under neutral or slight acidic conditions. The operation can be carried out only in an enclosed vessel. A temperature of 120°C, corresponding to an excess of steam pressure of about 15 lb/in<sup>2</sup> is suitable and higher temperatures (upto 130°C) may be attained in a high temperature dyeing machine.

### **2.2.2.3 Thermosol dyeing**

Shenai (2000) reported that the thermosol dyeing involves two stages in dyeing. The first stage consists of padding of polyester in a dispersion of dye, thickening agent and organic solvent followed by drying at 270°F with good air circulation. The second stage consists of passing the dye padded polyester through roller oven at 390-410°F for dissolving and penetration of dye into polyester followed by scouring to remove traces of loose dye and then drying. Shade dept and fastness properties are comparable to those obtained by HTHP dyeing. In this method, instead of dyeing in aqueous medium, dry heat is employed. Complete penetration of dye in the fibre can be achieved in one minute. It is a continuous process involving padding, drying, heat fixing and washing and excellent dye utilization (75-90%) is achieved. Not many auxiliaries are normally used in this method.

#### **2.2.2.4 Solvent dyeing**

Pajgrt and Reichstadter (1979) reported that dyeing in organic solvent brings great improvement in the dyeing of polyester with disperse dyes. Organic solvents considerably shorten the dyeing time, give outstanding level dyeing without any risk of stripiness and ensure perfect dye penetration. Polyester can be dyed using perchloroethylene as dyeing medium with disperse dyes at 110°C in enclosed dyeing machines under atmospheric pressure.

#### **2.2.2.5 Spun dyeing**

Press (1959) stated that the spun dyeing is an economical method which has the advantages of 100 per cent dye yield, completely safe dye house equipment and partial machinery savings in winding rooms and finishing plant. The other advantages are that the mechanical wear of the textile material is also greatly reduced and effluent water treatment is eliminated. Although the colour fastness is high, but the disadvantages are that it is impossible to produce small lot, there is less flexibility in production, there is difficulty in producing shades and machinery cost is high. Spun dyeing is achieved by adding the dyestuff, most often insoluble pigments, into the initial raw material or directly into the polymer melt before spinning. The outstanding colour fastness is due to the fact that the fibre mass protects the pigment against degradation due to external effects.

### **2.2.2.6 Supercritical fluid dyeing**

Achwal (1993) reported the use of supercritical CO<sub>2</sub> medium for dyeing polyester with disperse dyes. This innovative technology is of great interest from ecological point of view. The new process uses gas (supercritical CO<sub>2</sub> at 120°C temperature and 130 bar pressure in case of polyester dyeing) as medium replacing water and other solvents and the dyeing time can be shortened to 20 minutes. The dyed sample remains dry and dye powder remains at bottom the bottom of machine eliminating the need of after washing. Also the Tg of polyester lowers by 20 – 30°C, indicating a carrier effect of supercritical fluid. The selected dyes showed depth of shades comparable with aqueous dyeing at the same percentage of shade with excellent uniformity in dyeing. The fastness properties are quite comparable and no filtering effects of the dye aggregates by the fabric are expected, as occur in aqueous dyeing and thus the need of reduction clear treatment can be eliminated.

### **2.3 Dyeing of polyester with natural dyes**

Yukio and Kawabo (1986) reported colour reactions of onion dye with metal ions for dyeing of polyester, nylon, wool, silk, kapok and cotton.

Gupta and Gulrajani (1993) studied the dyeing of polyester along with five other fibres, namely wool, human hair, silk, nylon with juglone

(5-hydroxy 1, 4-naphthoquinone), a natural dye obtained from the hulls of raw walnut. The dyeing was carried out till equilibrium. The dye also exhibited a tendency to aggregate inside the fibre (characteristic of disperse dyes). The isotherms indicated the partition mechanism of dyeing corresponding to the solid-solution model observed in dyeing of hydrophobic fibres with disperse dyes. The temperature had less marked effect on the affinity of juglone for wool as compared to other fibres, i.e. in polyester and other fibres except wool, dye affinity increases with increase in temperature until equilibrium is reached. The polyester and nylon also showed higher affinity than rest of fibres so it can be inferred that juglone has a higher affinity for hydrophobic fibres as compared to hydrophilic fibres.

Rathi and Padhye (1994) investigated the effect of carrier method and heat-set method for application of natural dyes, namely madder, *chavalikodi*, *harda* flower, pomegranate rind and *haldi* on polyester fabric using alum, chrome and copper sulphate as mordants. They reported that the carrier method resulted in a brighter clear shades while heat-set method gave dull brown shades. The dye uptake was also high in the case of carrier method. The fastness ratings seemed to depend on type of mordant used rather than the dye. Copper sulphate had better efficiency, followed by alum and chrome. No clear trend was seen regarding which method gave

better light fastness for different mordants except copper sulphate where fabric dyed with heat-set method showed good (4-5) light fastness. The wash fastness ranged from poor (1) to good (4-5). Again copper sulphate resulted in higher wash fastness ratings followed by chrome and alum and no specific trend was seen regarding wash fastness rating, for two methods. Both the methods resulted in good (3-4) rubbing fastness with negligible difference between dry and wet rubbing fastness. The effect of acidic and alkaline perspiration was reported to be insignificant on the two methods of dyeing except in the case of yellow dyes applied by heat-set method which showed an increase in depth of colour.

Achwal (1998) reported the results of the experiments carried out for dyeing polyester with tea, madder, logwood, cochineal, & henna. A light staining was seen in the case of tea and madder. The results did not improve even with pre-mordanting. A little deeper dye was obtained in the case of logwood having the light fastness rating 3, wash fastness rating 2 and good perspiration and rubbing fastness. The depth of shade and fastness improved with high temperature dyeing (120°C).

Lokhande *et al* (1998) studied the effect of dyeing polyester with natural dyes, namely onion, lac and turmeric using various mordants like alum, copper sulphate, ferrous sulphate, stannous chloride, tannic acid, *harda* powder and aluminium sulphate by employing open bath and high

temperature and high pressure techniques of dyeing. The high temperature and high pressure technique showed good results as compared to open bath technique. In the case of onion, tin and iron mordants showed total colour difference in comparison to standard dyed sample whereas the other mordants showed very little colour difference. The light fastness was improved upto 3-4 on mordanting with copper and iron. Washing and sublimation fastness was reported good for all the mordanted and unmordanted dyed samples. The mordanting did not show any increase in colour value but added variety in shades. In the case of lac dye, mordanting was found to be necessary for dyeing polyester with lac dye. The light fastness was good (3-4) only in the case of copper and iron mordanted fabric. The wash and sublimation fastness of all the dyed fabrics were very good. Again high temperature high pressure dyeing of polyester gave better results. In the case of open bath dyeing with turmeric, mordanting with iron resulted in high colour difference as compared to other mordants which showed only marginal colour difference while in case of HTHP dyeing. Mordanting with tannic acid and tin showed marginal increase in the colour value. Light fastness was not reported to be satisfactory, whereas wash and sublimation fastness were very good.

Gulrajani *et al* (1999a) dyed polyester with carotenoid dye bixin-the colour component of annatto. It was observed that polyester had good

affinity for the dye but the rate of dye uptake was somewhat lower due to compact structure of polyester fibre and the lower temperature of dyeing which were not the optimum temperature of dyeing polyester. The mechanism of dyeing polyester corresponded to the partition mechanism which was observed in dyeing of hydrophobic fibres with disperse dyes. The light fastness was reported to be 2-3 but improved with increasing depth while wash fastness was moderate to good. The poor light fastness was due to photo-oxidation of conjugated double bond chains present in annatto colour dye.

Gulrajani *et al* (1999c) dyed polyester fibres with a natural dye extracted from ratanjot (*Onosma echoides*). It was found that the dye had good affinity for polyester. The colour obtained was deep in colour and dyeing corresponded to the partition mechanism, characteristic of dyeing polyester with disperse dyes. The fastness of dye to both light and washing was reported to be very good at 5-6 and 4, respectively.

#### **2.4 Dyeing of other synthetic fibres**

Kashino *et al* (1986) studied the dye affinity of cochineal for different fibres. For cochineal, a high degree of affinity was shown by nylon fibre while lower or no affinity was shown by fibres other than wool and silk. The effects of dyeing time, temperature, mordant concentration on

the exhaustion degree of cochineal for different fibres was also investigated.

Pasculescu *et al* (1986) studied dyeing of nylon with vegetable dyes. The dyeing properties of some plants, indigenous to Romania were discussed and the advantages and disadvantages of the dyes in dyeing of nylon carpet yarns were reported.

Kashino *et al* (1990) studied the effect of tannic acid on the dyeing with cochineal dye. Tannic acid pretreatment of the cellulose fibres was carried out to improve a low degree of affinity of cochineal. Chromaticity and colour fastness of dyed fabrics were investigated. The chromaticity of dyed fabrics by the tannic acid fixation changed with increase in fixed amount of tannic acid. The extent of the change in chromaticity of the dyed fabrics was the greatest in nylon fabric and the marked change took place when the fixed amount of tannic acid was greater than about 30 mg per gram of the fibre. Dyed fabrics treated with tannic acid showed higher light colour fastness than those untreated fabrics.

Badri and Burkinshaw (1993) dyed nylon and wool with henna and lawsone. It was found that the dye uptake increased with decreasing pH. After chroming improved both the wash and light fastness of henna extract and lawsone on each substrate and also altered the depth, chroma and colour of dyeings. Comparative dyeings obtained using lawsone suggested

that the sample of henna used in the work contain luteolin (a flavonoid colorant).

Gupta and Gulrajani (1994) studied kinetics and thermodynamics of the natural dye lawsone (2-hydroxy-1, 4-naphthalquinone) on six substrates at various temperatures. All the substrates showed high dye uptake and were dyed to deep shades.

Rathi and Singhee (1994) dyed 100 per cent acrylic, 100 per cent wool and 70/30 acrylic/wool blend with *chavalikodi* and red sandalwood using alum, copper sulphate and stannous chloride following three mordanting methods, namely pre, post and simultaneous mordanting. The colour from *chavalikodi* dye ranged from yellow to brown and pink except with  $\text{Cu SO}_4$  on wool which gave green colour while the colour from red sandalwood dye ranged from yellow brown to dull orange in all fibres except for wool with  $\text{CuSO}_4$  which again gave green colour. The simultaneous mordanting method had no showed best results on wool and acrylic/wool blend with both dyes while mordanting method has no effect on dye uptake in acrylic. The light fastness of red sandalwood (1-2 to 5) dyed samples was poor as compared to *chavalikodi* (1-2 to 8). Acrylic showed maximum light fastness when pre mordanted with  $\text{CuSO}_4$  and dyed with *chavalikodi*. The wash fastness of *chavalikodi* dyed samples ranged from 1-2 to 5 and that of red sandalwood dyed samples ranged from 1-2 to

4 while rubbing fastness with respect to change in colour rated 4 to 5 and 5 for both the dyes. Perspiration fastness for all the samples was between 4-5.

Ryder (1994) studied dyeing of nylon with henna which gave a yellow hue. Application could be done with an alum mordant or by the after-chrome process to give improved light and washing fastness.

Bruckner *et al* (1997) applied extracts containing natural dyes representing several chemical classes within the range of 50-500 per cent (o.w.f.) to obtain dyeing on nylon, acrylic and wool fabrics. Besides traditional dyes like cochineal, henna, logwood, madder, saffron and turmeric other plant extracts included were beetroot, bilberry, parsley, stinging nettle and tea. Alum, alum/tartar emetic and cupric sulphate were used selectively as mordants. It was found that only pastel colours were obtained in most of the cases and ratings of fastness to light, washing, perspiration and rubbing were moderate to poor in general.

Wakida *et al* (1998) studied the effect of low temperature plasma treatment on colour of wool and nylon – 6 fabrics dyed with natural dyes. The wool and nylon – 6 fabrics treated with oxygen, carbon tetrafluoride and ammonia low temperature plasmas were dyed with several natural dyes as cochineal, chinese cork tree, madder and groomwell dyes. The plasma treatment increased rate of dyeing with cochineal, chinese cork tree and

madder because these are water soluble dyes but not with groomwell which is less water soluble. In case of wool fabrics, plasma treatment also increased brightness of dyed fabric. However, plasma treatment had no affect on the dyeing behaviour of natural dyes on nylon-6 fabric.

Gulrajani *et al* (1999a) dyed nylon fibres with carotenoid dye bixin – the colour component of annatto to study its mechanism of dyeing. It was found that the dye has high affinity for nylon as well as high rate of dye uptake and diffusion. The colour obtained on nylon was reported to be fast to washing but not so fast to light.

Gulrajani *et al* (1999b) dyed acrylic fibres with berberine, (a yellow dye extracted from the roots of *Berberis aristate*) to study its mechanism of dyeing. It was found that berberine dye had high affinity for acrylic fibres and was capable of dyeing in bright yellow shades. The dye on acrylic fibres exhibited moderate to good wash fastness but poor light fastness due to presence of a non-localised positive charge which resonates in the structure of dye.

Gulrajani *et al* (1999c) dyed nylon with a natural dye extracted from ratanjot (*Onosma echoides*). The dye was found to have good affinity for nylon so rate of dye uptake was also higher and the colour obtained was deep in shade. The fastness of dye to light and washing were reported to be 5-6 and 4, respectively.

Lokhande and Dorugade (1999) dyed nylon fabric with three natural dyes derived from onion, lac, turmeric using seven mordants, namely alum, copper sulphate, ferrous sulphate, stannous chloride, tannic acid, *harda* and aluminium sulphate by employing open bath and HTHP dyeing method. It was found that HTHP gave better results as compared to the open bath dyeing. The wash fastness rating of 4 was obtained in all the three natural dyes. The light fastness rating was higher (4-5) in the case of onion and lac as compared to fastness rating of turmeric (3-4).

Patel and Agarwal (2000) extracted the dye from babool bark and applied on nylon mordanted with ferrous sulphate, copper sulphate and stannous chloride following open bath and HTHP dyeing methods. Pre-mordanting and meta-mordanting techniques were used for application of mordants. HTHP dyeing method showed higher dye uptake as compared to exhaust dyeing method on nylon. It was found that the colour strength increased with the increase in concentration of dye for both control and mordanted samples. Among, different mordants, combination of ferrous sulphate and copper sulphate showed better performance. HTHP resulted in slight variation in tone and hue of dyed nylon as compared to open bath dyeing which resulted in much variation in tone and hue with different mordants. The fastness to washing, rubbing and light of nylon dyed with

open bath as well as HTHP dyeing methods was reported to be good to excellent for both control as well as mordanted samples.

Gulrajani *et al* (2001) investigated the colour value and fastness of the nylon dyed with three natural dyes, namely annatto, ratanjot and berberine employing exhaust dyeing method. The optimum pH for these three dyes was found to be 9, 6 and 4, respectively. The K/S values of all dyed samples increased with increase in dye concentration for three dyes. With the three dyes, the wash fastness was found to be very good (4/5-5), good (4) and (1 – 2) poor, respectively. In case of light fastness, berberine was found to have rating of 1 (poor) followed by ratanjot for which increase in ratings from 1-3 was seen with increase in dye concentration and annatto dye which exhibited moderate fastness to light with a rating of 3.

## **2.5 Dyeing of natural fibres with natural dyes used in study**

Torimoto (1986) reported the use of onion cuticle along with indigo to obtain a green colour on cotton and also studied its fastness properties.

Malhotra (1989) conducted a study on dyeing wool with madder roots (*Rubia tinctorium*), Indian kamala (*Mallotus phillipinensis*), tessu flower (*Butea monosperma*), corel jasmine flowers (*Nyctanthes arbortritis*), turmeric, pomegranate rind, henna, onion, tea and catch. The

dyeing conditions for each dye were optimized and fastness properties were assessed.

Agarwal *et al* (1992) optimized the concentration of dye material, extraction time, dyeing time, concentration of mordants, mode of mordanting, mordanting time and combination of mordants for dyeing wool with turmeric dye using alum, chrome, iron and tin as mordant. A wide range of colours was obtained by the use of different mordants and their combinations. The light fastness of unmordanted sample was reported poor (2) which improved to fairly good (3-4) with use of mordants and their combination. The samples mordanted with alum and stannous chloride had good (4-5) light fastness. The wash fastness ranged from fair to good (3-4) for most of mordant combinations while those mordanted with chrome exhibited good wash fastness.

Das (1992) reported the use of onion skins for dyeing silk using alum, chrome, iron and tin as mordants resulting in production of bright golden yellow, deeper gold, greenish brown and brassy yellow colours in dyed samples, respectively.

Gulrajani *et al* (1992a) dyed wool and silk with turmeric dye, extracted by boiling for 45 minutes, in a dyebath for 45 minutes keeping material to liquor ratio of 1:40 using alum, chrome, copper sulphate, ferrous sulphate, stannous chloride, *harda* and tannic acid as mordants. The

light fastness of both wool and silk samples was poor while wash fastness was fair on wool and good on silk. The mordanting process seemed to have no effect on the fastness characteristics of turmeric dye.

Gulrajani *et al* (1992b) reported the dyeing of wool and silk with onion dye for 45 minutes at boil keeping M:L ratio 1:40 using alum, copper sulphate, ferrous sulphate, chrome, stannous chloride, *harda* and tannic acid as mordants. The colour obtained varied for different mordants on both wool and silk. On wool, unmordanted sample was of clay colour and on silk it was of fawn color. The difference in colours of samples using different mordants indicated the capability of dye for forming dye-metal complexes. The fastness ratings showed the average light fastness rating on wool and silk (3-5) while the wash fastness was reported good with very little staining.

Burde and Mehra (1994) dyed plain bleached pine apple leaf fibres with *haldi* and red sandalwood using natural and synthetic mordants and employing pre, simultaneous and post-mordanting. They found that *haldi* gave colours in range of yellow to brown while red sandalwood gave colours in pink to brown and red. The light fastness with natural and chemical mordants ranged from poor to good (3 – 5) in *haldi* and red sandalwood both. The wash fastness of *haldi* dyed samples ranged from 3

to 5 whereas in red sandalwood dyed samples no change or slight change was observed (5 to 4) with no staining on adjacent white cotton fabric.

Ouron (1994) investigated the dyeing of wool fibres with the bark of cluster pine in different conditions using different mordants and types of mordanting at varying pH and temperature and determined colorimetric and fastness values for resulting dyed fibres.

Rathi and Facho (1994) dyed two varieties of coir, Angengo and Vycome with *haldi* using alum and ferrous sulphate as mordants by pre, simultaneous and post-mordanting methods and reported colours in the range of golden yellow to brown. The dye uptake of both the varieties of coir was similar irrespective of the method of application, concentration of dye and concentration of mordants.

Teli *et al* (1994a) studied the effect of ten mordants and their combination on the dye colour and fastness properties of cotton dyed with turmeric. The use of mordants and their combinations resulted in variation in depth of shade, as well as change in hue and shade. The light fastness of turmeric, which is poor, improved slightly with the use of mordants and best light fastness was found of samples treated with copper sulphate and ferrous sulphate. The rubbing fastness was reported to be good (4-5) and dry rubbing was better than wet rubbing. The washing fastness was also reported to be very good (4).

Dhillon (1995) conducted a study on the effect of different vegetable dyes – onion skin, turmeric, *tessu* flowers, henna, and *harda* on wool. Synthetic mordants were used to obtain a variety of shades. These dyes were fairly fast to washing but possess poor resistance to sunlight. These dyes gave harmonizing colours, rare colour ideas and impart moth resistance property to wool.

Gogoi (1998) studied the effect of dyeing eri silk using alum, chrome, ferrous sulphate and stannous chloride as mordants and reported bright yellow, olive green, brownish green and bright orange colour of dyed samples with each mordant, respectively. The colour fastness to washing, rubbing and light improved with the use of mordants as indicated by the ratings given by maximum number of respondents in favour of negligible change in colour and negligible staining.

Lotika (1998) conducted a study on dyeing angora wool with natural dyes extracted from beefwood, black myrobolan, flame of forest, golden drop and Indian gooseberry. The dyeing processes were optimized and fastness properties were tested.

Deo and Paul (2000) dyed ecru denim samples with onion extract using natural mordants such as *harda*, tartaric acid and tannic acid separately and in combinations. They found that there was an overall improvement in performance properties of samples dyed with various

combinations over that of the single mordanted and the control samples. The dyed samples exhibited fair to good washing, rubbing and light fastness in pre, post and simultaneous mordanted samples.

Shah *et al* (2000) studied the dyeing behaviour of catechu and turmeric dyes on wool with respect to dye exhaustion and fastness characteristics. In both the dyes, as the percentage shade increased, dye exhaustion values also increased and the values were between 30 to 50 percent. Except light fastness, the results of catechu were encouraging while for turmeric dye light, washing and rubbing fastness properties were inferior.

Dayal *et al* (2001) extracted dye from bark and needles of *Pinus roxburghii* and applied to silk, wool and cotton using alum, copper sulphate, potassium dichromate and stannous chloride as mordants, employing exhaust dyeing and post mordanting method followed by fixing with sodium carbonate solution. A range of colour from yellow to brown and peach was obtained with different mordants and in different extraction medias. The wool and silk exhibited moderate (3-4) to very good (5) wash fastness; 2-4 ratings for light fastness and very good (4-5) perspiration fastness except for rubbing fastness for which dyed silk rated very good (3-4 to 4-5) and wool rated 2 to 3. In case of fastness of dyed cotton fabric light fastness was found to 1-2 to 4; washing fastness 1-2 to 3-4,

perspiration fastness 3 to 3-4 and rubbing fastness 2 to 4 for different mordants.

## **2.6 Dyeing of natural fibres with other natural dyes**

### **2.6.1 Dyeing of cellulosic fibres**

#### **2.6.1.1 Dyeing of cotton**

##### **2.6.1.1.1 Dyeing with vegetable dyes**

Gupta (1990) studied substitute of *harda* as mordant. *Harda* which contains tannic acid, has traditionally been used in India as a vegetable mordant in the dyeing of cotton with natural dyes. Pomegranate rind which was cheaper than *harda* could be used as a substitute mordant giving comparable or even deeper shades and equal fastness properties.

Padhya and Rathi (1990) studied the effect of mordants on dyeing of cotton with vegetable dyes. The effect of six different mordants was studied. Best results were obtained with potassium dichromate and worst with alum. Although the wash fastness was moderate to poor in all the cases. The fabrics should , therefore, be washed under mild condition or drycleaned.

Subramaniam (1994) reported dyeing of cotton yarn in twenty different shades of natural dyes. Vegetable soap materials were also used for scouring and bleaching to produce 100 per cent organically

manufactured yarns. Vegetable dyeing took 2-4 times longer duration than conventional manmade dyestuff dyeing and were not wash fast but several yellow colours showed promising results.

Teli *et al* (1994b) studied the effect of various mordants and their combinations on the cotton dyed with catechu. It was found that the light fastness of the dyed sample was good to better (5-6); wash fastness was very good (4); wet rubbing fastness was poor and dry rubbing fastness was excellent which reduced on application of mordants and their combinations. Pre-treatment of tannic acid followed by mordanting with copper sulphate, all the fastness were improved while with alum, citric and ferrous sulphate the different fastness remained almost same as stated earlier.

Etters (1996) studied recent progress in indigo dyeing of cotton denim yarn. It was observed that dye uptake by cotton fibre and the extent of penetration of indigo dye into denim yarn cross section were influenced strongly by the pH-controlled ionic species of both dye and cotton in the dye bath. High dyebath pH resulted in high level of ionization of both indigo dye and cotton cellulose resulting in decreased substantivity and increased penetration of the yarn, fibre bundle by the dye with associated lower colour yield. On the other hand, moderate dyebath pH promoted lower ionization of both indigo dye and the fibre resulting in increased substantivity and thus greater colour yield and a denim garment that

washed down quickly and consistently to produce the current appearance demanded by fashion.

Kalyani and Jacob (1998) used red gulmohar flower extract to dye cotton fabric using synthetic mordants and found gulmohar flowers showed good green, black and brown shades of colours with optimum concentration of copper sulphate and potassium dichromate with aqueous dye extract and ferrous sulphate and cobalt sulphate with alkaline dye extracts. The dyed cotton exhibited fair to excellent dry rubbing fastness; poor to good wet rubbing and washing fastness; poor to excellent perspiration fastness and good to excellent light fastness.

Katyayini and Jacob (1998) optimized the dyeing conditions for mesta calyx dye which was applied on cotton to produce pink, pinkish brown and violet colours with alum, copper sulphate and stannous chloride respectively. The unwashed samples exhibited fair to good rubbing fastness and fair light fastness while washed samples exhibited fair to good fastness to rubbing, fair resistance to washing and poor resistance to sunlight.

Kumar and Bharti (1998a) reported the use of *Eucalyptus* hybrid bark for dyeing cotton which produced bright brown colour. The dye exhibited medium wash and light fastness on cotton. It was found that the fastness to washing and light increased by the use of metal salts or tannic acid whose application also resulted in production of wide range of shades.

Kumar and Bharati (1998b) reported the production of bright brown dye from the *Mangifera indica* bark which exhibited medium wash and light fastness on cotton. The wash and light fastness increased by the use of metal salts or tannic acid. A wide range of shades were obtained by the use of metal salts as mordants.

Samy and Raja (1998) dyed cotton and silk fabrics with the extract from arecanut without mordants and assessed for fastness properties. It was concluded that the natural dye gave a range of shades from beige to black with satisfactory fastness properties on both fabrics.

Singh *et al* (2000) studied the effect of *Jatropha integrima* on physical properties of cotton fabric. They reported that tensile strength in warp direction and flexural rigidity increased while tensile strength decreased in weft direction and elongation percent decreased in both warp and weft direction. Tin and iron with pre mordanting method had no adverse effect on physical properties of dyed fabric rather they improved properties when compared to control fabric.

Gaba *et al* (2001) studied the effect of *Thevetia peruviana* on physical properties of cotton fabric. The dyed fabric had good appearance, lusture and texture but showed increase in tensile strength, elongation and flexural rigidity in warp direction and decrease in tensile strength and elongation in weft direction.

#### **2.6.1.1.2 Dyeing with animal dyes**

Saxena *et al* (1997) standardized technique of dyeing cotton fabric with lac. Chitosan, a naturally occurring polymer was used for pretreatment before dyeing. The dye produced violet shade on cotton fabric when dyed in acidic medium. The dyed fabric had medium light fastness, good rubbing and perspiration fastness and poor wash fastness which can be improved either by a cross-linking treatment or by use of mordants.

#### **2.6.1.2 Dyeing of jute**

Sayeed *et al* (1990) dyed jute fibres with catechu tannic acid and catechin using various synthetic mordants. Shades produced by catechu tannic acid along with mordanting and bleaching were even, attractive and fast to light, wash and urine. The shade of catechin dyed fibres was comparable to that of catechu tannic acid dyed fibre. Chromium sulphate and copper sulphate mordanted fibres gave better and more permanent shade. Bleaching has no better effect on colour fastness of jute fibre dyed with catechu tannic acid and catechin.

Islam and Islam (1992) reported that the jute fibres could be dyed grey by finely powdered myrobolan fruit in the presence of ferrous sulphate mordant or golden yellow without mordant. In case of pre-mordanting or post mordanting, there was little effect on the colour value

due to washing, light, perspiration and rubbing while the fibre faded or stained significantly when simultaneous mordanting is carried out.

Chattopadhyay (1997) studied the effect of mordants and mordanting techniques on the dyeing of jute and cotton fabric with natural dye. With jute, simultaneous mordanting was found better while in case of cotton, pre and post mordanting gave comparatively better results. With both jute and cotton, the colour depth was found higher with ferrous sulphate than with the alum.

Bhattacharya *et al* (1998) dyed bleached jute fibres with natural dyes in different colour and shades using *Acacia catechu*, *Onosma echiodes*, *Indigofera tinctoria*, *Artocarpus integrifolia*, *Adenanthera pavonina*, *Rubia cardifolia*, *terminalia chebula*, etc. using different synthetic mordants. All the samples showed good dye uptake and exhibited good light, washing and perspiration fastness.

### 2.6.1.3 Dyeing of cellulosic blends

Dedhia and Fernandes (1995) used extract of leaves of dwarf goldmohur peacock tree to dye 100 per cent jute, jute /cotton (75/25) and jute/viscose/cotton (53/22/25) fabrics using synthetic mordants. They found that the colour ranged from yellow green to yellow brown. Copper sulphate gave better results in terms of dye uptake. All the three fabrics exhibited poor light fastness Tannic acid gave best result for wash fastness which

was very good for all fabrics with jute showing least colour change followed by jute/cotton and jute/viscose/cotton.

Moses (1999) dyed jute/cotton fibres using natural colours such as turmeric, tamarind and manjistha with the help of synthetic mordants to get different colours which exhibited good washing as well as light fastness. The colours were obtained in shades of yellow, brown and grey, respectively with three dyes.

Moses (2000) investigated the application of different natural dyes, myrobolan, pomegranate, red creeper and parijatham as such or their application in conjunction with other natural dye or mordants on jute/cotton woven fabrics and reported production of different attractive colours which exhibited moderate to good washing and light fastness.

#### **2.6.1.4 Dyeing of other cellulosic fibres**

Bisht and Goel (1999) examined the effect of bleaching and mordanting on the colour fastness of the kilmora root dye on *bhimal* fibres. They found that evenness in colour application was in unbleached control and unbleached and bleached alum mordanted samples. The colour fastness to washing of chemical mordanted and dyed samples was found good while light fastness was rated good for the samples mordanted with natural mordants. A variety of shades were produced and fastness improved with mordanting.

## 2.6.2 Dyeing of silk

### 2.6.2.1 Dyeing with vegetable dyes

Gulrajani *et al* (1992c) conducted a study on natural yellow dyes with special reference to quinones, henna and dolu. Analysis of the naturally occurring quinones, henna and dolu (*Indian rhubarb*) was presented. The origins of the use of these substances as dyes were traced and the results obtained on silk with various mordants.

Singh *et al* (1993) studied fastness properties of silk dyed with madder dye using different mordants. The control silk sample exhibited good light fastness (4) and poor wash fastness (2). The rating for light and wash fastness remained almost same after mordanting. The best fastness was obtained with copper sulphate.

Katti *et al* (1996) dyed silk with combinations of natural dyes namely mango leaves, manjith and french marigold flower extracts. The fastness ratings of light, washing and perspiration were reported to be fair, good and very good, respectively. The fastness ratings improved on washing of dyed fabric with soap solution.

Singh *et al* (1996) studied dyeing of silk with dye extracted from fruits of kamala (*Mallotus philippensis*) and reported that the dyed samples

exhibited fair light fastness (3) and very good wash fastness (4-5). The difference in fastness was insignificant with use of different mordants.

Ahmed *et al* (1997) dyed mulberry silk fabric with kamala, indigo, turmeric and henna using indigenous dyeing methods. The dyes were reported to have even penetration into the fabric. After dyeing the breaking strength of the silk material increased in warp direction.

Gogoi *et al* (1997) applied kamala, indigo, turmeric and henna on silk fabric using indigenous dyeing methods which resulted in even dyeing of fabric. The dyed samples exhibited good wash fastness and rubbing fastness while light fastness was good in case of indigo dye only.

Venkidusamy and Arunkumar (1997) dyed silk with shoe flower extract using different mordants which result in production of variety of shade. The dye was reported to have excellent fastness to washing (5) and light (6-8).

Gogoi and Kalita (1999) studied the effect of mordants on the physical properties and colour fastness of silk fabric dyed with henna using alum as mordant. They found that the silk fabric mordanted with 3 per cent concentration of alum showed better results with respect to physical as well as colour fastness properties.

Devi *et al* (2001) reported production of dazzling orange shade on silk with annatto dye using synthetic mordants and different mordanting methods. The dyed samples exhibited good washing fastness; good to excellent dry rubbing fastness; good perspiration fastness and fair to good light fastness.

### **2.6.3 Dyeing of wool**

#### **2.6.3.1 Dyeing with vegetable dyes**

Garg *et al* (1991) investigated the effect of mordants on dyeing wool with natural dye extracted from *tessu* flowers (*Butea monosperma*). Best results were obtained with 25 per cent alum, 4 per cent chrome, 4 per cent iron and 4 per cent tin (o.w.f.) applied for one hour by pre-mordanting method. Chrome mordanted samples showed better fastness to light than other three mordants.

Jahan and Gupta (1991a) optimized the conditions of dyeing wool with balsam (*Impatiens balsamina*) flowers. It was found that the dye gave best results at concentration of five grams in 100 ml water, extracted for one hour and dyed for 2 hours. Simultaneous mordanting gave best results for all mordants except chrome which gave best results on pre-mordanting. Mordant concentrations were also optimized. Different shades of yellow, orange and brown were obtained. Light and wash fastness were good.

Jahan and Gupta (1991b) optimized the dyeing conditions for parijataka flower dye on Australian merino wool. Wool was dyed with the dye extracted from yellow tubes of flowers. Alum, chrome, ferrous sulphate, copper sulphate and stannous chloride were used as mordants in different concentration.

Kaur (1991) studied the effect of dyeing parameters on the fastness of natural dyes on wool. Dyeing processes for carrot leaves, coffee seeds, fenugreek seeds, spinach leaves and walnut bark on wool were optimized and fastness properties were tested.

Nicolai and Nachwatal (1991) studied the dependency of depth of dyeing and fastness of wool dyed with natural dyes, madder, safflower, logwood and weld after application of an aluminium mordant. Mordanting with aluminium sulphate was more favourable than alum.

Agarwal *et al* (1992) developed a suitable dyeing process for dyeing of wool with henna. Best results were obtained at 2g dye concentration extracted in 100 ml water for 120 minutes. Optimum concentration of mordants were 25, 6, 5 and 7 per cent (o.w.f.) for alum, chrome, ferrous sulphate and stannous chloride, respectively. Post-mordanting method gave best results with alum and chrome and simultaneous method was suitable for ferrous sulphate and stannous chloride.

Batra (1992) conducted a study on dyeing wool with flower dyes. The conditions of dyeing wool with coreopsis (*Coreopsis tinctoria*), dahlia (*Dahlia variabilis*), Indian laburnum (*Cassia fistula*), marigold (*Tagetes erecta*) and safflower (*Carthamus tinctorious*) were optimized and fastness properties were evaluated.

Gulrajani *et al* (1992d) conducted a study on dyeing wool with natural yellow dyes, namely *Terminalia chebula* and *Punica granatum*. Both dyes gave different shades of yellow and brown with different mordants. *Harda* dyed samples exhibited better light fastness when mordanted with iron, copper and chrome than alum, tin and tannic acid. Pomegranate rind dye showed better fastness with alum, copper, iron and chrome over unmordanted samples.

Gulrajani *et al* (1992e) investigated dyed conditions of wool for harshingar, safflower and barberry dyes. All the dyes gave yellow colours. The barberry dye exhibited poor to moderate washing and light fastness. The light fastness of harshingar and safflower dyed samples were found moderate but washing fastness was good to excellent with no staining on either wool or cotton fabrics.

Gulrajani *et al* (1993) conducted a study on dyeing wool with three red natural dyes, namely *Rubia cordifolia* (majith) *Caesalpinia sappan* (patangwood) and *Adenantha pavonina* (lal chandan). Effect of mordants

on the colour of dyed wool was studied by calculating CIE LAB values. Fastness properties of the dyes were tested and impact of mordants was evaluated.

Chirila and Calmus (1994) developed a method of dyeing wool with plant extracts. Wool was dyed by mordanting with  $K_2Cr_2O_7$ ,  $KAl(SO_4)_2$ ,  $FeSO_4$ ,  $Ca(NO_3)_2$ ,  $SnCl_2$  or other mordants, introducing into a water containing extracts from plants at various stages of development at  $70^\circ C$ , heating the wool in this bath, boiling for 30-120 minutes and developing the dye for 15-25 minutes in a bath containing  $CuSO_4$ ,  $F_2SO_4$ ,  $KAl(SO_4)_2$ ,  $K_2Cr_2O_7$  or  $NH_4Cl$ .

Tsatsanni and Eleftheriadis (1994) dyed wool with the aqueous extract of saffron containing a-crocin as the main colourant species. The dyeings were carried out with and without metal salts as mordants. The wash and light fastness of the dyed fabric was studied. The colour of the fabric was investigated in terms of CIE LAB values.

Verma and Gupta (1994) used eucalyptus bark to dye worsted knitting yarn of Bharat merino wool (a newly developed crossbred sheep at CSWRI). It was found that dyeing with natural dyes caused less strength loss than synthetic dyes. Light fastness of the eucalyptus dyed wool was lower than samples dyed with synthetic dyes while wash fastness was very

good. Simultaneous mordanting was found best for tin, chrome and alum while post mordanting for chrome.

Yamamoto (1994) investigated the application of a natural dye extracted from cultured *Euphorbia millii* (a red colour extract) to textiles. The colouring matter is capable of dyeing wool at low pH. Through the application of different salts as post-mordants a wide range of colours were obtained.

Kaur (1995) studied the dye affinity of wool for flower dyes, namely china rose (*Hibiscus rosa-sinensis*), gulmohar (*Delonix regia*), kikar (*Acacia arabica*) and sunflower (*Helianthus annus*). The dyes were extracted by treatment with water and ethyl alcohol and colour yield was 21.6, 15.20, 14.20 and 12.80 percent in water and 20.5, 14.5, 13.8 and 12.2 percent in ethyl alcohol, respectively. The dyeing conditions, namely dye extraction time, dye concentration, dyeing time, mordant concentration and mordanting method were optimized for each dye.

Verma and Gupta (1995) studied dyeing of wool with wattle bark. Dyeing was done after mordanting with alum, chrome, copper and ferrous sulphate. Light fastness of the dye at different pH was at par with light fastness of many synthetic dyes and also the wash fastness was very good. Tenacity of the woolen yarn was adversely affected by the wattle bark

colour. Use of mordants produced a deteriorating effect on the tensile properties of woollen yarn.

Chan and Yuen (1997) considered the potential for extraction of pigments from tea residues for textile industry. The raw materials and dyes concerned are described. Coloured matter was extracted from Chinese tea, but its affinity for wool was found to be low.

Rao (1997) conducted a study on powder from natural dyes available in market. Samples of wool were dyed with kamila, manjith, babool, lac, mango, rust red and mud yellow using alum as a mordant and as per recommendations of manufacturers. It was concluded that the method of application for a specific substrate suggested by manufacturer may not yield satisfactory results in many cases. Mango gave best results while kamila gave poor results.

Singh and Goel (1997) conducted a comparative study on dyeing wool fabric with reactive, acid and natural dye (katha). It was observed that treatment with acid dye reduced the fabric strength and fabric treated with natural dye showed highest strength. Use of mordants did not show any effect on fabric strength. It was concluded that acid dyes showed excellent washing fastness while natural dye showed good fastness to light.

Bohra (1998) performed a series of experiments for extracting colours from gudhal flower to dye wool samples. The test results showed

fair to good light fastness and poor to very good washing fastness in both mordanted and unmordanted woollen yarns.

Pant (1998) dyed woollen knitting yarn with African marigold, kethula flower and oak tree bark at different concentrations, different temperatures for different time periods, with different mordant concentrations and mordanting methods. One percent concentration of marigold, 2 per cent concentration of kethula and 4 percent concentration of oak bark was found to be optimum. Optimum temperature of dyeing was found to be 80-100°C and a range of shades was obtained with different mordants.

Verma *et al* (1998) studied eco-friendly dyeing of wool with eucalyptus and wattle bark. After dye extraction mordants, namely tin, iron, chrome, alum, were applied using all the three mordanting methods. A wide range of shades was obtained. A complete colour range of dark brown to dark grey was produced. Washing fastness was found to be at par with many synthetic dyes while light fastness observed was in the range of 4-5, which is suitable for many woollen products.

Bhattacharya and Shah (1999) conducted a study on dyeing wool with two natural dyes, namely tea leaves (*Thea sinensis*) and poi fruits (*Basella rubra*). The mordants used were iron, tin and potassium dichromate. In the case of tea dyeings, light, wash and rubbing fastness

were very good while in the case of poi dyeings, light fastness was poor but wash and rubbing fastness were good. For mordanting, pre and simultaneous methods were employed. Both were good for tea while simultaneous method was not suitable for poi dye.

Dayal and Dobhal (2000) conducted a study on dyeing wool with poplar bark (*Populus deltoides*). Dyeing process was optimized. Acidic medium was used for dyeing as the electrostatic forces between the positively charged side chain of the protein fibre and dye molecules would be expected to play a major role in dye adsorption. All mordants increased the L\* value of the dyed samples. Except alum and iron all increased a\* and b\* values also.

Kiranmai *et al* (2000) reported the use of dye from *Ixora singaporensis* flowers for wool. Alum and myrobolan were selected as mordants. Acidic method of extraction was found to be best because of maximum dye extraction. To obtain light and dark shades 3g and 7g flowers were used, respectively. Pre-mordanting method was best suited. The optimum extraction and dyeing time were 30 and 60 minutes, respectively. Fifteen percent alum and 20 percent myrobolan gave best results. All samples exhibited excellent fastness to light and rubbing. Fastness to washing was good to very good and to perspiration was good.

Singh and Yadav (2000) conducted a study to optimize dyeing conditions for wool using barberry bark. Best results were obtained by extracting dye for 60 minutes and dyeing for 45 minutes. Optimum dye material concentration was 2 per cent for lighter shade and 8 per cent for darker shade. Four synthetic mordants were applied by three different methods using two different dyeing media (acid and alkaline). Fastness properties were found to be good.

Bansal and Sood (2001a) conducted a study on dyeing of wool with barberry roots. Four mordants, namely alum, copper sulphate, ferrous sulphate and chrome were selected for the study and three media of dye extraction (aqueous, alkaline and acidic) were tried to extract maximum dye. Dyeing conditions, namely dye material concentration, dyeing time, mordant concentration and mordanting method were optimized and fastness properties of the dyed samples were tested.

Bansal and Sood (2001b) studied development of vegetable dye on wool from eupatorium leaves. Selected concentrations of dye material were 1.5 and 3.0 g for light and dark shades. Extraction and dyeing time was 45 minutes and the best extraction was done in alkaline medium. It was concluded that greenish sienna and greenish umber were the best shades on the basis of colour fastness

Mathur and Bhandari (2001) obtained beet sugar (*Beta vulgaris*) colourant by concentrating beet sugar juice under reduced pressure and evaporating it to dryness. It showed three absorption bands at 220, 280 and 530 nm. The optimum concentration of beet sugar colourant for dyeing Bharat merino wool was found to be 0.03 g per g of wool at pH 4.5 and temperature 97.5°C. The wool dyed as such did not meet the requirements of BIS colour fastness standards. However when the wool treated with metal ions such as Al (III), Cr (VI), Cu (II), Fe (II) and Sn (II) and rare earths such as La (III) and Sn (III) was dyed with beet sugar colourant, it could withstand the requirement of BIS fastness standards.

#### **2.6.3.2 Dyeing with animal dyes**

Agarwal (1987) reported that lac dissolved in hydrochloric acid is able to dye wool directly giving beautiful orange – red shade which is fast to light. The amount of mordant to be used varied from 2-5 per cent (o.w.f.) depending upon amount of dye used and shade required.

#### **2.7 Dyeing with natural dyes from microbes**

Mounter (1997) used different type of fungi to create natural dyes. The dyeing and mordanting methods used were same as for any other natural dye. The use of different mordants with various types of fungi resulted in production of a range of colours on wool.

Shirata *et al* (2000) isolated *Janthinobacterium lividum* from wet silk thread whose colour became bluish-purple. The pigment was extracted with methanol and was identified as a mixture of violacein and deoxyviolacein. This pigment was used to dye natural fibres like silk, cotton and wool and synthetic fibres like nylon and vinylon. The shade of colour depended on the material. Silk, cotton and wool showed a bluish-purple colour, nylon a dark blue colour and acetate a purple colour. Dyeing was performed by either dipping in pigment extract or boiling with the bacterial cells. The colour fastness of the dyed material was about the same as that of materials dyed with vegetable dyes but the colour faded easily on exposure to sunlight. The light fastness rating 1, wash fastness rating 2 to 3-4, perspiration fastness rating 3 and rubbing fastness rating 2-3 was reported.

## **CHAPTER III**

# **MATERIALS AND METHODS**

The materials and methods used in the present research study are discussed below:

### **3.1 Textile material**

Pure white polyester fabric was used for the research work. It was purchased from the local market.

### **3.2 Natural dye materials**

A total of ten natural dye materials having affinity for polyester were selected for the study as listed in Table 3.1 and shown in plates 1a to 9a. These were obtained from plants grown in Punjab state. The dye materials were examined carefully to remove extraneous matter. These were dried, chipped (in the case of bark, gum, rhizome and root) or crushed (in the case of leaves) and then ground to powder form as shown in plates 1b to 9b and 10.

Table 3.1 Natural dyes used in the study

S.No.	English name	Local name	Botanical name	Family	Part used	Source	Cost Rs./kg.
1.	Ashoka	Devdar	<i>Polyalthia longifolia</i>	Annonaceae	Leaves	PAU Campus, Ludhiana	Free
2.	Banyan	Barr	<i>Ficus bengalensis</i>	Moraceae	Leaves	-do-	Free
3.	Beef wood	Jor-tor	<i>Casuarina equisetifolia</i>	Casuarinaceae	Bark	-do-	Free
4.	Flame of forest	Palash	<i>Butea monosperma</i>	Leguminosaceae	Gum	Local market	160
5.	Golden drop	Ratanjot	<i>Onosma echtioides</i>	Verbenaceae	Root	Local market	70
6.	Indian blackberry	Jamun	<i>Syzygium cumini</i>	Myrtaceae	Bark	Local market	50
7.	Onion	Pyaz	<i>Allium cepa</i>	Liliaceae	Skin	Local market	Free
8.	Pine	Chir	<i>Pinus roxburguii</i>	Pinaceae	Bark	PAU	Free
9.	Rubber plant	Rubber	<i>Ficus elastica</i>	Moraceae	Leaves	Campus, Ludhiana PAU	Free
10.	Turmeric	Haldi	<i>Curcuma domestica</i>	Zingiberaceae	Rhizome	campus, Ludhiana Local market	45



(a)



(b)

**Plate 1 (a) Ashoka tree (b) Dried and ground ashoka leaves**

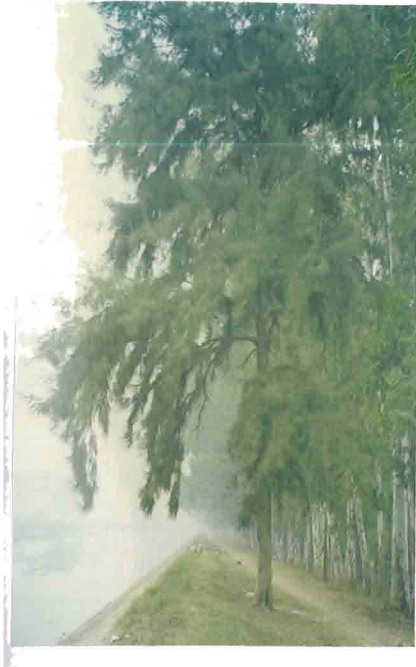


(a)



(b)

**Plate 2 (a) Banyan tree (b) Dried and ground banyan leaves**



(a)



(b)

**Plate 3 (a) Beefwood tree (b) Dried and ground beefwood bark**



(a)



(b)

**Plate 4 (a) Flame of forest tree (b) Dried and ground flame of forest bark**

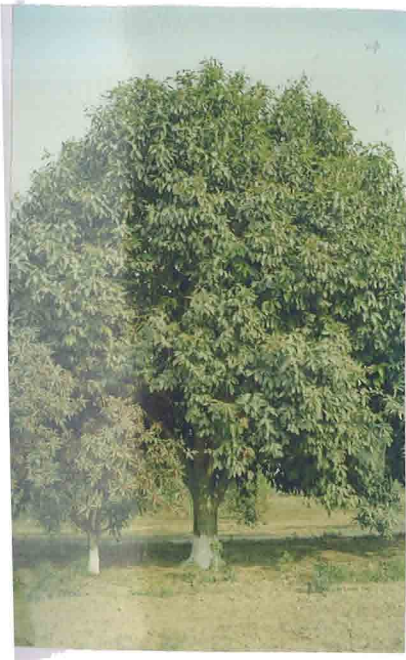


(a)



(b)

**Plate 5 (a) Golden drop tree (b) Dried and ground golden drop roots**



(a)



(b)

**Plate 6 (a) Indian blackberry (b) Dried and ground Indian blackberry bark**



(a)



(b)

**Plate 7 (a) Onion bulb (b) Dried and ground onion skin**



(a)



(b)

**Plate 8 (a) Pine tree (b) Dried and ground pine bark**



(a)



(b)

**Plate 9 (a) Rubber plant tree (b) Dried and ground rubber plant leaves**



(a)

**Plate 10 Dried and ground turmeric rhizomes**

**Table 3.2 Chemicals used in the study**

S. No.	Chemical	Molecular formula	Purpose	Supplier
1.	Acetic acid	CH <sub>3</sub> COOH	pH regulation	Thames Chemicals, Ludhiana
2.	Benzoic acid	C <sub>7</sub> H <sub>6</sub> O <sub>2</sub>	As carrier	-do-
3.	Cupric sulphate	CuSO <sub>4</sub> .5H <sub>2</sub> O	Mordanting	-do-
4.	Ferrous sulphate	Fe SO <sub>4</sub> .7H <sub>2</sub> O	Mordanting	-do-
5.	m-cresol	CH <sub>3</sub> C <sub>6</sub> H <sub>4</sub> OH	Identification of polyester (solubility test)	-do-
6.	Methanol	CH <sub>3</sub> OH	For dissolving carrier	-do-
7.	Petroleum ether	-	For removal of spin finish	-do-
8.	Alum (Potassium aluminium sulphate)	Al K (SO <sub>4</sub> ) <sub>2</sub> .12H <sub>2</sub> O	Mordanting	-do-
9.	Chrome (Potassium dichromate)	K <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub>	Mordanting	-do-
10.	Sodium carbonate	Na <sub>2</sub> CO <sub>3</sub>	pH regulation	-do-
11.	Sodium chloride	NaCl	Used in perspirometer for alkaline perspiration solution	-do-
12.	Sodium nitrate	NaNO <sub>3</sub>	For relative humidity maintenance in light fastness tester	-do-
13.	Urea	NH <sub>2</sub> CONH <sub>2</sub>	Used in perspirometer for acidic perspiration solution	-do-

**Table 3.3 Instruments used in the study**

S. No.	Instrument	Purpose	Manufacturer
1.	Crockmeter	Rubbing fastness testing	Paramounts Pvt. Ltd., N. Delhi
2.	Digital light fastness tester (Digi-light)	Light fastness testing	Paramounts Pvt. Ltd., N. Delhi
3.	Digital pH meter	pH measurement	Shri Ganesh Electrical System, Ambala Cantt.
4.	Electronic balance AD – 2008	Weighing of dyes, chemical and mordants	Adair, Dutt Instruments Pvt. Ltd., Calcutta
5.	Fabric thickness gauge	Fabric thickness measurement	Paramount Instruments Pvt. Ltd., N. Delhi
6.	Grinder	Grinding of dye materials	Remi Anupum Mixie Ltd, Mumbai
7.	Hot air sterilizer	Drying of dye materials (bark)	Yorco Scientific Industries, Delhi
8.	Laundrometer	Washing fastness testing	Paramount Instruments Pvt. Ltd., N. Delhi
9.	Multi-tray shade solar dryer	Drying of dye materials (leaves)	College of Agricultural Engineering, P.A.U., Ludhiana
10.	Perspirometer	Perspiration fastness testing	SASMIRA, Mumbai
11.	Pick glass	Fabric count measurement	Paramount Instruments Pvt. Ltd., New Delhi
12.	Rectangular hot plate	Dyeing	Shri Ganesh Electrical System, Ambala Cantt.
13.	Spectro-310 spectrophotometer.	Computerized colour measurement	

### 3.3 Mordants

The four synthetic mordants used for the study have been presented in Table 3.2.

### 3.4 Other chemicals used

The chemicals used in the study for various pre-treatments, dyeing process and in the testing of fastness of dyed samples are given in Table 3.2.

### 3.5 Instruments used

The instruments used in the study have been listed in Table 3.3.

### 3.6 Identification of textile material

To ensure the purity of textile material used in study microscopic, burning and chemical tests were conducted to identify it.

### 3.7 Specifications of the fabric

The type of weave, fabric count, weight and thickness of the polyester used were determined and are given in Table 3.4.

**Table 3.4 Specifications of the polyester fabric**

<b>Specification</b>	
Weave	Plain
Fabric count	35/35 per sq. cm
Weight	76 g/sq m
Thickness	1.20 mm

### **3.8. Sample preparation**

#### **3.8.1. Sample size**

Samples of polyester fabric of 1g each were weighed. A sample of 10 x 13 cms dimension weighed 1g.

#### **3.8.2 Preparation of samples for dyeing**

##### **3.8.2.1. Scouring**

The fabric was soaked in warm detergent solution containing 5g detergent (surf) in 1 litre of water for 1 hour. After scrubbing, kneading and squeezing, the fabric was rinsed with excess tap water to remove traces of detergent completely. After this fabric was dried.

##### **3.8.2.2 Removal of spin finishes**

The fabric was soaked in petroleum ether for 24 hours for removal of spin finishes.

#### **3.8.3 Carrier treatment**

Conventionally, the high temperature dyeing is used for polyester. But it has been found that by addition of certain low molecular weight substances, as carrier, dyeing can be carried out at low temperatures. The carriers can be insoluble in water such as o-phenylphenol, diphenyl or chlorinated benzenes or water soluble as phenol and benzoic acid (Peters

1975). In the present study a water-soluble carrier benzoic acid was used because the insoluble carriers fall in categories of banned compounds as well as cause problems in application and level dyeing. Also benzoic acid as a carrier is cheap and has good efficiency, medium washability, good leveling ability and low effect on fastness to light. (Pajgrt and Reichstadter 1979).

#### **3.8.4. Soaking**

The fabric samples were soaked in water prior to dyeing to remove air from it for rapid dye penetration.

### **3.9. Optimization of dyeing parameters**

A series of experiments were carried out to determine optimum values of nine dyeing parameters, namely carrier concentration, carrier treatment time, dye extraction pH, dye extraction time, dyeing pH, dye material concentration, dyeing time, mordanting method and mordant concentration. The detailed procedure followed in optimization of each variable is discussed below.

#### **3.9.1 Optimization of carrier concentration**

Five different aqueous concentrations of benzoic acid, i.e. 5, 10, 15, 20 and 25 percent (o.w.f.) were prepared for this 0.1g, 0.2g, 0.3 g, 0.4 g and 0.5 g of benzoic acid were dissolved in methanol @ 1 ml for 0.2g in

separate beakers and made upto 100 ml by adding boiling water. The solutions of benzoic acid were prepared in this manner as benzoic acid dissolves in water to the extent of 97 per cent only. Then wetted samples of polyester fabrics of 1g each were added into these five carrier solutions at boiling temperature and treatment was continued for an hour. The carrier solutions were stirred after every 5 minutes for even application of carrier on the samples. After this treatment, the samples were added to dyebaths in different beakers without rinsing. The dyebaths were prepared employing 4 per cent dye solution (2g dye in 50 ml water) and pH maintained at 7.0. The dyeing was carried out at boil for an hour with continuous stirring. The dyebaths were allowed to cool down after dyeing to prevent sudden change in temperature. The samples were then taken out, rinsed in tap water and dried in shade. The dried samples were examined for their colour. The carrier concentration at which maximum dye had been absorbed was judged by the darkness of the colour of dyed samples and was selected as optimum carrier concentration. For this evaluation the dyed samples were shown to a panel of five judges having experience of working with natural dyes. The judges were asked to rank the samples one to five on the basis of their colour, the best sample being given rank 1 and the poorest sample being given rank 5. The ranks given by all the judges for each sample were summed up. The sample having the minimum sum of ranks was selected as

the best sample and the carrier concentration used for that sample was selected as the optimum carrier concentration.

### **3.9.2. Optimization of carrier treatment time**

The carrier treatment was given for 15, 30, 45, 60 and 75 minutes to five fabric samples of 1g each with optimum carrier concentration, 1:100 material liquor ratio. The treated samples were then entered into dyebath prepared with 4 per cent dye solution (2 g dye in 50 ml water) and dyed for an hour with continuous stirring maintaining volume of dye liquor constant by adding hot water when needed. The dye bath was cooled to room temperature prior to removal of dyed samples, which were then rinsed to remove superfluous dyer and dried in shade. The optimum carrier treatment time was selected in the same way as the optimum carrier concentration as explained in the section 3.9.1.

### **3.9.3 Optimization of dye extraction pH**

The dye was extracted by boiling 1 g dye material in 50 ml water at seven different pH values, i.e. 4, 5, 6, 7, 8, 9 and 10 for an hour maintaining the liquor volume constant. The liquors were then strained through eight layers of scoured muslin fabric. The clear filterates were adjusted to pH 7 and then used as dyebaths.

Seven fabric samples of 1g each, presoaked in water and treated with carrier, were taken. One sample was added to each dyebath at 50°C, employing material-liquor ratio of 1:50. The temperature of dyebaths was gradually raised to boiling. The dyeing was carried out at this temperature for one hour stirring the samples frequently and maintaining liquor volume constant by adding hot water when needed. After dyeing, the dyebaths were allowed to cool down to room temperature to avoid sudden change in temperature during rinsing. The samples were then taken out and rinsed in tap water to remove superfluous dye material. The samples were finally dried in shade. The colour of dried samples was then examined to determine optimum dye extraction pH as explained in the section 3.9.1.

#### **3.9.4. Optimization of dye extraction time**

Extraction of dye was carried out at optimum pH for 15, 30, 45, 60, 75, 90 and 105 minutes. Seven samples of 1 g each were dyed in separate dyebaths employing material to liquor ratio of 1:50 at boiling temperature for one hour stirring the sample frequently. After dyeing the dyebaths were allowed to cool down to room temperature to avoid sudden temperature change during rinsing. The samples were then taken out and rinsed in tap water to remove superfluous dye material. The samples were finally dried in shade. The optimum dye extraction time was selected in the same way as optimum carrier concentration.



### **3.9.5. Optimization of dyeing pH**

Dyes were extracted at optimum pH for optimum time keeping other variables constant as in optimisation of dye extraction pH and dye extraction time. Seven dyebaths were prepared each having different pH values, i.e. 4, 5, 6, 7, 8, 9 and 10. Seven presoaked and carrier treated samples of 1 g each were taken and dyed in each bath employing material to liquor ratio of 1:50 at boiling temperature for one hour stirring the sample frequently. After dyeing the dyebaths were allowed to cool down to room temperature to avoid sudden temperature change during rinsing. The samples were then taken out and rinsed in tap water to remove superfluous dye material. The samples were finally dried in shade. The optimum dyeing pH was determined in the way as explained in 3.9.1.

### **3.9.6. Optimization of dye material concentration**

Seven concentrations of dye material, i.e. 0.5 g, 1 g, 2g, 3g, 4g, 5g, and 6g were extracted separately at optimum pH for optimum time. Seven presoaked and carrier treated samples of 1g each were dyed (one in each bath) at optimum pH employing material to liquor ratio of 1:50 at boiling temperature for one hour stirring the sample frequently. After dyeing the dyebaths were allowed to cool down to room temperature to avoid sudden temperature change during rinsing. The samples were then taken out and rinsed in tap water to remove superfluous dye material. The samples were

finally dried in shade. The optimum dye material concentration was determined on basis of darkness of colour as described in the section 3.9.1.

### **3.9.7. Optimization of dyeing time**

Dyes were extracted by boiling the optimum concentrations of dye materials at optimum pH for optimum time period. The dyeing was then carried out at optimum pH for seven different time periods, i.e. 15, 30, 45, 60, 75, 90 and 105 minutes. While dyeing 1:50 material to liquor ratio was maintained at boiling temperature. After dyeing the dyebaths were allowed to cool down to room temperature to avoid sudden temperature change during rinsing. The samples were then taken out and rinsed in tap water to remove superfluous dye material. The samples were finally dried in shade. Optimum dyeing time was also determined on the basis of darkness of the colour of dyed samples as described in the section 3.9.1.

### **3.9.8 Optimization of mordanting method**

Synthetic mordants were applied on the fabric samples by three methods, namely pre-mordanting, simultaneous mordanting and post-mordanting. The mordants, namely chrome, cupric sulphate and ferrous sulphate were employed at 2 percent concentration (o.w.f.) and one mordant, namely alum was employed at 10 percent concentration (o.w.f.) and applied with material to liquor ratio maintained at 1:50, for half an hour at pH 7. Dyes were extracted and applied under optimum conditions.

The dyed samples were evaluated for washing, light, rubbing and perspiration fastness.

Optimum mordanting method was selected on the basis of washing, light, rubbing and perspiration fastness grades of the dyed samples. The grades were summed up and the mean scores were obtained. The three mordanting methods were ranked on the basis of these scores. The mordanting method obtaining maximum mean score was selected as optimum method.

### **3.9.9 Optimization of mordant concentration**

Five different concentrations of mordants, i.e. 5, 10, 15, 20 and 25 percent (o.w.f.) (in the case of alum) and 1, 2, 3, 4 and 5 percent (o.w.f.) (in the case of ferrous sulphate, potassium dichromate and cupric sulphate) were applied under conditions mentioned in the section 3.9.8. Mordanted samples were dyed under optimum conditions. The dyed samples were evaluated for their colour fastness properties. Optimum concentration was decided on the basis of the washing, light and rubbing fastness ratings as explained in the section 3.9.8.

### **3.10. Evaluation of colour fastness of the dyed samples**

All the dyed samples except those for optimization of dye extraction pH and dye extraction time were evaluated for their colour fastness against

washing, light, rubbing and perspiration, to see the effect of different variables, i.e. carrier concentration, carrier treatment time, dyeing pH, dye material concentration, dyeing time, mordanting method and mordant concentration on the fastness properties.

### 3.10.1. Evaluation<sup>of</sup> colour fastness to washing

For determining the washing fastness of the dyed samples, the Test-3 prescribed by Indian standard IS : 764 – 1979 was used. The washed samples were assessed against standard grey scales for colour change and colour staining on adjacent fabrics of composite samples. The different grades in the grey scale for change in colour and staining of colour indicate following level of fastness.

Grey scale rating	Colour change	Colour staining
5	Excellent	No staining
4	Very Good	Slight staining
3	Good	Noticeable staining
2	Fair	Considerable staining
1	Poor	Exclusive staining

### 3.10.2 Evaluation of colour fastness to light

For determining the colour fastness of the dyed samples to light, the test prescribed by IS : 2154 – 1967 was employed.

The test samples were exposed to the light of MBTL fading lamp for standard time periods (at least 5 hours). The exposure was continued until

the test specimen faded to a contrast equal to grade 4 on grey scale of colour change. The approximate number of hours required to fade each blue wool standard to a contrast equal to grade 4 of grey scale on such exposure are given below:

<b>Blue wool standard no.</b>	<b>Number of hours</b>	<b>Fastness level</b>
1	5	Very poor
2	10	Poor
3	20	Fair
4	40	Fairly good
5	80	Good
6	160	Very good
7	320	Excellent
8	640	Outstanding

### **3.10.3 Evaluation<sup>of</sup> colour fastness to rubbing**

Colour fastness of dyed samples to dry and wet rubbing was determined using the test prescribed by IS : 766-1956. The standard rubbing pieces were assessed for colour staining against standard grey scales.

### 3.10.4 Evaluation of colour fastness to perspiration

The colour fastness of dyed samples to perspiration was determined by employing the test prescribed by IS:971-1956. The change in colour of dyed samples and the staining on adjacent undyed fabrics of composite sample was evaluated with the help of standard grey scales.

The following abbreviations will be used in tables showing results related to the effect of different dyeing parameters on the fastness properties of different dyes in Chapter IV.

WF	-	Washing fastness
LF	-	Light fastness
RF	-	Rubbing fastness
PF	-	Perspiration fastness
CC	-	Change in colour
CSC	-	Colour staining on cotton
CSP	-	Colour staining on polyester
CSW	-	Colour staining on wool
W	-	Wet
D	-	Dry
Al	-	Alkaline
Ac	-	Acidic
MS	-	Mean Score

### 3.11 Numerical measurement of colour of dyed samples

The methods of numerical measurement of colour was established by the Commission Internationale del 'Eclairage (CIE) in 1931 and 1976. This method provides uniform colour differences in relation to visual differences. Also, the measurement of colour is precise and colour is specified by a set of numbers which can be used to re-create the original colours at any other time and place, i.e. reproduction is possible. This method extends use of computer in colour-match prediction, i.e. to correct the colour of a recipe obtained by the use of the computer. Thus, the use of computer in measurement of colour increases the objectivity of experimental results of dyeing (Giles 1974 and Valia 1987).

The colour of dyed samples was measured numerically through computerized colour matching machine at NITRA, Ghaziabad. The reflectance spectra of the dyed samples were observed by Spectro – 310 spectrophotometer and K/S values and the CIE Lab co-ordinates  $L^*$ ,  $a^*$  and  $b^*$  were calculated. This spectrophotometer uses CIE Lab (76) colour space, D65 illuminant and 420 nm wavelengths to measure the actual colour and changes in colours. The CIE Lab colour space uses  $L^*$ ,  $a^*$ ,  $b^*$  scales to describe colour.

$L^*$  is a measure of lightness of an object and ranges from 0 (black) to 100 (white),  $a^*$  is a measure of redness (+ve  $a^*$ ) or greenness (-ve  $a^*$ ) and

$b^*$  is a measure of yellowness (+ve  $b^*$ ) or blueness (-ve  $b^*$ ) and  $c^*$  is a measure of dullness/brightness.

### **3.12 Naming of colours**

The colours of the dyed polyester samples were assigned the names of those colours given in the Methuen's Handbook of Colour (Kornerup and Wanschler 1978) which matched them the most.

## **CHAPTER IV**

# **RESULTS AND DISCUSSION**

The present study was conducted to optimize the processes of dyeing polyester with ten natural dyes using four synthetic mordants in each case. The effect of synthetic mordants on various parameters of dyeing was investigated and the colour fastness properties of the dyed polyester were evaluated. The results obtained with each dye are discussed under the following subheads :

- (i) Optimum dyeing conditions without mordanting.
- (ii) Optimum mordanting conditions.
- (iii) Effect of dyeing parameters on fastness of dyes.
- (iv) Effect of mordants on CIE LAB values of dyed polyester.

### **4.1 Dyeing with ashoka dye**

#### **4.1.1. Optimum dyeing conditions without mordanting**

Details of the ranks for the optimization of each parameter, namely carrier concentration, carrier treatment time, dye extraction pH, dye

**Table 4.1 Optimization of carrier concentration, carrier treatment time, dye extraction pH, dye extraction time, dyeing pH, dye material concentration and dyeing time of ashoka dye on polyester on the basis of mean ranks given by a panel of judges.**

Variable	Rank	Optimum value
Carrier concentration (percent)		
5	4	
10	3	
15	2	20
20	1	
25	5	
Carrier treatment time (minutes)		
15	5	
30	2	
45	4	60
60	1	
75	3	
Dye extraction pH		
4	7	
5	5	
6	5	
7	5	10
8	3	
9	2	
10	1	
Dye extraction time (minutes)		
15	7	
30	3	
45	1	
60	2	45
75	4	
90	5	
105	6	
Dyeing pH		
4	3	
5	4	
6	2	
7	1	7
8	5	
9	6	
10	7	
Dye material concentration (g/g)		
0.5	3	
1	1	
2	2	
3	6	1
4	4	
5	5	
6	7	
Dyeing time (minutes)		
15	6	
30	7	
45	5	
60	4	105
75	3	
90	2	
105	1	

extraction time, dyeing pH, dye material concentration and dyeing time and their optimum values have been presented in Table 4.1.

#### **4.1.1.1 Optimum carrier concentration**

It can be seen from the Table 4.1 that the colour of the dyed polyester improved with the increase in the concentration of carrier, the optimum carrier concentration being 20 per cent, beyond which it decreased.

#### **4.1.1.2 Optimum carrier treatment time**

It can be seen from the table that the dyeing improved with the increase in the time of carrier treatment, the optimum time being 60 minutes, beyond which the colour decreased again.

#### **4.1.1.3 Optimum dye extraction pH**

It can be seen from the Table 4.1 that the extraction of dye from the leaves powder increased with increase in the pH and optimum pH for dye extraction was 10. Singh et al (1996) also found that increasing the amount of alkali upto 0.3 g/g fabric in the extraction bath increased the extraction of *kamala* dye.

#### **4.1.1.4 Optimum dye extraction time**

Table indicates that a duration of 45 minutes was adjudged the best for maximum extraction of the dye. Extraction for longer period was not

found helpful in increasing the dye extraction. This may be because after a particular time some substances other than dye also start to leach out from the plant material which may hinder the dye absorption. Similar observations were made by Singh et al (1993a) in the case of madder dye.

#### **4.1.1.5 Optimum dyeing pH**

It is clear from the table that as the pH increased, the dyeing improved and was the best at neutral pH. Further increase in pH resulted in duller colours. The neutral pH, i.e. pH 7 was, therefore, adjudged optimum for application of ashoka dye.

#### **4.1.1.6 Optimum dye material concentration**

The table shows that the optimum dye material concentration was 1g per gram of polyester. The boiling of increased amount of dye resulted in reduced dye extraction. This may be because of the saturation of the extraction bath. Similar results were obtained by Bahl and Gupta (1988) in the case of cutch dye on silk.

#### **4.1.1.7 Optimum dyeing time**

The colour of polyester improved with increase in the duration of dyeing as depicted in Table 4.1 and the optimum dyeing time for ashoka dye was 105 minutes.

#### 4.1.2. Optimum mordanting conditions

The mordanting method and mordant concentration were optimized on the basis of overall fastness ratings of ashoka dye dyed polyester employing the carrier concentration, carrier treatment time, dye extraction pH, dye extraction time, dyeing pH, dye material concentration and dyeing time optimized earlier without mordanting. The fastness grades for optimization of mordanting method and mordant concentration are given in Tables 4.2 and 4.3, respectively. The optimum mordanting and mordant concentration for the four mordants have been presented in Table 4.4.

**Table 4.2 Optimization of mordanting method for ashoka dye on polyester on the basis of fastness grades.**

Mordant	Mor- danting method	WF			LF	RF		PF				MS		Rank	
		CC	CSC	CSP	CC	W	D	Al		Ac					
						CSC	CSC	CC	CSP	CSW	CC	CSP	CSW		
Alum	Pre	2	5	5	1	2/3	3/4	3	5	5	4/5	5	5	3.87	3
	Sim	1/2	5	5	2	4/5	3	4	5	5	4	5	5	4.08	2
	Post*	2	5	5	2	4	3/4	4	5	5	4	5	5	4.125	1
Chrome	Pre	2/3	5	5	3	3/4	4	4	5	5	2/3	5	5	4.12	3
	Sim*	3	5	5	3	3/4	3/4	4/5	5	5	4/5	5	5	4.33	1
	Post	3	5	5	3	3/4	3/4	4/5	5	5	4	5	5	4.29	2
Cupric sulphate	Pre	1	5	5	3	3/4	3/4	2/3	5	5	4	5	5	3.95	2.5
	Sim*	2	5	5	3	4	3/4	3	5	5	3	5	5	4.04	1
	Post	2	5	5	3	3	3/4	3	5	5	3	5	5	3.95	2.5
Ferrous sulphate	Pre	4	5	5	3	2/3	2/3	3/4	5	4/5	3/4	5	4/5	4.00	2
	Sim*	3/4	5	5	2	4	3	4	5	5	4	5	5	4.20	1
	Post	2	5	5	3	3	3	3	5	5	3	5	5	3.91	3

\* Optimum mordanting method.

### 4.1.2.1 Optimum mordanting method

The fastness ratings for different methods of mordanting have been presented in Table 4.2. The table reveals that the simultaneous mordanting method of mordant application was the best (highest mean score) for all the mordants except alum for which the post-mordanting method gave the best fastness grades.

**Table 4.3 Optimization of mordant concentration for ashoka dye on polyester on the basis of fastness grades.**

Mordant	Mordant concentration	WF			LF		RF		PF				MS	Rank	
		W		D		Al		Ac							
		CC	CSC	CSP	CC	CSC	CSC	CC	CSP	CSW	CC	CSP			CSW
Alum	5*	2/3	5	5	3	3/4	2/3	4/5	5	5	4	5	5	4.16	1
	10	2	5	5	2	4	3/4	4	5	5	4	5	5	4.12	2
	15	2	5	5	2	4	2/3	4	5	5	4	5	5	4.04	3
	20	1/2	5	5	1	3	2	4	5	5	3/4	5	5	3.75	5
	25	1	5	5	1	3/4	2	4	5	5	4/5	5	5	3.83	4
Chrome	1	3	5	5	2	3	2	4/5	5	5	4/5	5	5	4.08	5
	2	3	5	5	3	3/4	3/4	4/5	5	5	4/5	5	5	4.33	2
	3	4/5	5	5	2	3/4	3	4/5	5	5	4	5	5	4.29	3
	4	4	5	5	2	4	3	4	5	5	4	5	5	4.25	4
	5*	4/5	5	5	2	4	3/4	4	5	5	4/5	5	5	4.37	1
Cupric sulphate	1	3/4	5	5	2	3/4	2	3	5	5	3	5	5	3.91	4
	2	2	5	5	3	4	3/4	3	5	5	3	5	5	4.04	2.5
	3*	3	5	5	2	3	2/3	4	5	5	4/5	5	5	4.08	1
	4	3/4	5	5	2	3	2/3	4	5	5	3/4	5	5	4.04	2.5
	5	2	5	5	1	3/4	2/3	3/4	5	5	3/4	5	5	3.83	5
Ferrous sulphate	1	1	5	5	1	3/4	2/3	CI	5	5	CI	5	5	3.80	5
	2*	3/4	5	5	2	4	3	4	5	5	4	5	5	4.20	1
	3	2	5	5	2	4/5	2/3	3	5	5	3	5	5	3.91	3
	4	2	5	5	2	4	2/3	2/3	5	5	3/4	5	5	3.95	2
	5	2	5	5	1	3/4	1/2	4/5	5	5	4	5	5	3.87	4

\* Optimum mordant concentration.

#### 4.1.2.2 Optimum mordant concentration

The fastness grades for different concentrations of mordants have been given in Table 4.3. The table makes it clear that the overall fastness grades of the chrome treated dyed samples increased with the increase in mordant concentration. In the case of cupric sulphate and ferrous sulphate, the overall fastness grades increased with increase in mordant concentration upto certain level. Beyond that concentration the fastness rating decreased. The overall fastness grade of alum treated samples decreased with increase in mordant concentration. The optimum concentrations of alum, chrome, cupric sulphate and ferrous sulphate were 5, 5, 3 and 2 per cent, respectively (Table 4.4).

**Table 4.4 Optimum mordanting conditions for dyeing polyester with ashoka dye.**

Mordant	Mordating method	Mordant concentration (percent)
Alum	Post	5
Chrome	Sim	5
Cupric sulphate	Sim	3
Ferrous sulphate	Sim	2

#### 4.1.3 Effect of dyeing parameters on the fastness of ashoka dye

##### 4.1.3.1 Effect of carrier concentration

The washing fastness, light fastness, rubbing fastness and perspiration fastness grades of ashoka dye on polyester at different carrier concentrations have been presented in Table 4.5.

**Table 4.5 Effect of carrier concentration on fastness of ashoka dye on polyester.**

Carrier concentration (percent)	WF			LF	RF			PF				
	CC	CSC	CSP	CC	W	D	CC	Al		Ac		
					CSC	CSC		CSP	CSW	CC	CSP	CSW
5	1	5	5	2	3	3/4	4	5	5	4	5	5
10	1	5	5	3	3	3/4	3	5	5	4	5	5
15	1	5	5	3	3/4	3/4	4/5	5	5	3/4	5	5
20	1	5	5	2	3/4	3/4	4	5	5	4	5	5
25	1	5	5	1	3	3/4	3	5	5	3	5	5

The washing fastness of the dye remained the same for all carrier concentrations and the fastness grades indicated poor washing fastness with grade at 1 for change in colour but no staining was observed on adjacent cotton and polyester fabrics of composite samples.

The light fastness grades showed an increase with increase in carrier concentration upto 15 per cent but decreased on further increase in carrier concentration. Then light fastness grades were in the range of 1-3 indicating poor to fair light fastness.

The rubbing fastness was almost similar for all the carrier concentrations. The dry rubbing fastness was slightly more than wet rubbing fastness for all except at 15 and 20 per cent concentrations of carrier. The rubbing fastness of the dyed sample was good with the grades in the range of 3 to 3/4.

The perspiration fastness of the samples was moderate to good with grades in the range of 3 to 4/5. The alkaline perspiration fastness and acidic

perspiration fastness both were the same except at 15 per cent carrier concentration where alkaline perspiration fastness was very good. No staining was observed on adjacent wool and polyester fabrics of composite samples.

#### 4.1.3.2 Effect of carrier treatment time

Table 4.6 shows the washing, light, rubbing and perspiration fastness of ashoka dye on polyester samples treated with carrier for five different time periods.

**Table 4.6 Effect of carrier treatment time on fastness of ashoka dye on polyester.**

Carrier treatment time (minutes)	WF			LF	RF		PF					
	CC	CSC	CSP	CC	W	D	Al			Ac		
					CSC	CSC	CC	CSP	CSW	CC	CSP	CSW
15	1	5	5	1	3	3	4	5	5	3	5	5
30	1	5	5	1	3	3	4	5	5	4/5	5	5
45	1	5	5	1	4	¾	5	5	5	5	5	5
60	1	5	5	2	¾	¾	4	5	5	4	5	5
75	1	5	5	1	¾	¾	3	5	5	3	5	5

The washing fastness of all the samples remained the same indicating that carrier treatment time has no effect on washing fastness. The washing fastness was poor as the grade was 1.

The light fastness also did not change with the increase in the duration of carrier treatment except for 60 minutes where it improved to grade of 2.

The wet and dry rubbing fastness of all the samples was good which increased slightly after carrier treatment for 45 minutes.

The alkaline and acidic perspiration fastness grades were almost the same showing an increase with increase in carrier treatment duration upto 45 minutes after which slight decrease was same in the fastness grades. The perspiration fastness grades were in the range of 3-5 showing good to excellent fastness. No staining was noticed on adjacent wool and polyester fabrics.

#### 4.1.3.3 Effect of dyeing pH

The fastness grades of washing, light, rubbing and perspiration of ashoka dye on polyester at different pH values are given in Table 4.7.

**Table 4.7 Effect of dyeing pH on fastness of ashoka dye on polyester.**

Dyeing pH	WF			LF	RF		PF					
					W	D	Al			Ac		
	CC	CSC	CSP	CC	CSC	CSC	CC	CSP	CSW	CC	CSP	CSW
4	1	5	5	2	4	3/4	4	5	5	4/5	5	5
5	1	5	5	2	3/4	3/4	4	5	5	4/5	5	5
6	2	5	5	2	3/4	3/4	4/5	5	5	4/5	5	5
7	1	5	5	2	3/4	3/4	4	5	5	4	5	5
8	3	5	5	1	4	3	3/4	5	5	3/4	5	5
9	3	5	5	1	3/4	2/3	3/4	5	5	3/4	5	5
10	3	5	5	1	3/4	3/4	4	5	5	3	5	5

The washing fastness was found to increase with increase in dyeing pH except at pH 7 where the fastness grade decreased to 1 from 2 at pH 6.

No staining was found on the adjacent cotton and polyester fabrics of composite samples.

Light fastness grades remained the same (2) upto pH 7 while they decreased with further increase in pH, i.e. in the case of samples dyed in alkaline medium. The light fastness of the dyed samples was poor with the grades 1 and 2.

The rubbing fastness of the ashoka dye showed slight change with the increase in the dyeing pH. The wet rubbing fastness grades were higher at pH 4 and 8 whereas the dry rubbing fastness grades decreased to 3 and 2/3 at pH 8 and 9, respectively.

The acidic perspiration fastness was found to have inverse relation with dyeing pH. As the dyeing pH increased, the fastness grades decreased from 4/5 to 3. The alkaline perspiration fastness initially increased to 4/5 at pH 6 and then decreased to 3/4 at 8 and pH 8 and 9. No staining was noticed on adjacent wool and polyester fabrics.

#### **4.1.3.4 Effect of dye material concentration**

Table 4.8 indicates the fastness grades of ashoka dye on polyester for washing, light, rubbing and perspiration for different concentrations of dye material.

The washing fastness of dye remained the same upto 3 g/g concentration and then increased from 1 to 3 with increase in concentration to 6 g/g. No staining was observed on adjacent cotton and polyester fabrics.

**Table 4.8 Effect of dye material concentration on fastness of ashoka dye on polyester.**

Dye concentration (g/g)	WF			LF	RF			PF			Ac		
	CC		CSC	CC	W	D	Al		CC	CSP	CC	CSP	CSW
	CC	CSC	CSP	CC	CSC	CSC	CC	CSP	CSW	CC	CSP	CSW	
0.5	1	5	5	3	3/4	3/4	2/3	5	5	2	5	5	
1	1	5	5	2	4/5	3/4	3/4	5	5	4	5	5	
2	1	5	5	2	3/4	3/4	4	5	5	4	5	5	
3	1	5	5	1	3	2/3	3	5	5	3	5	5	
4	2/3	5	5	1	3	3	4/5	5	5	4	5	5	
5	2/3	5	5	1	3	3	4/5	5	5	5	5	5	
6	3	5	5	1	3	3	4	5	5	4	5	5	

The light fastness was found to have inverse relation with concentration of dye material. The fastness grades decreased from 3 to 1 on increasing concentration of dye material from 0.5 g to 3 g. The fastness grades remained unchanged with further increase in the dye material concentration.

The wet rubbing fastness increased to 4/5 at 1g concentration of dye material and decreased to 3 at 3 g concentration. Further increase in dye material concentration did not result in any change in fastness grades. The dry rubbing fastness remained constant (3/4) upto 2g concentration, decreased to 2/3 at 3 g concentration, increased again to 3 at 4 g

concentration and then remained constant on further increase in dye material concentration.

The perspiration fastness grades showed no particular trend either by increasing or decreasing with increase in concentration of dye material. The alkaline perspiration fastness increased to 4 from 2/3 with increasing concentration upto 2g, decreased to 3 at 3g concentration, increased to 4/5 at 4 and 5g concentrations and again decreased to 4 at 6g concentrations. The acidic perspiration fastness grades on the other hand increased to 4 from 2 at 2g concentration, decreased to 3 at 3g concentration and then remained constant at 4 on further increase in dye material concentration upto 6g. No staining was observed on adjacent wool and polyester fabrics.

#### 4.1.3.5 Effect of dyeing time

The washing, light, rubbing and perspiration fastness grades of ashoka dye dyed on polyester for different time periods have been presented in Table 4.9.

**Table 4.9 Effect of dyeing time on fastness of ashoka dye on polyester.**

Dyeing time (minutes)	WF			LF	RF		PF					
	CC	CSC	CSP	CC	W	D	Al			Ac		
					CSC	CSC	CC	CSP	CSW	CC	CSP	CSW
15	1	5	5	1	4	4	4/5	5	5	4/5	5	5
30	1/2	5	5	1	4	4	4	5	5	3/4	5	5
45	1	5	5	1	4	3/4	3	5	5	3	5	5
60	1	5	5	2	3/4	3/4	4	5	5	4	5	5
75	1	5	5	2	3/4	3	3/4	5	5	3	5	5
90	1	5	5	2	3	3	3	5	5	2/3	5	5
105	1	5	5	2	3	2/3	3/4	5	5	4	5	5

The washing fastness grades remained constant with the increase in the duration of dyeing except when dyeing was carried out for 30 minutes where fastness grade was  $\frac{1}{2}$ . No staining was noticed on adjacent cotton and polyester fabrics.

The light fastness grades remained constant (1) upto 45 minutes, then increased to 2 with increase in duration of dyeing to 60 minutes and remained constant on further increase in duration of dyeing.

The wet and dry rubbing fastness grades decreased with the increase in the duration of dyeing and the rubbing fastness was good to very good.

The perspiration fastness grades showed no uniform increasing or decreasing trend with increase in dyeing time. Both alkaline and acidic perspiration fastness grades decreased to 3 from  $\frac{4}{5}$  at 45 minutes, increased to grade 4 at 60 minutes, decreased to grade of 3 and  $\frac{2}{3}$ , respectively at 90 minutes and then increased to  $\frac{3}{4}$  and 4 at 105 minutes of dyeing. No staining was observed on adjacent wool and polyester fabrics.

#### **4.1.3.6 Effect of mordanting method**

Table 4.2 depicts the washing, light, rubbing and perspiration fastness grades of ashoka dye on polyester employing different mordants and the three mordanting methods with each mordant.

##### **4.1.3.6.1 Alum**

It is clear from the table that on mordanting with alum, the washing fastness grades were the same (2) in case of pre and post-mordanting

method whereas in the case of simultaneous mordanting method, the washing fastness grade was  $\frac{1}{2}$ . The light, wet and dry rubbing and alkaline and acidic perspiration fastness grades were the same for simultaneous and post-mordanting methods and were minimum for pre-mordanting method except in the case of acidic perspiration fastness grade which was maximum.

No staining was observed on adjacent fabrics of composite samples in the case of washing and perspiration fastness.

#### **4.1.3.6.2 Chrome**

In the case of chrome, the light fastness and wet rubbing fastness grades remained the same for all three mordanting methods.

The washing and alkaline perspiration fastness grades were the same for simultaneous and post-mordanting method and the grades were minimum for pre-mordanting method.

The dry rubbing fastness grade was maximum with pre-mordanting method and the fastness grade decreased and was same for simultaneous and post-mordanting method.

No staining was observed on adjacent fabrics of composite samples in the case of washing and perspiration fastness.

#### **4.1.3.6.3 Cupric sulphate**

The wet rubbing fastness was the same for all three mordanting methods. The washing, dry rubbing and both perspiration fastness grades

were the same for simultaneous and post-mordanting methods. The washing and dry rubbing fastness grades were minimum while acidic perspiration fastness grade was maximum with pre-mordanting method. The wet rubbing fastness grade was maximum in the case of simultaneous mordanted sample, followed by that for pre and post-mordanted samples.

Again no staining was noticed on adjacent fabrics of composite samples of washing and perspiration fastness.

#### **4.1.3.6.4 Ferrous sulphate**

It can be seen from the table that on mordanting with ferrous sulphate, the washing fastness was the best in the case of pre-mordanted sample, followed by simultaneous and post-mordanted samples. The light fastness grade was the same for pre and post-mordanted sample and was minimum in the case of simultaneous mordanted sample.

The best dry rubbing fastness was obtained in simultaneous mordanted sample, followed by post and pre-mordanted samples. The dry rubbing fastness was the same for simultaneous and post-mordanted samples and was minimum in the case of pre-mordanted sample.

The acidic and alkaline perspiration fastness grades were similar and were maximum with simultaneous mordanting method, followed by grades obtained with pre and post-mordanting methods.

No staining was observed on adjacent fabrics except in the case of pre-mordanting method while slight staining was observed on wool fabric.

#### **4.1.3.7 Effect of mordant concentration**

The washing, light, rubbing and perspiration fastness grades of polyester samples dyed with ashoka dye using five different mordant concentrations, have been presented in Table 4.3.

##### **4.1.3.7.1 Alum**

It is clear from the table that on increasing the concentration of alum, the washing and light fastness grades for change in colour decreased. The dry and wet rubbing fastness grades initially increased and then decreased. The dry and wet rubbing fastness grades initially increased and then decreased. The alkaline perspiration fastness grade decreased at 10 per cent concentration and remained constant on further increase in the mordant concentration. While the acidic fastness grades remained constant upto 15 per cent concentration of mordant and then increased with increase in mordant concentration.

No staining was observed on adjacent fabrics of composite samples in the case of washing and perspiration fastness.

##### **4.1.3.7.2 Chrome**

Washing fastness grades increased with increase in the mordant concentration except for decrease in fastness grade at 20 per cent concentration of mordant.

The light fastness remained the same at all the mordant concentrations except at 10 per cent concentration where the light fastness grade increased to 3 from 2.

The dry and wet rubbing fastness grades increased with the increase in concentration of mordant except dry fastness grades at 3 and 4 per cent concentration where the grades decreased slightly after increasing at 2 per cent concentration of mordant.

No staining was noticed on adjacent fabrics of composite samples in the case of washing and perspiration fastness.

#### **4.1.3.7.3 Cupric sulphate**

In the case of cupric sulphate, the light, dry rubbing and both perspiration fastness grades initially increased and then decreased with the increase in concentration of mordant.

The wet rubbing fastness grades increased at 2 per cent concentration then decreased and remained the same at 3 and 4 per cent concentration and again increased at 5 per cent concentration.

The washing fastness grades decreased at 2 per cent concentration, increased at 3 and 4 per cent concentration and then decreased at 5 per cent concentration.

No staining was observed on adjacent fabrics of composite samples in the case of washing and perspiration fastness.

#### **4.1.3.7.4 Ferrous sulphate**

The washing fastness grade increased at 2 per cent concentration and decreased at 3 per cent concentration remaining constant on further increase in concentration of mordant.

The light fastness grade increased at 2 per cent concentration remaining constant upto 4 per cent concentration and again decreasing at 5 per cent concentration.

The wet rubbing fastness grade increased upto 3 per cent concentration and then decreased on further increase in concentration of mordant, while the dry rubbing fastness grade increased and decreased with every alternate concentration.

The alkaline and acidic perspiration fastness grades were the same and decreased upto 3 per cent concentration and then increased with increase in concentration.

No staining was noticed on adjacent fabrics of composite samples in the case of washing and perspiration fastness.

#### 4.1.3.8 Effect of the type of mordant

The washing, light rubbing and perspiration fastness grade for the polyester sample dyed with ashoka dye using different mordants under optimum conditions have been compared with those of control (unmordanted) sample in the Table 4.10.

**Table 4.10 Effect of mordant type on fastness of ashoka dye on polyester.**

Mordant	WF			LF			RF			PF		
							W			D		
	CC	CSC	CSP	CC	CSC	CSC	CC	CSP	CSW	CC	CSP	CSW
Control	1	5	5	2	3	2/3	3/4	5	5	4	5	5
Alum	2/3	5	5	3	3/4	2/3	4/5	5	5	4	5	5
Chrome	4/5	5	5	2	4	3/4	4	5	5	4/5	5	5
Cupric sulphate	3	5	5	2	3	2/3	4	5	5	4/5	5	5
Ferrous sulphate	3/4	5	5	2	4	3	4	5	5	4	5	5

A marked increase was seen in the washing fastness grades in the samples mordanted with alum, chrome, cupric sulphate and ferrous sulphate.

The light fastness grades remained the same in control as well as mordanted samples except in the case of alum treated sample where the fastness grade showed an increase by one unit.

The wet rubbing fastness grades increased in the case of alum, chrome and ferrous sulphate treated samples and remained same for cupric sulphate treated samples, whereas the dry rubbing fastness remained the

same except for samples mordanted with chrome and ferrous sulphate where the fastness grades exhibited an increase.

The alkaline fastness increased with application of mordants while acidic perspiration increased in the case of chrome and cupric sulphate treated samples and remained the same in the case of alum and ferrous sulphate.

No staining was observed on adjacent fabrics of composite samples in washing and perspiration fastness.

From the mean score of fastness grade, it can be concluded that chrome was the best mordant, followed by ferrous sulphate, alum and cupric sulphate.

#### **4.1.4 Effect of mordants on CIE LAB values**

The effect of different mordants on colour strength, chrome and intensity of the polyester samples dyed with ashoka dye under optimum conditions has been presented in Table 4.11 in terms of K/S and CIE LAB values.

The L\* values greater than that of control sample indicated that alum, chrome and ferrous sulphate treated samples were lighter in colour than that of control sample. The darkness increased only in cupric sulphate treated sample. The lower a\* values of mordanted samples depicted decrease in redness of mordanted samples as compared to control sample.

**Table 4.11 Effect of mordants on CIE LAB values of ashoka dye on polyester.**

Mordant	L*	a*	b*	C*	K/S	Colour obtained
Control	65.193	5.944	5.714	8.145	1.11	Rosy grey (Pastel Pink grey)
Alum	65.137	5.570	5.424	7.775	1.24	Magenta grey
Chrome	64.680	5.420	6.064	8.133	1.29	Greyish brown rosewood
Cupric sulphate	66.091	5.099	8.362	9.794	1.67	Rose greyish brown
Ferrous sulphate	62.824	3.386	3.504	4.873	1.26	Greyish brown red

The yellowness increased in the case of chrome and cupric sulphate treated samples and decreased in alum and ferrous sulphate treated samples as compared to the yellowness of control sample as shown by b\* values. But the brightness improve only in the case of chrome mordanted samples as indicated by higher C\* value.

The increase in K/S values of the mordanted samples as compared to control sample indicated increase in colour strength on application of mordants. The colours obtained with ashoka under optimized conditions ranged from magenta grey to greyish brown rosewood.

A total of 67 shades of ashoka dye were obtained in the process of optimization of different dyeing variables.

## **4.2 Dyeing with banyan dye**

### **4.2.1 Optimum dyeing conditions**

Details of the ranks for the optimization of each variable, namely carrier concentration, carrier treatment time, dye extraction pH, dye

**Table 4.12 Optimization of carrier concentration, carrier treatment time, dye extraction pH, dye extraction time, dyeing pH, dye material concentration and dyeing time of banyan dye on polyester on the basis of mean ranks given by a panel of judges.**

Variable	Rank	Optimum value
Carrier concentration (percent)		
5	5	
10	4	
15	2	25
20	3	
25	1	
Carrier treatment time (minutes)		
15	3	
30	4	
45	5	75
60	2	
75	1	
Dye extraction pH		
4	2	
5	6	
6	5	
7	1	7
8	4	
9	3	
10	7	
Dye extraction time (minutes)		
15	7	
30	3	
45	4	
60	6	90
75	5	
90	1	
105	2	
Dyeing pH		
4	5	
5	6	
6	1	
7	3	6
8	2	
9	4	
10	7	
Dye material concentration (g/g)		
0.5	2	
1	1	
2	3	
3	4	1
4	5	
5	6	
6	7	
Dyeing time (minutes)		
15	7	
30	6	
45	5	
60	3	90
75	2	
90	1	
105	4	

extraction time, dyeing pH, dye material concentration and dyeing time and their optimum values have been presented in Table 4.12.

#### **4.2.1.1 Optimum carrier concentration**

It can be seen from the Table 4.12 that the colour of dyed samples improved with the increase in the concentration of carrier and optimum carrier concentration was 25 per cent.

#### **4.2.1.2 Optimum carrier treatment time**

It can be seen from the table that the best colour on polyester was obtained when carrier treatment was given for 75 minutes and the colour decreased with the decrease in the duration of the carrier treatment.

#### **4.2.1.3 Optimum dye extraction pH**

It can be seen from the Table 4.12 that the extraction of dye from the leaves powder was best at neutral pH, i.e. pH 7. The extraction of dye decreased as the pH of extraction become more acidic or alkaline, i.e. either 4 or 10, respectively.

#### **4.2.1.4 Optimum dye extraction time**

It table indicates that the extraction of dye increased with the increase in the duration of extraction. The extraction was optimum at 90 minutes and extraction for further more time did not help in improving extraction.

#### **4.2.1.5 Optimum dyeing pH**

It is clear from the table that the best dyeing was obtained in slight acidic medium, i.e. pH 6. The dyeing increased with increase in pH from 4 to 6 and then decreased with increase in alkalinity of dye bath, i.e. pH 8 to 10.

#### **4.2.1.6 Optimum dye material concentration**

The table shows that the optimum dye material was 1 g/g of polyester. The colour of the dyed sample decreased with the increase in the dye material concentration. This was due to reduced extraction of dye in same volume of extraction medium with increase in amount of dye material. Similar results were obtained by Agarwal et al (1992) in the case of henna dye on merino wool.

#### **4.2.1.7 Optimum dyeing time**

The colour of dyed samples improved with the increase in duration of dyeing as depicted by Table 4.12 and the optimum dyeing time for banyan dye was 90 minutes. On further increase in time of dyeing, the colour of dyed sample decreased.

#### **4.2.2 Optimum mordanting conditions of banyan dye**

The mordanting method and mordant concentration were optimized on the basis of overall fastness ratings of banyan dye dyed polyester

employing the carrier concentration, carrier treatment time, dye extraction pH, dye extraction time, dyeing pH, dye material concentration and dyeing time optimized earlier without mordanting. The fastness grades for optimization of mordanting method and mordant concentration are given in Tables 4.13 and 4.14, respectively. The optimum mordanting and mordant concentration for the four mordants have been presented in Table 4.15.

**Table 4.13 Optimization of mordanting method for banyan dye on polyester on the basis of fastness grades.**

Mordant	Mor- danting method	WF			LF	RF		PF			MS	Rank			
		CC	CSC	CSP	CC	W	D	Al		Ac					
						CSC	CSC	CC	CSP	CSW			CC	CSP	CSW
Alum	Pre	2	5	5	3	3	3	2	5	5	4	5	5	4.0	3
	Sim	2/3	5	5	3	2/3	3	5	5	5	3/4	5	5	4.12	2
	Post*	4	5	5	2	3/4	4	4/5	5	5	3	5	5	4.25	1
Chrome	Pre*	2/3	5	5	4	2	3	4/5	5	5	4/5	5	5	4.20	1
	Sim	3	5	5	4	2	2/3	3/4	5	5	3	5	5	4.00	2.5
	Post	3/4	5	5	3	2	2	4	5	5	3/4	5	5	4.00	2.5
Cupric sulphate	Pre	2	5	5	2	2/3	3	2	5	5	CI	5	5	3.77	2
	Sim	2	5	5	3	2/3	2/3	4	5	5	1	5	5	3.75	3
	Post*	4	5	5	4	3	3	4	5	5	4	5	5	4.33	1
Ferrous sulphate	Pre	4	5	5	4	2	2/3	2/3	5	5	2/3	5	5	3.83	3
	Sim	2	5	5	2	2/3	3	CI	5	5	4	5	5	3.68	2
	Post*	3/4	5	5	4	2	2	CI	5	5	4/5	5	5	4.18	1

\*Optimum mordanting method

#### 4.2.2.1 Optimum mordanting method

The fastness ratings for different methods of mordanting have been presented in Table 4.13. The table reveals that the post-mordanting method was the best for all the mordants except chrome for which pre-mordanting method gave the best fastness grades (Table 4.13).

**Table 4.14 Optimization of mordant concentration for banyan dye on polyester on the basis of fastness grades.**

Mordant	Mordant concentration	WF			LF	RF		PF					MS	Rank		
		CC	CSC	CSP	CC	W	D	Al			Ac					
						CSC	CSC	CC	CSP	CSW	CC	CSP	CSW			
Alum	5	4/5	5	5	1	2/3	2/3	4	5	5	5	3/4	5	5	4.00	5
	10	4	5	5	2	3/4	4	4/5	5	5	5	3	5	5	4.25	3.5
	15*	4	5	5	3	3	3	4/5	5	5	5	4/5	5	5	4.33	1
	20	3/4	5	5	3	3	3	4/5	5	5	5	4/5	5	5	4.29	2
	25	4/5	5	5	1	3/4	3/4	4	5	5	5	4/5	5	5	4.25	3.5
Chrome	1	2/3	5	5	3	3	3	4/5	5	5	5	4	5	5	4.16	5
	2	2/3	5	5	4	2	3	4/5	5	5	5	4/5	5	5	4.20	4
	3*	3	5	5	3	3/4	3/4	4/5	5	5	5	4/5	5	5	4.33	1
	4	4	5	5	3	2	3	4	5	5	5	4	5	5	4.25	3
	5	4/5	5	5	3	2/3	3	4/5	5	5	5	4	5	5	4.29	2
Cupric sulphate	1	4	5	5	2	3	2/3	4	5	5	5	4/5	5	5	4.16	4
	2*	4	5	5	4	3	3	4	5	5	5	4	5	5	4.33	1
	3	3/4	5	5	3	3	2/3	4	5	5	5	4	5	5	4.16	4
	4	4/5	5	5	2	3	2/3	4	5	5	5	4	5	5	4.16	4
	5	4	5	5	2	3/4	3	4	5	5	5	4	5	5	4.20	2
Ferrous sulphate	1	3/4	5	5	2	2/3	2	4	5	5	5	4	5	5	4.00	5
	2	3/4	5	5	4	2	2	CI	5	5	5	4/5	5	5	4.18	4
	3*	4/5	5	5	5	2	2	CI	5	5	5	4/5	5	5	4.36	1
	4	4	5	5	3	2	2	4/5	5	5	5	4/5	5	5	4.25	3
	5	4	5	5	5	2	2	4/5	5	5	5	4/5	5	5	4.33	2

\*Optimum mordant concentration.

#### 4.2.2.2 Optimum mordant concentration

The fastness grades for different concentrations of mordants have been given in Table 4.14. The table makes it clear that the overall fastness grades increased with increase in mordant concentration upto certain level. Beyond that concentration the fastness ratings decreased. The optimum concentrations of alum, chrome, cupric sulphate and ferrous sulphate were 15, 3, 2 and 3 per cent, respectively (Table 4.15).

**Table 4.15 Optimum mordanting conditions for dyeing polyester with banyan dye.**

Mordant	Mordating method	Mordant concentration (percent)
Alum	Post	15
Chrome	Pre	3
Cupric sulphate	Post	2
Ferrous sulphate	Post	3

### 4.2.3 Effect of dyeing parameters on the fastness of banyan dye

#### 4.2.3.1 Effect of carrier concentration

The washing fastness, light fastness, rubbing fastness and perspiration fastness grades of banyan dye on polyester at different carrier concentrations have been presented in Table 4.16.

**Table 4.16 Effect of carrier concentration on fastness of banyan dye on polyester.**

Carrier concentration (percent)	WF			LF	RF		PF					
					W	D	Al			Ac		
	CC	CSC	CSP	CC	CSC	CSC	CC	CSP	CSW	CC	CSP	CSW
5	3	5	5	2	¾	3	4/5	5	5	4/5	5	5
10	3	5	5	2	¾	3	4	5	5	4	5	5
15	4	5	5	1	2/3	2/3	4	5	5	4	5	5
20	4/5	5	5	2	3	3	4/5	5	5	¾	5	5
25	CI	5	5	2	3	3	4/5	5	5	¾	5	5

The washing fastness of the dye remained constant till 10 per cent carrier concentration, then increased with increase in carrier concentration. Increase in colour of dyed fabric was observed at 25 per cent carrier concentration. The washing fastness was good to very good with fastness

grades in the range 3 to 4/5. No staining was observed on adjacent fabrics of composite samples.

The light fastness grade remained the same for all carrier concentrations except at 15 per cent carrier concentration where it showed a decrease. The light fastness grades were in the range 1-2 indicating poor to fair light fastness.

The wet rubbing fastness remained same till 10 per cent carrier concentration and decreased for 15 per cent carrier concentration. It then showed an increase for 20 per cent carrier concentration and remained the same with further increase in carrier concentration. The dry rubbing fastness remained the same for all carrier concentrations except 15 per cent where it decreased. The rubbing fastness of the dyed samples was moderate with the grades in the range of 3 to  $\frac{3}{4}$ .

The perspiration fastness of the samples was good to very good. The alkaline perspiration fastness decreased initially with carrier concentration upto 10 per cent and remained the same for 15 per cent carrier concentration. The grades increased at 20 per cent concentration and then remained the same with further increase in carrier concentration. The acidic perspiration fastness decreased with increase in carrier concentration. No staining was observed on adjacent fabrics of composite samples.

#### 4.2.3.2 Effect of carrier treatment time

Table 4.17 shows the washing, light, rubbing and perspiration fastness of banyan dye on polyester samples treated with carrier for five different time periods.

**Table 4.17 Effect of carrier treatment time on fastness of banyan dye on polyester.**

Carrier treatment time (minutes)	WF			LF	RF		PF					
					W	D	Al			Ac		
	CC	CSC	CSP	CC	CSC	CSC	CC	CSP	CSW	CC	CSP	CSW
15	$\frac{3}{4}$	5	5	2	$\frac{3}{4}$	4/5	2/3	5	5	2/3	5	5
30	4	5	5	2	4	4/5	3	5	5	3	5	5
45	4	5	5	2	4	4/5	$\frac{3}{4}$	5	5	3	5	5
60	3	5	5	2	$\frac{3}{4}$	3	4	5	5	4	5	5
75	3	5	5	2	3	3	4	5	5	4	5	5

The washing fastness of all the samples showed a slight initial increase with increase in duration. The fastness decreased for 60 minutes duration of carrier treatment and then remained the same for further increase in duration. The washing fastness was good with grades between 3 to 4.

The light fastness remained same indicating that carrier treatment time had no effect on light fastness. The light fastness was fair as the grade was 2.

The wet rubbing fastness showed a slight initial increase upto 60 minutes duration of carrier treatment it showed a decrease in grades. The dry rubbing fastness remained the same initially. It showed a decrease at 60

minutes duration of carrier treatment and then remained the same with further increase in duration. The rubbing fastness for all the samples was good with grades ranging from 3 to 4/5.

The alkaline and acidic perspiration fastness grades were almost the same and increased with increase in carrier treatment duration. The decrease in the perspiration fastness was in the range of 2/3 to 4.

No staining was observed on adjacent fabrics of composite samples in both washing and perspiration fastness.

#### 4.2.3.3 Effect of dyeing pH

The fastness grades of washing, light, rubbing and perspiration of banyan dye on polyester at different pH values are given in Table 4.18.

**Table 4.18 Effect of dyeing pH on fastness of banyan dye on polyester.**

Dyeing pH	WF			LF	RF		PF					
	CC	CSC	CSP	CC	W	D	Al			Ac		
					CSC	CSC	CC	CSP	CSW	CC	CSP	CSW
4	4	5	5	1	2/3	2/3	4	5	5	4	5	5
5	3	5	5	1	2/3	2/3	4	5	5	4	5	5
6	2/3	5	5	1	2	3	3	5	5	4/5	5	5
7	3	5	5	2	¾	3	4	5	5	4	5	5
8	2	5	5	1	2/3	¾	3	5	5	3	5	5
9	2	5	5	1	3	4/5	2	5	5	3	5	5
10	2	5	5	1	¾	4/5	1	5	5	2	5	5

The washing fastness was found to decrease with increase in dyeing pH except at pH 7 where the fastness grade increased slightly. No staining was found on adjacent cotton and polyester fabrics.

Light fastness grade remained the same (1) for all dyeing pH except at pH 7 where it increased. The light fastness of the dyed samples was poor with the grade 1

The wet rubbing fastness did not show any fixed pattern. It initially decreased till pH 6. At pH 7 it increased to  $\frac{3}{4}$  and then decreased to  $\frac{2}{3}$  at pH 8. Again at pH 9 and 10 it showed an increase with grades 3 and  $\frac{3}{4}$ , respectively. The dry rubbing fastness increased with increase in dyeing pH and was fair to very good.

The perspiration fastness grades were almost the same (4) except at pH 6 where alkaline perspiration fastness showed a decrease ( $\frac{3}{4}$ ) and acidic perspiration fastness showed an increase ( $\frac{4}{5}$ ). After pH 7 both alkaline and acidic perspiration fastness decreased to grades 1 and 2, respectively with increase in dyeing pH. No staining was noticed on adjacent wool and polyester fabrics.

#### **4.2.3.4 Effect of dye material concentration**

Table 4.19 indicates the fastness grades of banyan dye on polyester for washing, light, rubbing and perspiration for different concentrations of dye material.

The washing fastness remained the same (3) upto 3g/g concentration and then decreased with increase in concentration to 6g/g. The washing

fastness of dyed samples was fair to good. No staining was observed on adjacent cotton and polyester fabrics.

**Table 4.19 Effect of dye material concentration on fastness of banyan dye on polyester.**

Dye concentration (g/g)	WF			LF	RF			PF				
	CC	CSC	CSP	CC	W	D	CC	Al		Ac		
					CSC	CSC		CSP	CSW	CC	CSP	CSW
0.5	3	5	5	1	3	4	4/5	5	5	3/4	5	5
1	3	5	5	2	3	3	4	5	5	4	5	5
2	3	5	5	2	3/4	3	4	5	5	4	5	5
3	3	5	5	2	2	2/3	4	5	5	4	5	5
4	2/3	5	5	4	2/3	2/3	3/4	5	5	3/4	5	5
5	2	5	5	3	2	2	3/4	5	5	3/4	5	5
6	2	5	5	3	2	3	3/4	5	5	3/4	5	5

The light fastness initially increased to 2 at 1g/g concentration from 1 at 0.5 g/g. It then remained the same upto 3g/g concentration. At 4g/g it increased to 4 and then decreased to 3 at 5g/g and 6g/g concentration.

The wet rubbing fastness showed an increase to 3/4 at 2g/g concentration from 3 at 1g/g. At 3g/g concentration it decreased to 2 and then increases to 2/3 at 4g/g. It then again decreased to 2 at 5g/g and 6g/g concentrations. The dry rubbing fastness showed a gradual decrease from 4 to 2 with increase in dye concentration upto 5g/g. The grades then increased to 3 at 6g/g of polyester.

The alkaline perspiration fastness grades showed a gradual decrease from 4/5 at 0.5 g/g to 3/4 at 4g/g. The grades then remained the same with further increase in concentration. The acidic fastness grades initially

increased from  $\frac{3}{4}$  at 0.5g/g to 4 at 1g/g. Then the grades remained the same upto 3g/g. At 4g/g it decreased to  $\frac{3}{4}$  and then remained constant with further increase in concentration of dye. No staining was observed on adjacent wool and polyester fabrics.

#### 4.2.3.5 Effect of dyeing time

The washing, light, rubbing and perspiration fastness grades of banyan dye dyed on polyester for different time periods have been presented in Table 4.20.

**Table 4.20 Effect of dyeing time on fastness of banyan dye on polyester.**

Dyeing time (minutes)	WF			LF	RF		PF					
	CC	CSC	CSP	CC	W	D	Al		Ac			
					CSC	CSC	CC	CSP	CSW	CC	CSP	CSW
15	$\frac{1}{2}$	5	5	1	4	4	4	5	5	4	5	5
30	2	5	5	2	4	$\frac{4}{5}$	$\frac{3}{4}$	5	5	4	5	5
45	2	5	5	2	3	3	$\frac{3}{4}$	5	5	4	5	5
60	3	5	5	2	$\frac{3}{4}$	3	4	5	5	4	5	5
75	$\frac{2}{3}$	5	5	2	$\frac{3}{4}$	2	$\frac{3}{4}$	5	5	3	5	5
90	2	5	5	2	3	3	$\frac{2}{3}$	5	5	3	5	5
105	$\frac{1}{2}$	5	5	2	2	3	$\frac{1}{2}$	5	5	$\frac{2}{3}$	5	5

The wash fastness increased with increase in dyeing time upto 60 minutes from  $\frac{1}{2}$  to 3. Thereafter, it showed a decrease from  $\frac{2}{3}$  at 75 minutes to  $\frac{1}{2}$  at 105 minutes. No staining was noticed on adjacent cotton and polyester fabrics.

The light fastness showed an initial increase from 1 at 15 minutes to 2 at 30 minutes. It remained the same with further increase in dyeing time.

The wet rubbing fastness decreased from grade 4 at 15 minutes to 3 at 45 minute. For 60 minute dyeing time it showed a slight increase to  $\frac{3}{4}$  and then again decreased to 3 at 90 minutes and 2 at 105 minutes. The dry rubbing fastness decreased from 4 at 15 minutes to 2 at 75 minutes dyeing time. It shows an increase to 3 at 90 minutes and thereafter, remained the same.

The alkaline perspiration fastness decreased initially from 4 at 15 minutes to  $\frac{3}{4}$  at 30 minutes. Again it increased to 4 at 60 minutes. It decreased to  $\frac{3}{4}$  at 75 minutes and thereafter, showed a decrease with increase in dyeing time. The acidic perspiration fastness decreased from 4 at 15 minutes to  $\frac{2}{3}$  at 105 minutes dyeing time after remaining the same upto 1 hour of dyeing. No staining was observed on adjacent wool and polyester fabrics.

#### **4.2.3.6. Effect of mordanting method**

Table 4.13 depicts the washing, light, rubbing and perspiration fastness grades of banyan dye on polyester employing different mordants and the three mordanting methods with each mordant.

##### **4.2.3.6.1 Alum**

It is clear from the table that on mordanting with alum, the fastness grade of washing for change in colour was maximum when mordant was applied using post-mordanting method, followed by that for simultaneous

and pre-mordanting method. Light fastness grades were similar for pre and simultaneous mordanting method and was minimum in the case of post-mordanted sample. Wet rubbing fastness was maximum when post-mordanting method was used, followed by that for pre and simultaneous mordanting method. Dry rubbing fastness remained the same for pre and simultaneous mordanting method and was the best when post – mordanting method was used. Alkaline perspiration fastness was the best with simultaneous mordanting method, followed by that for post and pre-mordanting methods while acidic perspiration fastness was the best with pre-mordanting method, followed by that for simultaneous and post-mordanting method.

#### **4.2.3.6.2 Chrome**

In the case of chrome, maximum washing fastness grade for change in colour was obtained on application of post-mordanting method, followed by that for simultaneous and pre-mordanting methods. Light fastness grade was minimum with post-mordanting method and remained the same for pre and simultaneous mordanting methods. Wet rubbing fastness grades were similar for all the three mordanting methods but dry rubbing fastness was maximum with pre-mordanting method, followed by that for simultaneous and post-mordanting methods. Alkaline perspiration fastness was the best with pre-mordanting method, followed by that for post and simultaneous mordanting methods while fastness grades of acidic perspiration fastness

was the best pre-mordanting method, followed by that for post and simultaneous mordanting methods (in that order).

#### **4.2.3.6.3 Cupric sulphate**

The fastness grades of washing for change in colour the best when post-mordanting method was and remained the same for pre and simultaneous mordanting methods. Light fastness grades was maximum with pre-mordanting method, followed by that for simultaneous and post-mordanting methods. Wet rubbing fastness grades were the same with pre and simultaneous mordanting methods minimum with post-mordanting method. Dry rubbing fastness was minimum with simultaneous mordanting method and remained the same with pre and post-mordanting methods. Alkaline perspiration fastness grade was minimum with pre-mordanting method and remained the same for simultaneous and post-mordanting methods whereas acidic perspiration fastness grade was poor with simultaneous mordanting method and very good with post-mordanting method but the colour of pre-mordanted sample increased when acidic perspiration solution treatment was given.

#### **4.2.3.6.4 Ferrous sulphate**

It can be seen from the table that on mordanting with ferrous sulphate, washing fastness grade for change in colour was the best in the case of pre-mordanted sample, followed by that for post and simultaneous

mordanted samples. Light and wet rubbing fastness grades were the same in the case of pre and post-mordanted samples but fastness grade for light was the best and for wet rubbing was minimum in the case of simultaneous mordanting method. Dry rubbing fastness grade of simultaneous mordanting method was maximum, followed by that for pre and post-mordanting methods. Acidic perspiration fastness grades were maximum with pre-mordanting method, followed by that for simultaneous and post-mordanting methods. An increase in colour was noticed in simultaneous and post mordanted samples for alkaline perspiration and fastness grade of pre-mordanted sample was similar to that of acidic perspiration fastness grade for pre-mordanted sample.

The adjacent fabrics of composite samples of washing and perspiration fastness showed no staining with all the mordants.

#### **4.2.3.7 Effect of mordant concentration**

The washing, light, rubbing and perspiration fastness grades of polyester samples dyed with banyan dye using five different mordant concentrations, have been presented in Table 4.14.

##### **4.2.3.7.1 Alum**

It is clear from the table that on mordanting with alum, the fastness grades of washing for change in colour decreased with increase in the concentration of mordant and then increased at 25 per cent concentration.

Light fastness grades increased upto 20 per cent concentration and then decreased greatly at 25 per cent mordant concentration. Wet and dry rubbing fastness grades initially increased followed by decrease and increase in fastness with increase in the concentration of mordants. Alkaline perspiration fastness grades initially increased and remained the same upto 20 per cent concentration and then decreased slightly at 25 per cent mordant concentration. Acidic perspiration fastness initially decreased, then increased and remained constant on increase in mordant concentration.

No staining was noticed on adjacent fabrics of composite samples of washing and perspiration fastness.

#### **4.2.3.7.2 Chrome**

In the case of chrome, the washing fastness grades improved with increase in the concentration of mordant. Light fastness grades remained constant at different concentrations of mordant except at 2 per cent concentration where an increase was observed in fastness grade. The fastness grades of wet rubbing were minimum at 2 and 4 per cent concentration and maximum at 3 per cent concentration of mordant. Dry rubbing fastness grades remained the same at different mordant concentrations except at 3 per cent concentration where fastness grade increased slightly. Alkaline perspiration fastness grades remained the same

at different mordant concentration except 4 per cent concentration where fastness decreased slightly. Acidic perspiration fastness increased slightly at 2 and 3 per cent concentration of mordant.

The adjacent fabrics of composite samples showed no staining in case of both washing and perspiration fastness.

#### **4.2.3.7.3 Cupric sulphate**

Washing fastness grades initially decreased slightly at 3 per cent concentration, increased at 4 per cent concentration and decreased again on further increase in mordant concentration. Light fastness grades showed increase at 2 per cent concentration and decreased on further increase in the mordant concentration. Wet rubbing fastness grades remained the same with increase in mordant concentration upto 4 per cent and then increased slightly at 5 per cent mordant concentration. Dry rubbing fastness increased slightly at 3 and 5 per cent concentrations of mordant. Alkaline and acidic perspiration fastness remained the same on increase in the concentration of mordant except at 1 per cent concentration where the acidic perspiration fastness grade was slightly more.

No staining was noticed on the adjacent fabrics of composite samples of washing and perspiration fastness.

#### **4.2.3.7.4 Ferrous sulphate**

It can be seen from the table that on mordanting with ferrous sulphate, the fastness grade of washing for change in colour increased at 3 per cent concentration of mordant, then decreased and became constant on further increase in concentration of mordant. The light fastness grades improved with increase in concentration of mordant except at 4 per cent concentration. The wet and dry rubbing fastness grades remained the same at different mordant concentrations except 1 per cent concentration where wet rubbing fastness improved slightly. Acidic and alkaline perspiration fastness increased with concentration and remained the same on further increase in concentration of mordant except at 2 and 3 per cent concentrations of mordant where the colour of dyed samples increased after soaking of sample in alkaline perspiration solution.

The adjacent fabrics of composite samples showed no staining in case of washing and perspiration fastness test.

#### **4.2.3.8 Effect of the type of mordant**

The washing, light rubbing and perspiration fastness grade for the polyester sample dyed with banyan dye using different mordants under optimum conditions have been compared with those of control (unmordanted) sample in the Table 4.21.

**Table 4.21 Effect of mordant type on fastness of banyan dye on polyester.**

Mordant	WF			LF	RF		PF					
	CC	CSC	CSP	CC	W	D	Al			Ac		
					CSC	CSC	CC	CSP	CSW	CC	CSP	CSW
Control	2	5	5	2	3	3	2/3	5	5	3	5	5
Alum	4	5	5	3	3	3	4/5	5	5	4/5	5	5
Chrome	3	5	5	3	¾	¾	4	5	5	4/5	5	5
Cupric sulphate	4	5	5	4	3	3	4	5	5	4	5	5
Ferrous sulphate	4/5	5	5	5	2	2	CI	5	5	4/5	5	5

A marked increase was seen in the washing, light and both perspiration fastness grades of the samples mordanted with alum, chrome, cupric sulphate and ferrous sulphate. The colour increased in the case of ferrous sulphate treated samples or alkaline perspiration fastness. The wet and dry rubbing fastness grades increased in the case of chrome treated sample, remained the same for alum and cupric sulphate treated samples and decreased in the case of ferrous sulphate treated sample as compared to fastness grade of control sample.

No staining was observed on adjacent fabrics of composite samples of washing and rubbing fastness.

From the mean score given in Table 4.14, it can be concluded that ferrous sulphate was best mordant, followed by alum, chrome and cupric sulphate (with the same overall fastness grades).

#### 4.2.4 Effect of mordants on CIE LAB values

The effect of different mordants on colour strength, chroma and intensity of the polyester samples dyed with banyan dye under optimum conditions has been presented in Table 4.22 in terms of K/S and CIE LAB values.

**Table 4.22 Effect of mordants on CIE LAB values of banyan dye on polyester.**

Mordant	L*	a*	b*	C*	K/S	Colour obtained
Control	55.840	11.140	7.366	13.355	1.88	Light greyish rosewood
Alum	56.243	10.934	7.211	13.098	2.08	Dark greyish brown red
Chrome	57.225	10.616	9.080	13.969	2.83	Brownish rosewood
Cupric sulphate	56.572	9.048	8.339	12.305	2.71	Dark greyish brown madeira
Ferrous sulphate	53.728	6.073	4.232	7.402	2.69	Rose greyish brown

The ferrous sulphate treated sample was darker while the other mordanted samples were lighter than that of shade of control sample. The control sample was more red than mordanted sample as seen by lower a\* values. The yellowness increased in the case of chrome and cupric sulphate treated samples as shown by higher b\* values while the brightness increased only in the case of chrome treated sample as depicted by higher C\* value.

The K/S values increased with the application of different types of mordants and highest colour strength was seen in the case of chrome

treated sample. The colours obtained with banyan dye under optimized conditions ranged from light greyish rosewood to dark greyish brown red.

A total of 67 shades of banyan dye were obtained in the process of optimization of different dyeing variables.

### **4.3 Dyeing with beefwood dye**

#### **4.3.1 Optimum dyeing conditions without mordanting**

Details of the ranks for the optimization of each variable, namely carrier concentration, carrier treatment time, dye extraction pH, dye extraction time, dyeing pH, dye material concentration and dyeing time and their optimum values have been presented in Table 4.23.

##### **4.3.1.1 Optimum carrier concentration**

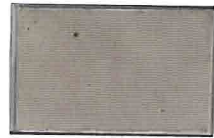
It can be seen from Table 4.23 that the colour of the dyed samples improved with the increase in the concentration of carrier, the optimum carrier concentration being 10 per cent. With further increase in concentration of carrier, the colour of dyed samples decreased.

##### **4.3.1.2 Optimum carrier treatment time**

It can be seen from the table that the dyeing improved with the increase in the time of carrier treatment, the optimum time being 60 minutes, beyond which the colour decreased again.



a



b



c



d



e

Ashoka



a



b



c



d



e

Banyan

**SS 1 : Polyester samples dyed with ashoka and banyan dyes under optimum dyeing conditions using different mordants (a - unmordanted sample, b - sample treated with ferrous sulphate, c - sample treated with cupric sulphate, d - sample treated with chrome, e - sample treated with alum).**

**Table 4.23 Optimization of carrier concentration, carrier treatment time, dye extraction pH, dye extraction time, dyeing pH, dye material concentration and dyeing time of beefwood dye on polyester on the basis of mean ranks given by a panel of judges.**

Variable	Rank	Optimum value
Carrier concentration (percent)		
5	5	
10	1	
15	4	10
20	3	
25	2	
Carrier treatment time (minutes)		
15	3	
30	2	
45	4	60
60	1	
75	5	
Dye extraction pH		
4	5	
5	6	
6	1	
7	2	6
8	4	
9	7	
10	3	
Dye extraction time (minutes)		
15	4	
30	3	
45	1	
60	2	45
75	7	
90	5	
105	6	
Dyeing pH		
4	5	
5	4	
6	3	
7	1	7
8	2	
9	6	
10	7	
Dye material concentration (g/g)		
0.5	7	
1	3	
2	1	
3	6	2
4	2	
5	4	
6	5	
Dyeing time (minutes)		
15	7	
30	6	
45	5	
60	1	60
75	4	
90	3	
105	2	

#### **4.3.1.3 Optimum dye extraction pH**

It can be seen from the Table 4.23 that the extraction of dye increased with the increase in the pH and extraction was optimum at pH 6, i.e. slightly acidic condition. Further increase in pH decreased the extraction of dye, i.e. in alkaline conditions.

#### **4.3.1.4 Optimum dye extraction time**

The table indicates that a duration of 45 minutes was adjudged the best for maximum extraction of the dye. Extraction for longer period was not found helpful in increasing the dye extraction. This may be because after a particular time some substances other than dye also start to leach out from the plant material which may hinder the dye absorption. Similar observations were made by Singh et al (1993a) in the case of madder dye.

#### **4.3.1.5 Optimum dyeing pH**

It is clear from the table that as the pH increased, the dyeing improve and was the best at neutral pH. Further increase in pH resulted in duller colours. The neutral pH, i.e. pH 7 was, therefore, adjudged optimum for application of beefwood dye.

#### **4.3.1.6 Optimum dye material concentration**

The mordanting method and mordant concentration were optimized on the basis of overall fastness ratings of beefwood dye dyed polyester employing the carrier concentration, carrier treatment time, dye extraction

pH, dye extraction time, dyeing pH, dye material concentration and dyeing time optimized earlier without mordanting. The fastness grades for optimization of mordanting method and mordant concentration are given in Tables 4.24 and 4.25, respectively. The optimum mordanting and mordant concentration for the four mordants have been presented in Table 4.26.

**Table 4.24 Optimization of mordanting method for beefwood dye on polyester on the basis of fastness grades.**

Mordant	Mor- danting method	WF			LF	RF		PF					MS	Rank	
		CC	CSC	CSP	CC	W		Al		Ac					
						CSC	CSC	CC	CSP	CSW	CC	CSP			CSW
Alum	Pre	1	5	5	3/4	2/3	2/3	4	5	5	4/5	5	5	4.00	3
	Sim	2	5	5	3/4	3/4	3/4	4	5	5	4	5	5	4.20	2
	Post*	3	5	5	3/4	3/4	2/3	4/5	5	5	4/5	5	5	4.29	1
Chrome	Pre	3	5	5	3/4	3	3/4	2	5	5	2	5	5	3.91	3
	Sim	4	5	5	3/4	4	4	3	5	5	4	5	5	4.37	2
	Post*	4	5	5	3/4	3/4	3	4/5	5	5	4/5	5	5	4.41	1
Cupric sulphate	Pre	2/3	5	5	CC	2	2/3	2/3	5	5	2/3	5	5	3.81	3
	Sim	2	5	5	3/4	3/4	3/4	3/4	5	5	4/5	5	5	4.16	2
	Post*	2	5	5	CI	4	4	4	5	5	4/5	5	5	4.45	1
Ferrous sulphate	Pre*	4/5	5	5	3/4	2	1/2	4	5	5	4/5	5	5	4.16	1
	Sim	1	5	5	3/4	3/4	3	4/5	5	5	2	5	5	3.95	3
	Post	1	5	5	3/4	3/4	3/4	4	5	5	4	5	5	4.12	2

\*Optimum mordanting method

#### 4.3.2.1 Optimum mordanting method

The fastness ratings for different methods of mordanting have been presented in Table 4.24. The table reveals that the post-mordanting method of mordant application was the best for all mordants (highest mean score) except ferrous sulphate for which pre-mordanting method gave the best fastness grades.

**Table 4.25 Optimization of mordant concentration for beefwood dye on polyester on the basis of fastness grades.**

Mordant	Mordant concentration	WF			LF		RF		PF			MS			Rank	
		CC	CSC	CSP	CC	W		D		Al			Ac			
						CSC	CSC	CC	CSP	CSW	CC	CSP	CSW			
Alum	5	3/4	5	5	2	3/4	3	4/5	5	5	4/5	5	5	4.25	2.5	
	10*	3	5	5	3/4	3/4	2/3	4/5	5	5	4/5	5	5	4.29	1	
	15	3	5	5	2	3/4	2/3	4/5	5	5	4/5	5	5	4.16	5	
	20	3	5	5	2	3/4	3	4/5	5	5	4/5	5	5	4.20	4	
	25	3	5	5	2	3/4	3/4	4/5	5	5	4/5	5	5	4.25	2.5	
Chrome	1	4	5	5	3	2/3	2/3	4/5	5	5	4/5	5	5	4.25	4.5	
	2*	4	5	5	3/4	3/4	3	4/5	5	5	4/5	5	5	4.41	1	
	3	4	5	5	3	3	2/3	4/5	5	5	4/5	5	5	4.29	3	
	4	4/5	5	5	3	3	2/3	4	5	5	4	5	5	4.25	4.5	
	5	4/5	5	5	3	3/4	3	4	5	5	4	5	5	4.33	2	
Cupric sulphate	1	2	5	5	3	3	3	3/4	5	5	3	5	5	3.95	5	
	2*	2	5	5	CI	4	4/5	4	5	5	4/5	5	5	4.45	1	
	3	3/4	5	5	4	3	3	4/5	5	5	4/5	5	5	4.37	3	
	4	4	5	5	5	2/3	2/3	4/5	5	5	4/5	5	5	4.41	2	
	5	4	5	5	4	2/3	2/3	4	5	5	4	5	5	4.25	4	
Ferrous sulphate	1	CI	5	5	4	3/4	2	4/5	5	5	4	5	5	4.00	5	
	2	4/5	5	5	3/4	2	1/2	4	5	5	4/5	5	5	4.16	4	
	3	4/5	5	5	3	3	2	4	5	5	CI	5	5	4.22	2	
	4	3	5	5	3	3	3	CI	5	5	CI	5	5	4.20	3	
	5*	4	5	5	2	3	3	CI	5	5	5	5	5	4.27	1	

\*Optimum mordant concentration

#### 4.3.2.2 Optimum mordant concentration

The fastness grades for different concentrations of mordants have been given in Table 4.3. The table makes it clear that the overall fastness grades increased with increase in mordant concentration upto certain level. Beyond that concentration the fastness ratings decreased. For ferrous sulphate, the fastness grades increased with increase in concentration. The optimum concentration of alum, chrome, cupric sulphate and ferrous sulphate were 10, 2, 2 and 5 per cent, respectively, (Table 4.26).

**Table 4.26 Optimum mordanting conditions for dyeing polyester with beefwood dye.**

Mordant	Mordating method	Mordant concentration (percent)
Alum	Post	10
Chrome	Post	2
Cupric sulphate	Post	2
Ferrous sulphate	Pre	5

### 4.3.3. Effect of dyeing parameters on the fastness of beefwood dye

#### 4.3.3.1 Effect on carrier concentration

The washing fastness, light fastness, rubbing fastness and perspiration fastness grades of beefwood dye on polyester at different carrier concentrations have been presented in Table 4.27.

**Table 4.27 Effect of carrier concentration on fastness of beefwood dye on polyester.**

Carrier concentration (percent)	WF			LF		RF		PF				
								Al			Ac	
	CC	CSC	CSP	CC	CSC	CSC	CC	CSP	CSW	CC	CSP	CSW
5	¾	5	5	2	¾	3	4	5	5	¾	5	5
10	¾	5	5	2	¾	3	4	5	5	4/5	5	5
15	3	5	5	1	3	3	4	5	5	3	5	5
20	3	5	5	2	3	3	4/5	5	5	4	5	5
25	3	5	5	1	¾	¾	4/5	5	5	4/5	5	5

The washing fastness of the dye remained constant till 10 per cent carrier concentration. It then decreased to 3 at 15 per cent carrier concentration and thereafter remained constant. The washing fastness grades indicate good fastness. No staining was observed on adjacent cotton and polyester fabrics.

The light fastness remained constant till 10 per cent carrier concentration. Then it decreased and increased in alternative manner on further increase in carrier concentration. The light fastness grades indicate poor to fair fastness.

The wet rubbing fastness was good with grade  $\frac{3}{4}$  at all concentrations except at 5 and 25 carrier concentration where it decreased slightly. The dry rubbing fastness remained constant till 20 per cent carrier concentration and then showed a slight increase.

The alkaline perspiration fastness increased slightly after remaining constant upto 15 per cent carrier concentration and then again remained constant. The acidic perspiration fastness showed a increase initially and decreased at 15 per cent carrier concentration. With further increase in carrier concentration acidic perspiration fastness increased. No staining was observed on adjacent wool and polyester fabrics of composite samples.

#### **4.3.3.2 Effect of carrier treatment time**

Table 4.28 shows the washing, light, rubbing and perspiration fastness of beefwood dye on polyester samples treated with carrier for five different time periods.

The washing fastness increased initially and then decreased with increase in carrier treatment time after 45 minutes. The washing fastness

was good with grades between 3–4. No staining was noticed on adjacent cotton and polyester fabrics.

**Table 4.28 Effect of carrier treatment time on fastness of beefwood dye on polyester.**

Carrier treatment time (minutes)	WF			LF	RF		PF					
	CC	CSC	CSP	CC	W	D	Al			Ac		
					CSC	CSC	CC	CSP	CSW	CC	CSP	CSW
15	¾	5	5	2	3	3	¾	5	5	¾	5	5
30	4	5	5	1	¾	3	3	5	5	4	5	5
45	4	5	5	2	¾	3	4	5	5	¾	5	5
60	¾	5	5	2	¾	3	4	5	5	4/5	5	5
75	3	5	5	1	3	3	¾	5	5	2	5	5

The light fastness did not show a fixed pattern of increase and decrease in grades. It decreased initially from 2 for 15 minutes to 1 for 30 minutes carrier treatment time. It then again increased at 45 minutes and then decreased at 75 minutes.

The wet rubbing fastness initially increased and then remained constant till 60 minutes carrier treatment time. It then again decreased for 75 minutes carrier treatment time. The dry rubbing fastness remained the same indicating no effect of carrier treatment time. The rubbing fastness was good with grades between 3-¾.

The alkaline perspiration fastness decreased initially with increase in carrier treatment time. It increased to 4 for 45 minutes carrier treatment time and then decreased to ¾ for 75 minutes carrier treatment time. The acidic perspiration showed a pattern of consecutive increase and decrease

in fastness with increase in carrier treatment time with grades ranging between 2 - 4/5. No staining was found on adjacent wool and polyester fabrics.

#### 4.3.3.3 Effect of dyeing pH

The fastness grades of washing, light, rubbing and perspiration of ashoka dye on polyester at different pH values are given in Table 4.29.

**Table 4.29 Effect of dyeing pH on fastness of beefwood dye on polyester.**

Dyeing pH	WF			LF	RF		PF			Ac		
	CC	CSC	CSP	CC	W	D	CC	CSP	CSW	CC	CSP	CSW
					CSC	CSC						
4	3	5	5	1	3/4	4	2/3	5	5	2/3	5	5
5	3	5	5	1	3/4	3/4	4	5	5	4	5	5
6	3	5	5	1	3/4	4	3	5	5	3	5	5
7	3/4	5	5	2	3/4	3	4	5	5	4/5	5	5
8	2	5	5	2	4	4/5	4	5	5	3/4	5	5
9	1	5	5	2	3/4	4/5	4/5	5	5	4/5	5	5
10	1	5	5	1	3	4/5	4	5	5	4	5	5

The washing fastness remained constant upto pH 6. It then showed a slight increase at pH 7. Thereafter it decreased with increase in dyeing pH. The washing fastness of dyed samples was poor to good. No staining was found on adjacent polyester and cotton fabrics.

The light fastness remained constant upto pH 6. It then increased at pH 7 and again remained constant upto pH 9. Then it showed a decrease at pH 10. The light fastness was poor to fair with grades ranging between 1-2.

The wet rubbing fastness remained constant at  $\frac{3}{4}$  except at pH 8 where it increased to 4 and pH 10 where it decreased to 3. The dry rubbing fastness decreased and increased with every alternating value of dyeing pH upto 8. Thereafter it remained the same on further rise in pH of dyeing.

The alkaline perspiration fastness increased initially from  $\frac{2}{3}$  at pH 4 to 4 at pH 5. It then remained almost the same with increase in pH except at pH 6 where it decreased to 3 and pH 9 where it increased slightly to  $\frac{4}{5}$ . The acidic perspiration showed a consecutive increasing and decreasing grades ranging between  $\frac{2}{3}$  –  $\frac{4}{5}$  with increase in dyeing pH. No staining was noticed on adjacent wool and polyester fabrics of composite samples.

#### **4.3.3.4. Effect of dye material concentration**

Table 4.30 indicates the fastness grades of beefwood dye on polyester for washing, light, rubbing and perspiration for different concentrations of dye material.

The washing fastness increased initially with increase in dye material concentration till 3g/g concentration. It then decreased at 4g/g concentration and thereafter remained the same with further increase in dye material concentration. No staining was observed on adjacent cotton and polyester fabrics of composite samples.

**Table 4.30 Effect of dye material concentration on fastness of beefwood dye on polyester.**

Dye concentration (g/g)	WF			LF	RF			PF				
	CC	CSC	CSP	CC	W	D	CC	Al		Ac		
					CSC	CSC		CSP	CSW	CC	CSP	CSW
0.5	1	5	5	1	3	3	3	5	5	3	5	5
1	3	5	5	1	2/3	2/3	4	5	5	3	5	5
2	3/4	5	5	2	3/4	3	4	5	5	4/5	5	5
3	4	5	5	2	3	3/4	4	5	5	4/5	5	5
4	3	5	5	1	3	3/4	3	5	5	4/5	5	5
5	3	5	5	1	3	3	4/5	5	5	4/5	5	5
6	3	5	5	1	3/4	3/4	4	5	5	4/5	5	5

The light fastness remained constant at 1 for all dye material concentrations except for 2g/g and 3g/g dye material concentrations where it increased to 2.

The wet and dry rubbing fastness showed similar trend of increase and decrease in grades. Both decreased initially and then increased at 2g/g. Then wet rubbing fastness remain constant till 5g/g after slight decrease at 3 g/g and then increased at 6g/g concentration while dry fastness decreased at 5g/g concentration and then again increased at 6g/g concentration.

The alkaline perspiration fastness initially showed an increase and then remained constant till 3g/g of dye material concentration. It then decreased and increased in alternative manner on further increase in dye material concentration. The acidic perspiration fastness remained constant for all carrier concentrations after increasing at 2 g/g concentration. No

staining was observed on adjacent wool and polyester fabrics of composite samples.

#### 4.3.3.5 Effect of dyeing time

The washing, light, rubbing and perspiration fastness grades of beefwood dye on polyester for different time period have been presented in Table 4.31.

The washing fastness increased initially and then remained the same except for 60 minutes of dyeing time where it is slightly higher. The washing fastness of the dyed samples was good. No staining was noticed on adjacent polyester and cotton fabrics.

**Table 4.31 Effect of dyeing time on fastness of beefwood dye on polyester.**

Dyeing time (minutes)	WF			LF	RF		PF					
					W	D	Al			Ac		
	CC	CSC	CSP	CC	CSC	CSC	CC	CSP	CSW	CC	CSP	CSW
15	1	5	5	1	3	3	3	5	5	3	5	5
30	3	5	5	1	2/3	2/3	4	5	5	3	5	5
45	3	5	5	2	2/3	2	3/4	5	5	4	5	5
60	3/4	5	5	2	3/4	3	4	5	5	4/5	5	5
75	3	5	5	1	3	3/4	3	5	5	4/5	5	5
90	3	5	5	1	3	3	4/5	5	5	4/5	5	5
105	3	5	5	1	3/4	3/4	4	5	5	4/5	5	5

The light fastness remained constant at 1 for all dyeing time except for 45 and 60 minutes of dyeing time where it increased to 2.

The rubbing fastness showed no fixed increasing and decreasing pattern. The wet rubbing fastness decreased initially at 30 minutes of

dyeing time and increased at 60 minutes. It then again decreased at 75 minutes and increased slightly at 105 minutes. The dry rubbing fastness decreased initially slightly and increased at 60 minutes and then shows consecutive increase and decrease with increase in dyeing time.

The alkaline perspiration fastness showed consecutive increasing and decreasing trend of grades with increase in dyeing time. The acidic perspiration fastness increased upto an hour and then remained constant with increase in dyeing time. No staining was observed on adjacent wool and polyester fabrics of composite samples.

#### **4.3.3.6. Effect of mordanting method**

Table 4.30 depicts the washing, light, rubbing and perspiration fastness of beefwood dye on polyester employing different mordants and the three mordanting methods with each mordant.

##### **4.3.3.6.1 Alum**

It can be seen from the table that on mordanting with alum, the fastness grades of washing was the best with pre-mordanting method, followed by that for simultaneous and post-mordanting methods. Light fastness grades were the same with there different mordanting methods. Wet rubbing fastness was minimum for pre-mordanted sample while dry rubbing fastness was the best for simultaneous mordanting method and remained the same with other two methods of mordanting. The acidic and

alkaline perspiration fastness grades were similar for pre and simultaneous mordanted samples and pre and post-mordanted samples, respectively.

#### **4.3.3.6.2 Chrome**

In the case of chrome mordanted samples, washing fastness grades were the same for simultaneous and post-mordanted samples and was minimum for pre-mordanted sample. Light fastness grades of three samples mordanted in different ways were the same. Wet rubbing fastness was maximum for simultaneous mordanted sample, followed by that for post and pre-mordanted samples. The fastness grade of dry rubbing was maximum for simultaneous mordanted sample, followed by that for pre and post-mordanted samples. The acidic and alkaline perspiration fastness both were maximum with pre-mordanting method, followed by that for simultaneous and post-mordanted samples (in that order).

#### **4.3.3.6.3 Cupric sulphate**

The fastness grades of washing and acidic perspiration for change in colour were same in the case of simultaneous and post-mordanted samples but were the best in the case of washing and decreased in the case of acidic perspiration in the case of pre-mordanted samples. The colour of dyed fabric treated with pre and post-mordanting methods changed and increased, respectively on exposure to light. Dry and wet rubbing and alkaline perspiration fastness grades were the best for post-mordanted

samples, followed by that for simultaneous and pre-mordanted samples whose grades decreased (in that order).

#### **4.3.3.6.4 Ferrous sulphate**

It is clear from the table that on mordanting with ferrous sulphate, the washing fastness grades for change in colour was the best in the case of pre-mordanted sample, followed by that for simultaneous and post-mordanted samples. The light fastness grades were the same with all three mordanting methods. Wet rubbing fastness was minimum in the case of pre-mordanted sample and was the same in the case of simultaneous and post-mordanted samples. The fastness grades of dry rubbing was the best for pre-mordanted samples, followed by that for simultaneous and post-mordanted samples. Alkaline perspiration fastness was the best for simultaneous mordanted sample and decreased slightly in the case of both pre and post-mordanted samples. Acidic perspiration fastness was the best with pre-mordanting method, followed by that for post and simultaneous mordanting methods (in that order).

No staining was noticed on adjacent fabrics of composite samples used in washing and perspiration fastness in the case of all the mordants.

#### **4.3.3.7. Effect of mordant concentration**

The washing, light, rubbing and perspiration fastness grades of polyester samples dyed with beefwood dye using five different mordant concentrations have been presented in Table 4.25.

##### **4.3.3.7.1 Alum**

The fastness grades decreased slightly at 10 per cent mordant concentration and remained the same on further increase in the concentration of mordant. The light fastness grades also remained unchanged except at 10 per cent mordant concentration where the fastness was the best. Dry rubbing fastness decreased at 10 per cent and increased with further increase in mordant concentration. The fastness grades of wet rubbing, alkaline and acidic perspiration showed no change with the increase in concentration of mordant.

##### **4.3.3.7.2 Chrome**

It can be seen from the table that on mordanting with chrome, the washing fastness remained the same upto 3 per cent concentration of mordant and increased slightly at 4 per cent concentration. Light fastness grades remained unchanged except at 2 per cent mordant concentration where slight increase was observed. Wet and dry rubbing fastness grades increased at 2 per cent concentration, decreased at 3 per cent concentration and increased on further increase in the mordant concentration. Acidic and

alkaline fastness remained the same upto 3 per cent concentration of mordant which decreased slightly with increase in mordant concentration.

#### **4.3.3.7.3 Cupric sulphate**

Washing fastness grades increased with increase in concentration of mordants. The colour of dyed sample decreased on exposure to light when 2 per cent mordant was applied. The light fastness grades improved upto 4 per cent mordant concentration and then decreased on further increase in mordant concentration. The wet and dry rubbing fastness showed an increase at 2 per cent and decreased on further increase in concentration of mordant. Acidic and alkaline perspiration fastness increased with increase in mordant concentration upto 4 per cent but decreased on further increase in concentration of mordant.

#### **4.3.3.7.4 Ferrous sulphate**

In the case of ferrous sulphate treated samples, the colour of the dyed sample increased on washing at 1 per cent and with increase in mordant concentration, fastness decreased upto 4 per cent concentration after which it again showed an improvement. Light fastness decreased as the concentration of mordant increased. Wet and dry rubbing fastness decreased at 2 per cent mordant concentration but increased on further increase in concentration of mordant. Alkaline perspiration fastness decreased while acidic perspiration fastness increased with increase in

concentration of mordant and colour of dyed samples increased in both alkaline and acidic perspiration solution treated samples at higher mordant concentrations.

No staining was noticed on adjacent fabrics of composite samples of both washing and perspiration fastness in the case of all the mordants.

#### 4.3.3.8 Effect of the type of mordant

The washing, light rubbing and perspiration fastness grade for the polyester sample dyed with beefwood dye using different mordants under optimum conditions have been compared with those of control (unmordanted) sample in the Table 4.32.

**Table 4.32 Effect of mordant type on fastness of beefwood dye on polyester.**

Mordant	WF			LF	RF		PF						
	CC	CSC	CSP	CC	W	D	Al			Ac			
					CSC	CSC	CC	CSP	CSW	CC	CSP	CSW	
Control	¾	5	5	2	¾	3	4	5	5	5	4/5	5	5
Alum	3	5	5	¾	¾	2/3	4/5	5	5	5	4/5	5	5
Chrome	4	5	5	¾	¾	3	4/5	5	5	5	4/5	5	5
Cupric sulphate	2	5	5	CI	4	4/5	4	5	5	5	4/5	5	5
Ferrous sulphate	4	5	5	2	3	3	CI	5	5	5	5	5	5

The washing fastness improved on application of chrome and ferrous sulphate and decreased in the case of alum and cupric sulphate in comparison with fastness grade of control sample.

The light fastness grade was the same in the case of ferrous sulphate and increased in the case of alum and chrome treated samples as compared with grade of control samples. The colour of sample treated with cupric sulphate increased on exposure to light.

The wet rubbing fastness remained the same on application of alum and chrome, increased and decreased in the case of cupric sulphate and ferrous sulphate treated samples, respectively when compared with fastness of control sample. While the dry rubbing fastness remained the same in the case of chrome and ferrous sulphate, increased and decreased in the case cupric sulphate and alum treated samples, respectively.

The acidic perspiration remained the same in the case of alum, chrome and cupric sulphate treated samples and increased in the case of ferrous sulphate treated sample. The alkaline perspiration fastness increased in the case of alum and chrome treated samples, remained the same in the case of cupric sulphate treated sample and the colour increased in the case of ferrous sulphate treated sample when compared with control sample.

No staining was observed on adjacent fabrics of composite samples used in washing and perspiration fastness.

From the mean scores of the fastness grades as given in Table 4.25, it can be concluded that cupric sulphate was the best mordant, followed by chrome, alum and ferrous sulphate.

#### 4.3.4 Effect of mordants on CIE LAB values

The effect of different mordants on colour strength, chrome and intensity of the polyester samples dyed with beefwood dye under optimum conditions has been presented in Table 4.33 in terms of K/S and CIE LAB values.

**Table 4.33 Effect of mordant on CIE LAB values of beefwood dye on polyester.**

Mordant	L*	a*	b*	C*	K/S	Colour obtained
Control	63.513	6.801	7.472	10.104	1.32	Brownish grey madeira
Alum	64.731	7.667	10.38	12.909	1.90	Greyish fawn
Chrome	65.527	7.139	11.575	13.599	2.24	Light greyish fawn
Cupric sulphate	64.767	6.834	10.983	12.936	2.23	Dull orangish fawn
Ferrous sulphate	63.387	6.233	9.017	10.962	1.86	Greyish brown madeira

The higher L\* values depicted lighter shades of mordanted samples except ferrous sulphate treated samples which showed darker shade as compared to control sample. Similarly all the mordanted samples except ferrous treated sample, were more red than control sample. All the mordanted samples were more yellow and brighter than the control sample as seen by higher b\* and C\* values.

The strength of colour increased on application of mordants as seen by higher K/S values for every mordanted sample than the value of control sample. The colours obtained with beefwood under optimized conditions ranged from brownish grey madeira to greyish fawn.

A total of 67 shades of beefwood dye were obtained in the process of optimization of different dyeing variables.

#### **4.4 Dyeing with Flame of forest dye**

##### **4.4.1 Optimum dyeing conditions without mordanting**

Details of the ranks for the optimization of each variable, namely carrier concentration, carrier treatment time, dye extraction pH, dye extraction time, dyeing pH, dye material concentration and dyeing time and their optimum values have been presented in Table 4.34.

###### **4.4.1.1 Optimum carrier concentration**

It can be seen from the Table 4.34 that the colour of the dyed samples improved with the increase in the concentration of carrier, the optimum carrier concentration being 15 per cent beyond which it decreased.

**Table 4.34 Optimization of carrier concentration, carrier treatment time, dye extraction pH, dye extraction time, dyeing pH, dye material concentration and dyeing time of flame of forest dye on polyester on the basis of mean ranks given by a panel of judges.**

Variable	Rank	Optimum value
<b>Carrier concentration (percent)</b>		
5	5	
10	4	
15	1	15
20	3	
25	2	
<b>Carrier treatment time (minutes)</b>		
15	3	
30	4	
45	5	60
60	1	
75	2	
<b>Dye extraction pH</b>		
4	6	
5	5	
6	4	
7	3	10
8	7	
9	2	
10	1	
<b>Dye extraction time (minutes)</b>		
15	5	
30	2	
45	1	
60	4	45
75	3	
90	6	
105	7	
<b>Dyeing pH</b>		
4	1	
5	2	
6	3	
7	4	4
8	5	
9	6.5	
10	6.5	
<b>Dye material concentration (g/g)</b>		
0.5	7	
1	5.5	
2	4	
3	1	3
4	2.5	
5	2.5	
6	5.5	
<b>Dyeing time (minutes)</b>		
15	7	
30	6	
45	4	
60	2	90
75	3	
90	1	
105	5	

#### **4.4.1.2 Optimum carrier treatment time**

It can be seen from the table that the dyeing improved with the increase in the time of carrier treatment, the optimum time being 60 minutes, beyond which the colour decreased again.

#### **4.4.1.3 Optimum dye extraction pH**

It can be seen from the Table 4.34 that the extraction of dye from the bark powder increased with increase in the pH and optimum pH for dye extraction was 10. Singh et al (1996) also found that increasing the amount of alkali upto 0.3 g/g fabric in the extraction bath increased the extraction of *kamala* dye.

#### **4.4.1.4 Optimum dye extraction time**

Table indicates that a duration of 45 minutes was adjudged the best for maximum extraction of the dye. Extraction for longer period was not found helpful in increasing the dye extraction. This may be because after a particular time some substances other than dye also start to leach out from the plant material which may hinder the dye absorption. Similar observations were made by Singh et al (1993a) in the case of madder dye.

#### **4.4.1.5 Optimum dyeing pH**

It is clear from the table that the best dyeing was obtained at pH 4, i.e. in acidic conditions. Further increase in pH, i.e. as alkalinity of dyeing medium increased, the dyeing decreased and no colour was absorbed at pH

9 and 10. Patel and Agarwal (2000) also reported 4.5 as the optimum pH for dyeing nylon with babool dye employing high temperature high pressure method.

#### **4.4.1.6 Optimum dye material concentration**

The table shows that the optimum dye material concentration was 3 g per gram of polyester. The use of increased amount of dye resulted in reduced dye extraction. Similar results were reported by Vednere et al (1990) in the case of chir bark on wools.

#### **4.4.1.7 Optimum dyeing time**

The colour of polyester improved with increase in the duration of dyeing as depicted in Table 4.34 and the optimum dyeing time was adjudged at 90 minutes. The colour of dyed samples decreased on further increase in dyeing time.

#### **4.4.2 Optimum mordanting conditions**

The mordanting method and mordant concentration were optimized on the basis of overall fastness ratings of flame of forest dye dyed polyester employing the carrier concentration, carrier treatment time, dye extraction pH, dye extraction time, dyeing pH, dye material concentration and dyeing time optimized earlier without mordanting. The fastness grades for optimization of mordanting method and mordant concentration are given in Tables 4.35 and 4.36, respectively. The optimum mordanting and

mordant concentration for the four mordants have been presented in Table 4.37.

**Table 4.35 Optimization of mordanting method for flame of forest dye on polyester on the basis of fastness grades.**

Mordant	Mor-danting method	WF			LF			RF			PF			MS	Rank
		CC	CSC	CSP	CC	W		D	Al		Ac				
						CSC	CSC		CC	CSP	CSW	CC	CSP		
Alum	Pre*	CI	5	5	CC	2/3	2/3	4	5	5	4/5	5	5	4.35	1
	Sim	CI	5	5	CI	3	2	4	5	5	4	5	5	4.30	2
	Post	CI	5	5	2	2/3	1/2	5	5	5	5	5	5	4.18	3
Chrome	Pre*	CC	5	5	CC	3	2/3	3	5	5	4	5	5	4.25	1
	Sim	4	5	5	3	2/3	2	3	5	5	2/3	5	5	3.91	3
	Post	3	5	5	2	2	3	4	5	5	3/4	5	5	3.95	2
Cupric sulphate	Pre	CI	5	5	3	2	1/2	3	5	5	3/4	5	5	3.90	2.5
	Sim	CC	5	5	CC	1/2	1/2	3	5	5	3	5	5	3.90	2.5
	Post*	CC	5	5	CC	2	2	4	5	5	3/4	5	5	4.15	1
Ferrous sulphate	Pre*	CI	5	5	CC	2/3	2/3	4	5	5	4	5	5	4.30	1
	Sim	CC	5	5	CC	2/3	2	2/3	5	5	4	5	5	4.10	2
	Post	3	5	5	2	2/3	2	3	5	5	2/3	5	5	3.75	3

\*Optimum mordanting method

#### 4.4.2.1 Optimum mordanting method

The fastness ratings for different methods of mordanting have been presented in Table 4.35. The table reveals that the pre-mordanting method of mordant application was the best (highest mean score for all the mordants except cupric sulphate for which post-mordanting method gave the best fastness grades. Vednere et al (1990) also reported pre-mordanting as the best method for conferring good fastness to chir bark dye on wool.

**Table 4.36 Optimization of mordant concentration for flame of forest dye on polyester on the basis of fastness grades.**

Mordant	Mordant concentration	WF			LF	RF		PF					MS	Rank	
		CC	CSC	CSP	CC	W	D	Al			Ac				
						CSC	CSC	CC	CSP	CSW	CC	CSP			CSW
Alum	5	CI	5	5	CC	2	2	3	5	5	3/4	5	5	4.05	5
	10	CI	5	5	CC	2/3	2/3	4	5	5	4/5	5	5	4.35	2.5
	15	CI	5	5	CC	2	2	4	5	5	4/5	5	5	4.25	4
	20*	CC	5	5	CC	2/3	2/3	4/5	5	5	4/5	5	5	4.40	1
	25	CC	5	5	CC	2/3	2/3	4/5	5	5	4	5	5	4.35	2.5
Chrome	1	CC	5	5	CC	2	2	3	5	5	3	5	5	4.00	5
	2	CC	5	5	CC	3	2/3	3	5	5	4	5	5	4.25	3
	3	CC	5	5	CC	2/3	2/3	3	5	5	3	5	5	4.10	4
	4	CC	5	5	CC	2/3	2/3	4	5	5	4	5	5	4.30	2
	5*	CC	5	5	CC	2/3	2/3	4/5	5	5	4/5	5	5	4.40	1
Cupric sulphate	1*	CC	5	5	CC	2/3	2/3	4/5	5	5	4/5	5	5	4.40	1
	2	CC	5	5	CC	2	2	4	5	5	3/4	5	5	4.15	5
	3	CC	5	5	CC	2	2/3	4/5	5	5	4/5	5	5	4.35	2
	4	CC	5	5	CC	2	2	4	5	5	CI	5	5	4.22	4
	5	CC	5	5	CC	2	2	4/5	5	5	4	5	5	4.25	3
Ferrous sulphate	1*	CI	5	5	CC	2/3	2/3	4/5	5	5	4/5	5	5	4.40	1
	2	CI	5	5	CC	2/3	2/3	4	5	5	4	5	5	4.30	4
	3	CI	5	5	CC	2/3	2	4	5	5	4/5	5	5	4.30	4
	4	CC	5	5	CC	2/3	2/3	4/5	5	5	CI	5	5	4.38	2
	5	CC	5	5	CI	2	2	4/5	5	5	4/5	5	5	4.30	4

\*Optimum mordant concentration

#### 4.4.2.2 Optimum mordant concentration

The fastness grades for different concentration of mordants have been given in Table 4.36. The table makes it clear that the overall fastness of cupric sulphate and ferrous sulphate decreased with increase in concentration of mordants while the reverse was found true in the case of alum and chrome treated samples, i.e. fastness grades increased with increase in concentration of mordants. The optimum concentrations were

20, 5, 1 and 1 per cent for alum, chrome, cupric sulphate and ferrous sulphate, respectively (Table 4.37).

**Table 4.37 Optimum mordanting conditions for dyeing polyester with flame of forest dye.**

Mordant	Mordating method	Mordant concentration (percent)
Alum	Pre	20
Chrome	Pre	5
Cupric sulphate	Post	1
Ferrous sulphate	Pre	1

#### 4.4.3 Effect of dyeing parameters on the fastness of flame of forest dye

##### 4.4.3.1 Effect of carrier concentration

The washing fastness light, light fastness, rubbing fastness and perspiration fastness grades of the Flame of forest dye on polyester at different carrier concentrations have been presented in Table 4.38.

**Table 4.38 Effect of carrier concentration on fastness of flame of forest dye on polyester.**

Carrier concentration (percent)	WF			LF		RF			PF			
	W		D		Al		Ac					
	CC	CSC	CSP	CC	CSC	CSC	CC	CSP	CSW	CC	CSP	CSW
5	4	5	5	2	2/3	3/4	2/3	5	5	3/4	5	5
10	3	5	5	3	2/3	3/4	3	5	5	3	5	5
15	3/4	5	5	2	3	3/4	4	5	5	4	5	5
20	3/4	5	5	2	1/2	2/3	3/4	5	5	3/4	5	5
25	4	5	5	2	2/3	2	2/3	5	5	3	5	5

The washing fastness of the dye showed a decrease initially with increase in carrier concentration and then increased with further increase in

carrier concentration. No staining was observed on adjacent fabrics of composite samples.

The light fastness remained the same for all carrier concentration except at 10 per cent carrier concentration where it increased to 3 from 2 at rest all concentrations.

The wet rubbing fastness showed an initial increase upto 15 per cent of carrier concentration. It then decreased to  $\frac{1}{2}$  at 20 per cent carrier concentration and then increased at 25 per cent carrier concentration. The dry rubbing fastness remained constant upto 15 per cent carrier concentration and then decreased with increase in carrier concentration.

The alkaline perspiration fastness increased initially upto 15 per cent carrier concentration and then decreased with increase in carrier concentration. The acidic perspiration fastness first decreased to 3 at 10 per cent carrier concentration from  $\frac{3}{4}$  at 5 per cent carrier concentration. It then increased to 4 at 15 per cent carrier concentration. Thereafter it showed a decrease with increase in carrier concentration. No staining was observed on adjacent fabrics of composite samples.

#### **4.4.3.2 Effect of carrier treatment time**

Table 4.39 shows the washing, light, rubbing and perspiration fastness of flame of forest dye on polyester samples treated with carrier for five different time periods.

**Table 4.39 Effect of carrier treatment time on fastness of flame of forest dye on polyester.**

Carrier treatment time (minutes)	WF			LF		RF			PF			
	CC	CSC	CSP	CC	CSC	W	D	Al		Ac		
						CC	CSP	CSW	CC	CSP	CSW	
15	CC	5	5	2	3	4/5	4/5	5	5	4	5	5
30	CC	5	5	CC	¾	4/5	4/5	5	5	4	5	5
45	4/5	5	5	2	3	4	4	5	5	¾	5	5
60	¾	5	5	2	3	¾	4	5	5	4	5	5
75	¾	5	5	2	3	¾	¾	5	5	4	5	5

The hue of colour changed on washing upto 30 minutes of carrier treatment time after which the washing fastness grades decreased with increase in carrier treatment time. No staining was found on the adjacent cotton and polyester fabrics of composite samples.

The light fastness remained the same for all time periods except at 30 minutes where the hue of colour changed on exposure to light.

The wet rubbing fastness remained the same for all carrier concentration time except at 30 minutes where it showed a slight increase. The dry rubbing fastness decreased from 4/5 to ¾ with increase in carrier treatment time.

The alkaline perspiration fastness decreased gradually with increase in carrier treatment time from 4/5 to 3/4 while acidic perspiration fastness remained the same for all carrier treatment time periods except at 45 minutes where it decreased slightly. No staining was noticed on adjacent wool and polyester fabrics of composite samples.

#### 4.4.3.3 Effect of dyeing pH

The fastness grades of washing, light, rubbing and perspiration of flame of forest dye on polyester at different pH values are given in Table 4.40.

**Table 4.40 Effect of dyeing pH on fastness of flame of forest dye on polyester.**

Dyeing pH	WF			LF	RF		PF					
	CC	CSC	CSP	CC	W	D	Al			Ac		
					CSC	CSC	CC	CSP	CSW	CC	CSP	CSW
4	CC	5	5	CC	2/3	2/3	4/5	5	5	4/5	5	5
5	CC	5	5	CC	2/3	2/3	4	5	5	4	5	5
6	¾	5	5	2	¾	¾	4/5	5	5	4	5	5
7	¾	5	5	2	3	¾	4	5	5	4	5	5
8	2/3	5	5	1	¾	¾	4	5	5	3	5	5
9	3	5	5	1	4	4	4	5	5	4	5	5
10	3	5	5	1	4/5	4/5	4	5	5	4	5	5

The hue of colour changed on washing up to pH 5. After that it showed a decrease in fastness grade at pH 8 and then increased to 3 at pH 9. No staining was found on the adjacent cotton and polyester fabrics.

The hue of colour changed on exposure to light upto pH 5. Thereafter it showed a decrease in grades with increase in pH. Finally after 8 pH fastness grades remained the same with increase in pH.

The wet rubbing fastness increased initially with increase in pH upto pH 6. It decreased to 3 at pH 7 and again increased to ¾ at pH 8. Then grades of wet rubbing fastness increased with increase in pH. The dry

rubbing fastness showed an increase in fastness grades with increase in pH from 2/3 to 4/5.

The alkaline perspiration fastness remained the same at 4 for most of the pH values except at pH 4 and 6 where it was slightly higher. The acidic perspiration also remained the same at 4 for most of pH values except pH 4 and 8 where the grades are 4/5 and 3, respectively. No staining was noticed on adjacent wool and polyester fabrics.

#### 4.4.3.4 Effect of dye material concentration

Table 4.41 indicates the fastness grades of flame of forest dye on polyester for washing, light, rubbing and perspiration for different concentrations of dye material.

**Table 4.41 Effect of dye material concentration on fastness of flame of forest dye on polyester.**

Dye concentration (g/g)	WF			LF	RF			PF				
					W	D	Al		Ac			
	CC	CSC	CSP	CC	CSC	CSC	CC	CSP	CSW	CC	CSP	CSW
0.5	4	5	5	CC	3/4	4	4/5	5	5	4/5	5	5
1	3/4	5	5	2	3/4	3/4	4/5	5	5	4	5	5
2	3/4	5	5	2	3	3/4	4	5	5	4	5	5
3	4/5	5	5	1	3	3/4	4	5	5	4	5	5
4	4/5	5	5	1	3	3/4	4	5	5	4	5	5
5	4/5	5	5	1	3	3	4	5	5	4/5	5	5
6	4	5	5	1	3	3	4	5	5	4	5	5

The washing fastness showed an initial decrease upto 2g/g concentration. It increased for 3g/g concentration and remained constant

upto 5g/g and then decreased slightly at 6g/g. No staining was observed on adjacent cotton and polyester fabrics.

The hue of colour changed on exposure to light for 0.5g/g dye material concentration. It remained the same at 2 upto 2g/g and then decreased to 1 at 3g/g. Thereafter remained constant with increase in dye concentration.

The rubbing fastness showed a decrease with increase in dye material concentration. The wet rubbing fastness decreased from  $\frac{3}{4}$  at 1g/g dye material concentration to 3 at 2g/g and then remained constant with increase in dye material concentration. The dry rubbing fastness decreased slightly initially from 4 at 0.5g/g to  $\frac{3}{4}$  at 1g/g dye material concentration. It remained constant upto 4g/g and then again decreased slightly at 5g/g dye material concentration.

The alkaline and acidic perspiration fastness remained constant (4) after initial decrease with rise in dye material concentration. No staining was observed on adjacent wool and polyester fabrics.

#### **4.4.3.5 Effect of dyeing time**

The washing, light, rubbing and perspiration fastness grades of flame of forest dye dyed on polyester for different dyeing time periods have been given in table 4.42.

**Table 4.42 Effect of dyeing time on fastness of flame of forest dye on polyester.**

Dyeing time (minutes)	WF			LF	RF		PF					
	CC	CSC	CSP	CC	W	D	Al			Ac		
					CSC	CSC	CC	CSP	CSW	CC	CSP	CSW
15	4/5	5	5	1	¾	¾	3	5	5	2	5	5
30	4	5	5	1	¾	¾	¾	5	5	¾	5	5
45	4	5	5	1	¾	4	¾	5	5	¾	5	5
60	¾	5	5	2	3	¾	4	5	5	4	5	5
75	¾	5	5	2	4/5	4/5	3	5	5	3	5	5
90	¾	5	5	1	4	¾	3	5	5	¾	5	5
105	3	5	5	1	4	¾	3	5	5	4	5	5

The washing fastness showed a gradual decrease with increase in dyeing time from 4/5 to 3. No staining was noticed on adjacent cotton and polyester fabrics.

The light fastness grades remained the same at 1 for all dyeing time except 60 and 75 minutes dyeing time where it increased to 2.

The wet rubbing fastness remained the same upto 45 minutes of dyeing time at ¾. It decreased to 3 at 60 minutes and increased to 4/5 at 75 minutes. Again it decreased to 4 with increase in dyeing time. The dry rubbing fastness also showed a random pattern of increase and decrease in alternating manner with increase in dyeing time.

The perspiration fastness did not show a fixed trend in increase or decrease of grades with increase in dyeing time. Both alkaline and perspiration fastness initially showed an increase upto 60 minutes of dyeing time and then decreased at 75 minutes. Thereafter alkaline

perspiration fastness remained constant while the acidic perspiration fastness increased with increase in dyeing time. No staining was observed on adjacent wool and polyester fabrics of composite samples.

#### **4.4.3.6 Effect of mordanting method**

Table 4.35 depicts the washing, light, rubbing and perspiration fastness grades of flame of forest dye on polyester employing different mordants and the three mordanting methods with each mordant.

##### **4.4.3.6.1 Alum**

On washing the samples treated with different mordanting method, the colour of the dyed samples increased in all the cases. On exposure to light, the colour of dyed sample increased in the case of simultaneous mordanted sample and changed in the case of pre-mordanted sample. The wet rubbing fastness was the same for pre and post-mordanted samples and was the best in the case of simultaneous mordanted sample. The dry rubbing fastness was the best with post-mordanted sample, followed by that for simultaneous and pre-mordanted samples. The pre-mordanted sample had the best fastness. Alkaline perspiration fastness was the best for post-mordanted sample and the same for samples treated with other two mordanting methods. Acidic perspiration fastness was also the best for post-mordanted sample, followed by that for pre and simultaneous mordanting methods.

#### **4.4.3.6.2 Chrome**

It is clear from the table that on mordanting with chrome, the colour of the dyed sample changed on washing as well as on exposure to light in the case of pre-mordanting method and the fastness was maximum in the case of simultaneous mordanted sample and was minimum on application of mordant by post-mordanting method. Wet rubbing fastness was the best with post-mordanting method, followed by that for simultaneous and pre-mordanting methods. Dry rubbing fastness was maximum with post-mordanting method, followed by that for pre and simultaneous mordanting methods. The fastness grades of alkaline perspiration were the same for pre and simultaneous mordanted samples but was maximum for post-mordanted sample. Acidic perspiration fastness was maximum for pre-mordanted sample, followed by that for post and simultaneous mordanted samples (in that order).

#### **4.4.3.6.3 Cupric sulphate**

In the case of cupric sulphate treated samples, the colour of the simultaneous and post-mordanted samples changed on washing and exposure to light while in the case of pre-mordanted sample the colour increased on washing. Wet rubbing fastness was the same for pre and post-mordanted samples and was minimum for simultaneous mordanted sample. Dry rubbing fastness grades were the same for pre and simultaneous

mordanted samples and maximum in the case of post-mordanted sample. Alkaline perspiration fastness grades were the same for pre and simultaneous mordanted sample and maximum in the case of post-mordanted sample while acidic perspiration fastness grades were the same for pre and post-mordanted samples and minimum in the case of simultaneous mordanted sample.

#### **4.4.3.6.4 Ferrous sulphate**

On washing, the colour of pre and simultaneous mordanted samples increased and changed, respectively. On exposure to light, the colour of both pre and simultaneous mordanted samples changed. Dry rubbing fastness was minimum for simultaneous mordanted sample and the same for pre and post mordanting samples while the wet rubbing fastness grades were same in the case of simultaneous and post-mordanted samples and the best in the case of pre-mordanted sample. Alkaline perspiration fastness was maximum in the case of pre-mordanted sample, followed by that for post and simultaneous mordanted samples. The fastness grades of acidic perspiration were the same in the case of pre and simultaneous mordanting methods and minimum with post-mordanting method.

No staining was noticed on adjacent fabrics of composites samples in both washing and perspiration fastness in the case of all the mordants.

#### **4.4.3.7 Effects of mordant concentration**

The washing, light, rubbing and perspiration fastness grades of polyester samples dyed with flame of forest dye using five different mordant concentrations, have been presented in Table 4.36.

##### **4.4.3.7.1 Alum**

It can be seen from the table that on mordanting with alum, the colour of the dyed sample increased on washing upto 15 per cent mordant concentration and the colour changed on washing in the case of 20 and 25 per cent mordant concentrations. The colour of dyed samples changed on exposure to light with increase in the concentration of mordants. Wet and dry rubbing fastness grades increased at 10 per cent concentration, followed by decrease and increase at 15 and 20 per cent concentrations, respectively. Alkaline perspiration fastness grades increased with increase in concentration of mordant while acidic perspiration fastness grades increased at 10 per cent and decreased at 25 per cent concentration after remaining constant at 15 and 20 per cent.

##### **4.4.3.7.2 Chrome**

The colour of dyed samples changed on washing as well as on exposure to light at all the concentrations of mordant. Wet rubbing fastness grade increased at 2 per cent, decreased at 3 per cent remaining same on further increase in mordant concentration. Dry rubbing fastness increased

at 2 per cent concentration and then remained the same on further increase in the concentration of mordant. Alkaline perspiration fastness increased with increase in concentration of mordants. Acidic perspiration fastness increased at 2 per cent mordant concentration, decreased at 3 per cent concentration and then increased with increase in mordant concentration.

#### **4.4.3.7.3 Cupric sulphate**

On washing and exposure to light the colour of dyed samples changed at all the concentration of cupric sulphate. Wet rubbing fastness decreased at 2 per cent concentration and remained the same on further increase in mordant concentration. Dry rubbing fastness increased slightly at 1 and 3 per cent concentrations of mordant. Alkaline perspiration fastness decreased slightly at 2 and 4 per cent concentrations. Acidic perspiration decreased at 2 and 5 per cent mordant concentrations and colour of dyed sample increased at 4 per cent concentration of mordant on exposure to acidic perspiration solution.

#### **4.4.3.7.4 Ferrous sulphate**

In the case of application of ferrous sulphate as mordant, the colour of dyed samples increased on washing upto 3 per cent mordant and changed with further increase in mordant concentration. On exposure to light also, the colour of dyed samples changed when treated upto 4 per cent mordant and increased when concentration of mordant was increased. Wet rubbing

fastness grade decreased slightly at 5 per cent mordant concentration whereas dry rubbing fastness grades decreased slightly at 3 and 5 per cent concentration of mordant. Alkaline perspiration fastness grades decreased slightly at 2 and 3 per cent concentration whereas acidic perspiration fastness grades decreased at 2 per cent concentration and colour of dyed sample increased on treatment with acidic perspiration solution at 4 per cent concentration of mordant.

No staining was observed on adjacent fabrics of composite samples in the case of washing and perspiration fastness for all the mordants.

#### 4.4.3.8 Effect of the type of mordant

The washing, light rubbing and perspiration fastness grade for the polyester sample dyed with flame of forest dye using different mordants under optimum conditions have been compared with those of control (unmordanted) sample in the Table 4.43.

**Table 4.43 Effect of mordant type on fastness of flame of forest dye on polyester.**

Mordant	WF			LF	RF		PF					
	CC	CSC	CSP	CC	W	D	Al			Ac		
					CSC	CSC	CC	CSP	CSW	CC	CSP	CSW
Control	3/4	5	5	2	3	3/4	4	5	5	4	5	5
Alum	CC	5	5	CC	2/3	2/3	4/5	5	5	4/5	5	5
Chrome	CC	5	5	CC	2/3	2/3	4/5	5	5	4/5	5	5
Cupric sulphate	CC	5	5	CC	2/3	2/3	4/5	5	5	4/5	5	5
Ferrous sulphate	CC	5	5	CC	2/3	2/3	4/5	5	5	4/5	5	5

The change in colour was noticed instead of fading on washing and on exposure to light in the case of all the mordanted samples.

The wet and dry rubbing fastness decreased slightly in all the mordanted samples as compared to fastness of control sample.

The alkaline as well as acidic perspiration fastness grades showed an increase on application of different mordants as compared to fastness grades of control sample.

No staining was noticed on adjacent fabrics of composite samples used in washing and perspiration fastness.

It is clear from the mean scores of fastness grades as given in Table 4.36 that all the mordants affect the performance of dye in the similar way.

#### **4.4.4 Effect of mordants on CIE LAB values**

The effect of different mordants on colour strength, chroma and intensity of the polyester samples dyed with flame of forest dye under optimum conditions has been presented in Table 4.44 in terms of K/S and CIE LAB values.

The L\* values decreased for all mordanted samples except alum treated sample indicating lighter shade of alum treated sample and darker shades of other mordanted samples as compared to shade of control samples. All the mordanted samples were less red and less yellow except



a



b



c



d



e

Beefwood



a



b



c



d



e

Flame of forest

**SS 2 : Polyester samples dyed with beefwood and flame of forest dyes under optimum dyeing conditions using different mordants (a - unmordanted sample, b - sample treated with ferrous sulphate, c - sample treated with cupric sulphate, d - sample treated with chrome, e - sample treated with alum).**

alum treated sample which was more yellow as shown by a\* and b\* values. All the samples were duller than control sample as depicted by lower C\* values of mordanted samples.

**Table 4.44 Effect of mordants on CIE LAB values of flame of forest dye on polyester.**

Mordant	L*	a*	b*	C*	K/S	Colour obtained
Control	64.015	11.755	6.349	13.360	1.02	Light greyish red
Alum	64.171	11.267	6.486	13.001	0.99	Greyish brown red
Chrome	63.268	11.137	4.839	12.143	0.94	Dark reddish grey
Cupric sulphate	63.874	11.156	6.149	12.738	0.99	Rosy grey
Ferrous sulphate	63.297	9.640	5.746	11.223	1.20	Light greyish rosewood

The K/S values increased only in the case of ferrous sulphate treated sample indicating increase in colour strength. The dye uptake decreased in all other mordanted sample as compared to control sample as shown by lower K/S values. The colour obtained with flame of forest dye under optimized conditions ranged from light greyish red to greyish brown red.

A total of 67 shades of flame of forest dye were obtained in the process of optimization of different dyeing parameters.

#### **4.5 Dyeing with golden drop dye**

##### **4.5.1 Optimum dyeing conditions without mordanting**

Details of the ranks for the optimization of each variable, namely carrier concentration, carrier treatment time, dye extraction pH, dye

**Table 4.45 Optimization of carrier concentration, carrier treatment time, dye extraction pH, dye extraction time, dyeing pH, dye material concentration and dyeing time of golden drop dye on polyester on the basis of mean ranks given by a panel of judges.**

Variable	Rank	Optimum value
Carrier concentration (percent)		
5	5	
10	1	
15	2	10
20	3	
25	4	
Carrier treatment time (minutes)		
15	3	
30	5	
45	4	60
60	1	
75	2	
Dye extraction pH		
4	7	
5	6	
6	2	
7	1	7
8	5	
9	4	
10	3	
Dye extraction time (minutes)		
15	6	
30	5	
45	4	
60	2	90
75	3	
90	1	
105	7	
Dyeing pH		
4	5	
5	4	
6	1	
7	2	6
8	3	
9	6	
10	7	
Dye material concentration (g/g)		
0.5	7	
1	6	
2	5	
3	1	3
4	3	
5	2	
6	4	
Dyeing time (minutes)		
15	7	
30	6	
45	5	
60	4	90
75	3	
90	1	
105	2	

extraction time, dyeing pH, dye material concentration and dyeing time and their optimum values have been presented in Table 4.45.

#### **4.5.1.1 Optimum carrier concentration**

It can be seen from the Table 4.45 that the colour of dyed polyester improved with increase in the concentration of carrier, the optimum carrier concentration being 10 per cent beyond which the colour of dyed polyester samples decreased.

#### **4.5.1.2 Optimum carrier treatment time**

It can be seen from the table that the dyeing improved with the increase in the time of carrier treatment, the optimum time being 60 minutes, beyond which the colour decreased again.

#### **4.5.1.3 Optimum dye extraction pH**

It can be seen from the Table 4.45 that the extraction of dye from the root powder was best at neutral pH, i.e. pH 7. The extraction of dye decreased as the pH of extraction become more acidic or alkaline, i.e. either 4 or 10, respectively.

#### **4.5.1.4 Optimum dye extraction time**

The table indicates that the extraction of dye increased with the increase in duration of extraction and optimum extraction was at 90 minutes, beyond which the extraction of dye from root powder decreased

Vednere et al (1990) also reported 2 hours as optimum dye extraction time for chir bark.

#### **4.5.1.5 Optimum dyeing pH**

It is clear from the table that the best dyeing was obtained at pH 6, i.e. in slightly acidic conditions. Further increase in pH, i.e. in alkaline conditions, the dye absorption decreased. The acidic pH was reported as the best medium for dyeing ratanjot on nylon by Gulrajani et al (2001).

#### **4.5.1.6 Optimum dye material concentration**

The table shows that the optimum dye material concentration was 3 g per gram of polyester. The use of increased amount of dye resulted in reduced dye extraction.

#### **4.5.1.7 Optimum dyeing time**

The colour of polyester improved with increase in duration of dyeing and optimum dyeing time was adjudged at 90 minutes. The colour of sample dyed for 105 minute was less indicating decrease in dye uptake with increase in duration of dyeing.

#### **4.5.2 Optimum mordanting conditions**

The mordanting method and mordant concentration were optimized on the basis of overall fastness ratings of golden drop dye dyed polyester employing the carrier concentration, carrier treatment time, dye extraction

pH, dye extraction time, dyeing pH, dye material concentration and dyeing time optimized earlier without mordanting. The fastness grades for optimization of mordanting method and mordant concentration are given in Tables 4.46 and 4.47, respectively. The optimum mordanting and mordant concentration for the four mordants have been presented in Table 4.48.

**Table 4.46 Optimization of mordanting method for golden drop dye on polyester on the basis of fastness grades.**

Mordant	Mor- danting method	WF			LF		RF		PF			MS			Rank
		CC	CSC	CSP	CC	W	D	CC	Al	CSP	CSW	CC	CSP	CSW	
Alum	Pre	1	5	5	4	3	4	½	5	5	1	5	5	3.70	3
	Sim	1	5	5	3	3	¾	3	5	5	¾	5	5	3.91	2
	Post*	3	5	5	3	4/5	2/3	4	5	5	4/5	5	5	4.29	1
Chrome	Pre	2	5	5	3	¾	4	3	5	5	3	5	5	4.04	2.5
	Sim	¾	5	5	3	3	3	3	5	5	3	5	5	4.04	2.5
	Post*	4	5	5	3	3	2/3	4/5	5	5	4/5	5	5	4.29	1
Cupric sulphate	Pre	1	5	5	5	3	2/3	3	5	5	1	5	5	3.79	3
	Sim	1	5	5	4	¾	4	1	5	5	1	5	5	3.85	2
	Post*	2/3	5	5	2	3	3	¾	5	5	4/5	5	5	4.04	1
Ferrous sulphate	Pre	½	5	5	4	2	1	¾	5	5	4/5	5	5	3.875	3
	Sim*	4/5	5	5	3	3	½	3	5	5	¾	5	5	4.04	1
	Post	4	5	5	2	2	2/3	4	5	5	3	5	5	3.95	2

\*Optimum mordanting method

#### 4.5.2.1 Optimum mordanting method

The fastness ratings for different methods for mordanting have been presented in Table 4.46. The table reveals that the post-mordanting method of mordant application was the best (highest mean score) for alum, chrome and cupric sulphate except ferrous sulphate for which the simultaneous mordanting method gave the best fastness grades.

**Table 4.47 Optimization of mordant concentration for golden drop dye on polyester on the basis of fastness grades.**

Mordant	Mordant concentration	WF			LF	RF		PF			MS	Rank			
		CC	CSC	CSP	CC	W	D	Al		Ac					
						CSC	CSC	CC	CSP	CSW			CC	CSP	CSW
Alum	5	2/3	5	5	1	3/4	1/2	4	5	5	4	5	5	3.87	3
	10*	3	5	5	3	4/5	2/3	4	5	5	4/5	5	5	4.29	1
	15	2/3	5	5	1	3/4	1/2	4	5	5	4	5	5	3.87	3
	20	1/2	5	5	1	3/4	1/2	4	5	5	4	5	5	3.79	4
	25	1/2	5	5	1	3/4	1/2	4/5	5	5	4/5	5	5	3.87	3
Chrome	1	4	5	5	2	2/3	1/2	4/5	5	5	4	5	5	4.04	2
	2*	4	5	5	3	3	2/3	4/5	5	5	4/5	5	5	4.29	1
	3	4/5	5	5	1	3	1/2	4	5	5	4	5	5	4.00	4
	4	4/5	5	5	1	3	1/2	4	5	5	4	5	5	4.00	4
	5	4	5	5	1	3	1/2	4	5	5	4/5	5	5	4.00	4
Cupric sulphate	1	2/3	5	5	1	3/4	1/2	4/5	5	5	4	5	5	3.91	2
	2*	2/3	5	5	2	3	3	3/4	5	5	4/5	5	5	4.04	1
	3	2	5	5	1	3	1/2	4	5	5	4	5	5	3.79	5
	4	2	5	5	1	3	1/2	4	5	5	4/5	5	5	3.83	4
	5	2	5	5	1	3	1/2	4/5	5	5	4/5	5	5	3.87	3
Ferrous sulphate	1	2/3	5	5	3	3	1/2	4	5	5	4/5	5	5	4.04	3.5
	2	4/5	5	5	3	3	1/2	3	5	5	3/4	5	5	4.04	3.5
	3	3/4	5	5	1	2/3	1/2	4/5	5	5	4/5	5	5	3.95	5
	4*	3/4	5	5	3	2/3	1/2	4/5	5	5	4/5	5	5	4.12	1
	5	4	5	5	3	2/3	1/2	CI	5	5	CI	5	5	4.10	2

\* Optimum mordant concentration

#### 4.5.2.2 Optimum mordant concentration

The fastness ratings for different concentrations of mordants have been given in Table 4.47. The table makes it clear that the overall fastness grades increased with increase in mordant concentration upto certain level. Beyond that concentration the fastness ratings decreased in the case of all the mordants. The optimum concentration of alum, chrome, cupric sulphate and ferrous sulphate was 10, 2, 2 and 4 per cent, respectively (Table 4.48).

**Table 4.48 Optimum mordanting conditions for dyeing polyester with golden drop dye.**

Mordant	Mordanting method	Mordant concentration (percent)
Alum	Post	10
Chrome	Post	2
Cupric sulphate	Post	2
Ferrous sulphate	Sim	4

### 4.5.3. Effect of dyeing parameters on the fastness of golden drop dye

#### 4.5.3.1 Effect of carrier concentration

The washing fastness, light fastness, rubbing fastness and perspiration fastness grades of the golden drop dye on polyester at different carrier concentrations have been presented in table 4.49.

**Table 4.49 Effect of carrier concentration on fastness of golden drop dye on polyester.**

Carrier concentration (percent)	WF			LF		RF			PF			
								Al		Ac		
	CC	CSC	CSP	CC	CSC	CSC	CC	CSP	CSW	CC	CSP	CSW
5	½	5	5	1	4	3	4	5	5	4	5	5
10	2	5	5	1	¾	2/3	¾	5	5	¾	5	5
15	2	5	5	1	3	4/5	3	5	5	¾	5	5
20	2	5	5	1	4	3	4	5	5	4	5	5
25	½	5	5	1	4	4/5	4	5	5	4	5	5

The washing fastness showed an initial increase at 10 per cent carrier concentration, remained constant upto 20 per cent carrier concentration and then decreased at 25 per cent carrier concentration again. The washing fastness of dyed samples was in the range ½-2. No staining was observed on adjacent fabrics of composite samples.

The light fastness remained the same indicating no effect of carrier concentration.

The wet rubbing fastness initially decreased upto 15 per cent carrier concentration. It increased at 20 per cent carrier concentration and after that remained constant with increase in carrier concentration. The dry rubbing fastness did not show a fixed trend. It decreased and increased in alternating manner with increase in carrier concentration. The rubbing fastness of the dyed samples was fair to very good.

The alkaline perspiration fastness showed a decrease upto 15 per cent carrier concentration, increased at 20 per cent, thereafter remaining constant with increase in carrier concentration. The acidic perspiration fastness showed a decrease initially with increase in carrier concentration and the again increased at 20per cent carrier concentration. The perspiration fastness grades of dyed samples were in the range 3-4. No staining was observed on adjacent fabrics of composite samples.

#### **4.5.3.2 Effect of carrier treatment time**

Table 4.50 shows the washing light, rubbing and perspiration fastness of golden drop dye on polyester samples treated with carrier for five different time periods.

The wash fastness remained the same upto 45 minutes of carrier treatment time, decreased at 60 minutes and thereafter remained constant

with increase in carrier treatment time. The washing fastness grade of dyed samples were in the range 2-2/3. No staining was found on the adjacent cotton and polyester fabrics of composite samples.

**Table 4.50 Effect of carrier treatment time on fastness of golden drop dye on polyester.**

Carrier treatment time (minutes)	WF			LF	RF			PF				
	CC	CSC	CSP	CC	W	D	CC	Al		CC	Ac	
					CSC	CSC		CSP	CSW		CSP	CSW
15	2/3	5	5	1	2/3	2	3	5	5	3	5	5
30	2/3	5	5	1	1/2	2	3	5	5	2/3	5	5
45	2/3	5	5	1	2	2	2/3	5	5	3	5	5
60	2	5	5	1	3/4	2/3	3/4	5	5	3/4	5	5
75	2	5	5	2	2/3	2/3	2	5	5	3/4	5	5

The light fastness remained constant (1) upto 60 minutes for and then showed an increase at 75 minutes of carrier treatment time.

The wet rubbing fastness did not show a fixed pattern of grades with increase in carrier treatment time. It decreased from 2/3 at 15 minutes to 1/2 at 30 minutes. It again showed an increase with increase in carrier treatment time and finally decreased at 75 minutes. The dry rubbing fastness remained constant upto 45 minutes. It then increased slightly and thereafter remained constant with increase in carrier treatment time. The rubbing fastness of the dyed samples was poor to good.

The alkaline perspiration fastness increased with increase in carrier treatment time except at 45 and 75 minutes where fastness grades decreased. The acidic perspiration showed an initial decrease and then

increased with increase in carrier concentration time. The perspiration fastness grades of dyed samples were in the range 2-3/4. No staining was noticed on adjacent wool and polyester fabrics of composite samples.

#### 4.5.3.3 Effect of dyeing pH

The fastness grades of washing, light, rubbing and perspiration of golden drop dye on polyester at different pH values are given in Table 4.51

**Table 4.51 Effect of dyeing pH on fastness of golden drop dye on polyester.**

Dyeing pH	WF			LF	RF		PF					
	CC	CSC	CSP	CC	W	D	Al			Ac		
					CSC	CSC	CC	CSP	CSW	CC	CSP	CSW
4	1	5	5	2	3	½	4	5	5	¾	5	5
5	1	5	5	2	4	2/3	4/5	5	5	4/5	5	5
6	2	5	5	2	4	2	3	5	5	3	5	5
7	2	5	5	1	¾	2/3	¾	5	5	¾	5	5
8	½	5	5	3	2/3	2	¾	5	5	4	5	5
9	½	5	5	2	2	½	4	5	5	4	5	5
10	2	5	5	2	2/3	3	4	5	5	4/5	5	5

The washing fastness showed an increase upto pH 7, followed by decrease at pH 8 and finally increased at pH 10. The washing fastness grades of dyed samples were in the range 1-2. No staining was found on adjacent cotton and polyester fabrics.

The light fastness remained constant upto pH 6. It decreased at pH 7 and increased at pH 8. Again at pH 9 it showed a decrease and constant thereafter. The light fastness grades were in the range 1-3.

The rubbing fastness did not show any fixed trend with increase in pH. The wet rubbing fastness increased initially upto pH 6, then decreased upto pH 9 and increased again at pH 10. The dry rubbing fastness increased and decreased in alternating manner upto pH 9 and finally increased at pH 10. The rubbing fastness grades were in the range  $\frac{1}{2}$ -4.

The perspiration fastness also did not show any fixed trend with increase in pH. The alkaline and acidic perspiration fastness firstly increased and then decreased at pH 6. It showed a gradual increase with further increase in dyeing pH. The perspiration fastness of the dyed samples was good to very good. No staining was noticed on adjacent wool and polyester fabrics.

#### **4.5.3.4 Effect of dye material concentration**

Table 4.52 indicates the fastness grades of golden drop dye on polyester for washing light, rubbing and perspiration for different concentrations of dye material.

The washing fastness remained constant with increase in dye concentration upto 2g/g dye concentration. It decreased slightly and again remained constant upto 5g/g dye concentration. Then it increased slightly at 6g/g dye material concentration. The washing fastness grades were in the range  $\frac{1}{2}$ -2. No staining was observed on adjacent cotton and polyester fabrics.

**Table 4.52 Effect of dye material concentration on fastness of golden drop dye on polyester.**

Dye concentration (g/g)	WF			LF	RF			PF				
	CC	CSC	CSP	CC	W	D	CC	Al		Ac		
					CSC	CSC		CSP	CSW	CC	CSP	CSW
0.5	2	5	5	2	2	½	3	5	5	4	5	5
1	2	5	5	1	2/3	2/3	4/5	5	5	2/3	5	5
2	2	5	5	1	¾	2/3	¾	5	5	¾	5	5
3	1	5	5	1	2	2	3	5	5	¾	5	5
4	1	5	5	1	2/3	½	4	5	5	¾	5	5
5	1	5	5	1	2	½	¾	5	5	4	5	5
6	½	5	5	1	2	2	3	5	5	4/5	5	5

The light fastness decreased initially at 1g/g dye concentration and thereafter remained constant (1) with increase in dye material concentration.

The wet rubbing fastness showed an initial increase upto 2g/g dye material concentration. At 3 g/g dye material concentration it decreased and remained constant on further increase in dye material concentration except 4g/g where it increased slightly. The dry rubbing fastness increased initially and showed decrease upto 5g/g dye material concentration and then at 6g/g dye material concentration it increased slightly.

The alkaline perspiration fastness showed an initial increase at 1g/g dye material concentration. It then decreased at 2g/g dye material concentration and again increased at 4g/g dye material concentration after which, it showed a gradual decrease. The acidic perspiration fastness decreased at 1g/g dye concentration and increased at 2g/g dye material

concentration. It then remained constant upto 4g/g dye material concentration and after that showed a gradual increase. The perspiration fastness grades were in the range 2/3-4/5. No staining was observed on adjacent wool and polyester fabrics.

#### 4.5.3.5 Effect of dyeing time

The washing, light, rubbing and perspiration fastness grades of golden drop dye dyed on polyester for different time period have been presented in Table 4.53.

**Table 4.53 Effect of dyeing time on fastness of golden drop dye on polyester.**

Dyeing time (minutes)	WF			LF	RF			PF				
	CC	CSC	CSP	CC	W	D	CC	Al		Ac		
					CSC	CSC		CSP	CSW	CC	CSP	CSW
15	1	5	5	1	2	2	3	5	5	3	5	5
30	1	5	5	1	2/3	2	3	5	5	2	5	5
45	1	5	5	1	2	2/3	3	5	5	3	5	5
60	2	5	5	1	¾	2/3	¾	5	5	¾	5	5
75	2	5	5	1	2	2	4	5	5	3	5	5
90	½	5	5	2	½	2/3	2	5	5	2	5	5
105	½	5	5	2	½	½	4	5	5	3	5	5

The washing fastness increased from 1-2 with increase in dyeing time upto 75 minutes of dyeing time. After that it decreased slightly with increase in dyeing time. No staining was noticed on adjacent cotton and polyester fabrics.

The light fastness remained the same (1) upto 75 minutes. It then increased to 2 at 90 minutes of dyeing time and thereafter remained constant.

The rubbing fastness did not show any fixed trend with the increase in dyeing time. The wet rubbing fastness increased and decreased in alternating manner with increase in dyeing time. Then it decreased at 90 minutes dyeing time. The dry rubbing fastness increased upto 60 minutes of dyeing time. It then decreased and increased in alternating manner after 75 minutes at 75 minutes. The rubbing fastness grades were in the range  $\frac{1}{2}$ - $\frac{3}{4}$  of dyeing time.

The alkaline perspiration fastness remained constant upto 45 minutes and thereafter increased with increase in dyeing time except at 90 minutes where it decreased slightly. The acidic perspiration fastness initially decreased initially upto 30 minutes and increased upto 60 minutes dyeing time. It then decreased upto 90 minutes and again increased at 105 minutes. No staining was observed on adjacent wool and polyester fabrics.

#### **4.5.3.6 Effect of mordanting method**

Table 4.46 depicts the washing, light, rubbing and perspiration fastness grades of golden drop dye on polyester employing different mordants and the three mordanting methods with each mordant.

#### **4.5.3.6.1 Alum**

It is clear from the table that on mordanting with alum, the fastness grades of washing and wet rubbing for pre and simultaneous mordanted samples were the same and were the best with post-mordanting method. Light fastness grade was maximum for pre-mordanted sample and was the same for post and simultaneous mordanted samples. Dry rubbing fastness grade was maximum for pre-mordanted sample, followed by that for simultaneous and post-mordanted samples. Both alkaline and acidic perspiration fastness grades were maximum for post-mordanted sample, followed by that for simultaneous and pre-mordanting methods (in that order).

#### **4.5.3.6.2 Chrome**

The washing fastness was the best for post-mordanted sample, followed by that for simultaneous and pre-mordanted samples. No effect of mordanting method was seen on the light fastness grades since they were the same for all mordanting methods. Wet rubbing fastness was the best for pre-mordanted sample and was the same with post and simultaneous mordanting methods. Dry rubbing fastness was also maximum for pre-mordanted sample, followed by that for simultaneous and post-mordanted samples. Both acidic and alkaline perspiration fastness grades were the

same for pre and simultaneous mordanted samples and the best with post-mordanting method.

#### **4.5.3.6.3 Cupric sulphate**

Washing and acidic perspiration fastness grades were the best with post-mordanted method and the same with pre and simultaneous mordanted method. Light fastness grade was maximum for pre-mordanted sample, followed by that for simultaneous and post-mordanted samples. Wet rubbing fastness grades were the same for pre and post-mordanted sample and maximum in the case of simultaneous mordanted sample. Dry rubbing fastness was maximum for simultaneous mordanted sample, followed by that for post and pre-mordanted samples. Alkaline perspiration fastness grade was maximum for post-mordanted sample, followed by that for pre and simultaneous mordanted samples. Acidic perspiration fastness grades were the same for pre and simultaneous mordanted samples and the best in the case of post-mordanted sample.

#### **4.5.3.6.4 Ferrous sulphate**

In the case of ferrous sulphate application as mordant, the fastness grade of washing was the best with simultaneous mordanting method, followed by that for post and pre-mordanting methods. Light fastness was maximum for pre-mordanting method, followed by that for simultaneous and post-mordanting methods. Wet rubbing fastness of pre and post-

mordanted samples was the same and was the best with simultaneous mordanting method. Dry rubbing fastness was the best with post-mordanting method, followed by that for simultaneous and pre-mordanting methods. Alkaline perspiration fastness grade was maximum in the case of post-mordanted sample and decreased with pre and simultaneous mordanting method, respectively. Acidic perspiration fastness was maximum of pre-mordanted sample and decreased with simultaneous and post-mordanting method.

No staining was observed on adjacent fabrics of composite samples of washing and perspiration fastness in the case of all the mordants.

#### **4.5.3.7. Effect of mordant concentration**

The washing, light, rubbing and perspiration fastness grades of polyester sample dyed with golden drop dye using five different mordant concentrations, have been presented in Table 4.47.

##### **4.5.3.7.1 Alum**

The washing fastness grades for change in colour increased at 10 per cent, decreased at 15 per cent and remained the same on further increase in concentration of mordant. Light and wet and dry rubbing fastness grades showed an increase at 10 per cent concentration and then became constant on further increase in the concentration of mordant. Alkaline perspiration fastness grades increased at 25 per cent concentration after remaining

constant upto 20 per cent mordant concentration whereas acidic perspiration fastness increased slightly at 10 and 25 per cent concentrations.

Staining on adjacent fabrics of composite samples was not observed in the case of both washing and perspiration fastness.

#### **4.5.3.7.2 Chrome**

It is clear from the table that on mordanting with chrome, the fastness grades for washing remained the same except at 3 and 4 per cent concentration where slight increase was observed. Light fastness grades increased at 2 per cent concentration and decreased on further increase in the mordant concentration. Wet rubbing fastness grades increased and remained constant after 2 per cent concentration whereas dry rubbing fastness increased slightly at 2 per cent concentration and remained the same at rest of mordant concentrations. Alkaline perspiration fastness decreased at 3 per cent and remained constant on further increase in the concentration of mordant whereas acidic perspiration fastness grades increased slightly at 2 and 5 per cent mordant concentrations. No staining was noticed on adjacent fabrics of composite samples of both washing and perspiration fastness.

#### **4.5.3.7.3 Cupric sulphate**

The fastness grades of washing and wet rubbing remained constant on increase in mordant concentration after decreasing slightly at 3 and 2 per cent concentrations. Light and dry rubbing fastness remained constant at different mordant concentrations except at 2 per cent where fastness grades were the best. The alkaline perspiration fastness decreased at 2 per cent and then increased with increase in mordant concentration. The acidic perspiration fastness grades decreased slightly at 1 and 3 per cent concentrations remaining the same at rest of mordant concentrations. No staining was observed on adjacent fabrics of composite samples of washing and perspiration fastness.

#### **4.5.3.7.4 Ferrous sulphate**

In the case of ferrous sulphate, the washing fastness grades for change in colour increased at 2 per cent concentration, decreased at 3 per cent concentration and again increased at 5 per cent concentration. Light fastness grades remained the same except at 3 per cent concentration where marked decrease was observed. Wet rubbing fastness grades decreased at 3 per cent concentration and remained the same on further increase in mordant concentration. Dry rubbing fastness grades remained constant at all the concentrations of mordant. Both alkaline and acidic perspiration fastness grades decreased at 2 per cent increased at 3 per cent concentration.

and colour of dyed samples increased at 5 per cent concentration. No staining was noticed on adjacent fabrics in composite samples of washing and perspiration fastness.

#### 4.5.3.8 Effect of the type of mordant

The washing, light rubbing and perspiration fastness grade for the polyester sample dyed with golden drop dye using different mordants under optimum conditions have been compared with those of control (unmordanted) sample in the Table 4.54.

**Table 4.54 Effect of mordant type on fastness of golden drop dye on polyester.**

Mordant	WF			LF	RF		PF					
	CC	CSC	CSP	CC	W	D	Al			Ac		
					CSC	CSC	CC	CSP	CSW	CC	CSP	CSW
Control	½	5	5	2	½	2/3	2	5	5	2	5	5
Alum	3	5	5	3	4/5	2/3	4	5	5	4/5	5	5
Chrome	4	5	5	3	3	2/3	4/5	5	5	4/5	5	5
Cupric sulphate	2/3	5	5	2	3	3	¾	5	5	4/5	5	5
Ferrous sulphate	¾	5	5	3	2/3	½	4/5	5	5	4/5	5	5

A marked increase was seen in the washing, wet rubbing and alkaline and acidic perspiration fastness grades on application of all the mordants when compared with fastness grades of control sample.

The light fastness grade remained the same for cupric sulphate treated sample and increased by a unit in the case of rest of mordants when compared with fastness grade of control sample.

The dry rubbing fastness grades remained the same for alum and chrome treated samples, increased for cupric sulphate treated sample and decreased for ferrous sulphate treated sample when compared with control sample.

No staining was noticed on adjacent fabrics of composite samples used in washing and perspiration fastness.

It can be seen from the mean scores of fastness grades as given in Table 4.47 that alum and chrome were best mordants, followed by cupric sulphate and ferrous sulphate.

#### 4.5.4 Effect of mordants on CIE LAB values

The effect of different mordants on colour strength, chroma and intensity of the polyester samples dyed with golden drop dye under optimum conditions has been presented in Table 4.55 in terms of K/S and CIE LAB values.

**Table 4.55 Effect of mordants on CEI LAB values of golden drop dye on polyester.**

Mordant	L*	a*	b*	C*	K/S	Colour obtained
Control	59.699	2.634	3.712	4.552	1.44	Ruby grey
Alum	58.808	2.494	4.410	2.865	1.05	Dark brownish grey red
Chrome	59.173	2.493	2.521	3.545	1.25	Brownish grey red
Cupric sulphate	60.048	2.300	4.410	4.974	1.37	Magenta grey
Ferrous sulphate	60.079	2.781	6.149	6.749	2.51	Purplish grey

The L\* values increased in the case of mordanted samples except alum treated sample as compared to value of control sample indicating that

the mordanted samples were of darker shade. All the mordanted samples were less red except ferrous sulphate treated sample than the control sample as shown by the values of  $a^*$ . The yellowness increased in the case of alum, cupric sulphate and ferrous sulphate treated samples as shown by higher  $b^*$  values as compared to the values of control sample. But only cupric and ferrous sulphate treated samples were brighter than control sample as depicted by their  $C^*$  values which were higher than the value of control sample.

The K/S values increased only in the case of ferrous sulphate treated sample indicating increase in strength of colour of sample mordanted with ferrous sulphate as compared to control sample. The colour strength of other mordanted samples was lower than control sample.

The colour obtained with golden drop dye under optimized conditions ranged from purplish grey to dark brownish grey red.

A total of 67 shades of golden drop dye were obtained in the process of optimization of different dyeing parameters.

## **4.6 Dyeing with Indian blackberry dye**

### **4.6.1 Optimum dyeing conditions without mordanting**

Details of the ranks for the optimization of each variable, namely carrier concentration, carrier treatment time, dye extraction pH, dye

**Table 4.56 Optimization of carrier concentration, carrier treatment time, dye extraction pH, dye extraction time, dyeing pH, dye material concentration and dyeing time of Indian blackberry dye on polyester on the basis of mean ranks given by a panel of judges.**

Variable	Rank	Optimum value
Carrier concentration (percent)		
5	2	
10	1	
15	3	10
20	4	
25	5	
Carrier treatment time (minutes)		
15	4	
30	3	
45	2	60
60	1	
75	5	
Dye extraction pH		
4	7	
5	6	
6	5	
7	4	8
8	1	
9	2.5	
10	2.5	
Dye extraction time (minutes)		
15	6	
30	5	
45	1	
60	2	45
75	3	
90	4	
105	7	
Dyeing pH		
4	5	
5	3	
6	2	
7	1	7
8	4	
9	6	
10	7	
Dye material concentration (g/g)		
0.5	3	
1	2	
2	1	
3	4	2
4	5	
5	6	
6	7	
Dyeing time (minutes)		
15	7	
30	6	
45	5	
60	4	105
75	2.5	
90	2.5	
105	1	

extraction time, dyeing pH, dye material concentration and dyeing time and their optimum values have been presented in Table 4.56.

#### **4.6.1.1 Optimum carrier concentration**

It can be seen from the Table 4.56 that the colour of the dyed polyester improved with the increase in the concentration of carrier, the optimum carrier concentration being 20 per cent, beyond which it decreased.

#### **4.6.1.2 Optimum carrier treatment time**

It can be seen from the table that the dyeing improved with the increase in the time of carrier treatment, the optimum time being 60 minutes, beyond which the colour decreased again.

#### **4.6.1.3 Optimum dye extraction pH**

It can be seen from the Table 4.56 that the extraction of dye from the bark powder increased with increase in the pH and optimum pH for dye extraction was 8, i.e. in slightly alkaline conditions. The extraction of dye was minimum at pH 4, i.e. in acidic conditions.

#### **4.6.1.4 Optimum dye extraction time**

The table indicates that a duration of 45 minutes was adjudged the best for maximum extraction of the dye. Extraction for longer period was not found helpful in increasing the dye extraction. This may be because

after a particular time some substances other than dye also start to leach out from the plant material which may hinder the dye absorption. Similar observations were made by Singh et al (1993a) in the case of madder dye.

#### **4.6.1.5 Optimum dyeing pH**

It is clear from the table that the best dyeing was obtained at neutral pH, i.e. optimum pH was 7 and the dye uptake was reduced with increase in pH of dyebath towards acidity (pH 4) and alkalinity (pH 10) conditions.

#### **4.6.1.6 Optimum dye material concentration**

The table shows that the optimum dye concentration was 2g per gram of polyester. The use of increased amount of dye resulted in reduced dye extraction and hence, reduced dye uptake.

#### **4.6.1.7 Optimum dyeing time**

The colour of polyester improved with the increase in duration of dyeing and the optimum dyeing time for Indian blackberry dye was 105 minutes. Similar results were reported by Agarwal et al (1992) for henna dye on merino wool.

#### **4.6.2 Optimum mordanting conditions**

The mordanting method and mordant concentration were optimized on the basis of overall fastness ratings of Indian blackberry dye dyed polyester employing the carrier concentration, carrier treatment time, dye

extraction pH, dye extraction time, dyeing pH, dye material concentration and dyeing time optimized earlier without mordanting. The fastness grades for optimization of mordanting method and mordant concentration are given in Tables 4.57 and 4.58, respectively. The optimum mordanting and mordant concentration for the four mordants have been presented in Table 4.59.

**Table 4.57 Optimization of mordanting method for Indian blackberry dye on polyester on the basis of fastness grades.**

Mordant	Mor- danting method	WF			LF	RF			PF			MS	Rank		
		CC	CSC	CSP	CC	W		D		Al		Ac			
						CSC	CSC	CC	CSP	CSW	CC	CSP		CSW	
Alum	Pre	1	5	5	2	3	2/3	1	5	5	2	5	5	3.45	3
	Sim	5	5	5	2	2	2	4	5	5	4/5	5	5	4.12	2
	Post*	3/4	5	5	3	3/4	3	4/5	5	5	4/5	5	5	4.33	1
Chrome	Pre	2	5	5	3	3	3/4	2	5	5	3/4	5	5	3.91	2
	Sim*	3/4	5	5	1	2/3	2/3	4/5	5	5	4/5	5	5	4.04	1
	Post	3	5	5	3	3	2/3	2	5	5	3	5	5	3.87	3
Cupric sulphate	Pre	3	5	5	4	2	2/3	4	5	5	3	5	5	4.04	2
	Sim	3	4/5	5	3	3	3	2	5	5	1	5	5	3.70	3
	Post*	4	5	5	2	3	2/3	4	5	5	4	5	5	4.12	1
Ferrous sulphate	Pre	1	5	5	3	1/2	1/2	3/4	5	5	4	5	5	3.70	3
	Sim	2	5	5	3	2/3	2/3	3	5	5	3	5	5	3.83	2
	Post*	3	5	5	2	3	2/3	4/5	5	5	4/5	5	5	4.12	1

\*Optimum mordanting method

#### 4.6.2.1 Optimum mordanting method

The fastness rating for different methods of mordanting have been presented in Table 4.57. The table reveals that the post-mordanting method of mordant application was the best (highest mean score) for alum, cupric sulphate and ferrous sulphate whereas in the case of chrome, simultaneous mordanting method gave the best fastness results grades. Dayal and Dobhal

(2000) also reported post-mordanting as the best method for conferring good fastness results to poplar bath dye on wool, silk and cotton.

**Table 4.58 Effect of mordant concentration on fastness of Indian blackberry dye on polyester.**

Mordant	Mordant concentration	WF			LF	RF		PF					MS	Rank	
		CC	CSC	CSP	CC	W		Al		Ac					
						CSC	CSC	CC	CSP	CSW	CC	CSP			CSW
Alum	5*	4	5	5	4	4	¾	4/5	5	5	4/5	5	5	4.54	1
	10`	¾	5	5	3	¾	3	4/5	5	5	4/5	5	5	4.33	2
	15	¾	5	5	2	¾	2/3	4/5	5	5	4/5	5	5	4.20	3
	20	¾	5	5	2	¾	2	4/5	5	5	4/5	5	5	4.16	4
	25	3	5	5	2	¾	2	4/5	5	5	4/5	5	5	4.12	5
Chrome	1	¾	5	5	2	2/3	2/3	4/5	5	5	4/5	5	5	4.12	4
	2	¾	5	5	1	2/3	2/3	4/5	5	5	4/5	5	5	4.04	5
	3	4	5	5	2	3	2/3	4/5	5	5	4/5	5	5	4.20	3
	4	4	5	5	2	¾	2/3	4/5	5	5	4/5	5	5	4.25	2
	5*	4/5	5	5	4	3	2	4/5	5	5	4/5	5	5	4.37	1
Cupric sulphate	1	4	5	5	2	3	2/3	4/5	5	5	4/5	5	5	4.20	2
	2	4	5	5	2	3	2/3	4	5	5	4	5	5	4.12	4
	3*	¾	5	5	CI	2/3	2/3	4	5	5	4	5	5	4.22	1
	4	4	5	5	2	2/3	2/3	4/5	5	5	4/5	5	5	4.16	3
	5	4/5	5	5	2	3	2/3	¾	5	5	¾	5	5	4.08	5
Ferrous sulphate	1*	4/5	5	5	CI	3	2/3	4/5	5	5	4/5	5	5	4.45	1
	2	3	5	5	2	3	2/3	4/5	5	5	4/5	5	5	4.12	3
	3	3	5	5	2	2	½	4	5	5	4/5	5	5	3.91	4
	4	3	5	5	1	2	½	4/5	5	5	4/5	5	5	3.87	5
	5	3	5	5	CC	2	½	4/5	5	5	4/5	5	5	4.13	2

\*Optimum mordant concentration

#### 4.6.2.2 Optimum mordant concentration

The fastness grades for different concentrations of mordants have been given in Table 4.58. The table makes it clear that the overall fastness grades decreased with increase in concentration of mordants in the case of alum and ferrous sulphate while the fastness grades decreased upto certain level and then increased with increase in concentration of chrome. In the

case of cupric sulphate the overall fastness grades increased with increase in mordant concentration upto certain level. Beyond that concentration the fastness rating decreased. The optimum concentration of alum, chrome, cupric sulphate and ferrous sulphate was 5, 5, 3 and one per cent, respectively (Table 4.59).

**Table 4.59 Optimum mordanting conditions for dyeing polyester with Indian blackberry dye.**

Mordant	Mordanting method	Mordant concentration (percent)
Alum	Post	5
Chrome	Sim	5
Cupric sulphate	Post	3
Ferrous sulphate	Post	1

### 4.6.3. Effect of dyeing parameter on the fastness of Indian blackberry dye

#### 4.6.3.1 Effect of carrier concentration

The washing fastness, light fastness, rubbing fastness and perspiration fastness grades of Indian Blackberry dye on polyester at different carrier concentration have been presented in Table 4.60.

The washing fastness decreased initially at 5 per cent and then remained the same (2) with increase in carrier concentration. No staining was observed on adjacent fabrics of composite samples.

The light fastness remained the same (3) with increase in carrier concentration indicating no effect of carrier concentration.

**Table 4.60 Effect of carrier concentration on fastness of Indian blackberry dye on polyester.**

Carrier concentration (percent)	WF			LF	RF		PF					
	CC	CSC	CSP	CC	W	D	Al			Ac		
					CSC	CSC	CC	CSP	CSW	CC	CSP	CSW
5	2/3	5	5	3	3	3	4/5	5	5	4/5	5	5
10	2	5	5	3	2/3	2/3	4/5	5	5	5	5	5
15	2	5	5	3	2/3	2/3	4/5	5	5	4/5	5	5
20	2	5	5	3	3/4	2/3	4	5	5	4/5	5	5
25	2	5	5	3	2/3	2/3	4	5	5	4	5	5

The wet rubbing fastness decreased upto 15 per cent carrier concentration. At 20 per cent it showed an increase ( $\frac{3}{4}$ ) and then settled back to  $\frac{2}{3}$  at 25 per cent carrier concentration. The dry rubbing fastness showed an initial decrease and thereafter remained the same ( $\frac{2}{3}$ ) with increase in carrier concentration.

The alkaline perspiration fastness remained the same ( $\frac{4}{5}$ ) upto 15 per cent carrier concentration, then decreased slightly and again remained the same (4). The acidic perspiration showed an increase at 10 per cent carrier concentration. Then it showed a decrease with increase in carrier concentration. No staining was observed on adjacent fabrics of composite samples in both washing and perspiration fastness.

#### 4.6.3.2 Effect of carrier treatment time

Table 4.61 shows the washing, light, rubbing and perspiration fastness of Indian blackberry dye on polyester samples treated with carrier for five different time periods.

**Table 4.61 Effect of carrier treatment time on fastness of Indian blackberry dye on polyester.**

Carrier treatment time (minutes)	WF			LF	RF			PF				
	CC	CSC	CSP	CC	W	D	CC	Al		CSW	Ac	
					CSC	CSC		CSP	CC		CSP	CSW
15	4/5	5	5	3	3	4	4	5	5	4	5	5
30	4/5	5	5	3	2/3	3	3	5	5	3/4	5	5
45	3/4	5	5	3	2/3	3	3/4	5	5	3/4	5	5
60	2	5	5	3	2/3	2/3	4	5	5	4/5	5	5
75	2	5	5	4	3	4/5	4	5	5	4	5	5

The washing fastness showed a gradual decrease with increase in carrier treatment time from 4/5 to 2. No staining was found on the adjacent polyester and cotton fabric.

The light fastness remained constant (3) upto 60 minutes and then increased at 75 minutes.

The wet rubbing fastness showed an initial decrease and then remained constant (2/3) upto 60 minutes of carrier treatment time. It then increased at 75 minutes.

The dry rubbing fastness showed a gradual decrease with increase in carrier treatment time. It then increased to 4/5 at 75 minutes.

The perspiration fastness did not show any fixed pattern with increase in carrier treatment time. The alkaline perspiration fastness decreased initially and increased gradually upto 60 minutes of carrier treatment time. The acidic perspiration fastness also decreased initially and then increased with increase in carrier treatment time but it again showed a

decrease at 75 minutes. No staining was noticed on adjacent wool and polyester fabrics of composite samples.

#### 4.6.3.3 Effect of dyeing pH

The fastness grades of washing, light, rubbing and perspiration of Indian blackberry dye on polyester at different pH values are given in Table 4.62.

**Table 4.62 Effect of dyeing pH on fastness of Indian blackberry dye on polyester.**

Dyeing pH	WF			LF	RF			PF				
	CC	CSC	CSP	CC	W	D	CC	Al		Ac		
					CSC	CSC		CSP	CSW	CC	CSP	CSW
4	4	5	5	3	3	4	3	5	5	4	5	5
5	3	5	5	3	3	4	4	5	5	4	5	5
6	2	5	5	3	3	¾	4	5	5	4	5	5
7	2	5	5	3	2/3	2/3	4	5	5	4/5	5	5
8	1	5	5	3	2/3	4	3	5	5	3	5	5
9	1	5	5	2	3	4/5	1	5	5	1	5	5
10	1	5	5	2	2/3	4	1	5	5	1	5	5

The washing fastness showed a gradual decrease from 4-1 with increase in pH upto 8 and then remained the same at pH 9. No staining was found on adjacent cotton and polyester fabrics of composite samples.

The light fastness remained constant upto pH 8, then decreased slightly and remained constant. The light fastness grade of dyed samples were in the range 2-3.

The wet rubbing fastness remained constant upto pH 6. It decreased slightly at pH 7, increased at pH 9 and again decreased at pH 10. The dry

rubbing fastness showed a decrease upto pH 7 and then increased upto pH 9. At dyeing pH 10 it again showed a decrease. The rubbing fastness of the dyed samples was fair to very good.

The alkaline perspiration fastness increased and then remained constant upto pH 7. Further it decreased with increase in dyeing pH and finally became constant at pH 9 and 10. The acidic perspiration fastness remained constant upto pH 6 and showed a slight increase at pH 7. It then decreased with further increase in dyeing pH and finally became constant. The perspiration fastness of the dyed sample was poor to very good. No staining was noted on adjacent wool and polyester fabrics.

#### 4.6.3.4 Effect of dye material concentration

Table 4.63 indicates the fastness grades of Indian blackberry dye on polyester for washing light, rubbing and perspiration for different concentrations of dye material.

**Table 4.63 Effect of dye material concentration on fastness of Indian blackberry dye on polyester.**

Dye concentration (g/g)	WF			LF	RF		PF					
	CC	CSC	CSP	CC	W	D	Al			Ac		
					CSC	CSC	CC	CSP	CSW	CC	CSP	CSW
0.5	1	5	5	3	3/4	4/5	3	5	5	4/5	5	5
1	1	5	5	3	3/4	4/5	4	5	5	4/5	5	5
2	2	5	5	3	2/3	2/3	4	5	5	4/5	5	5
3	2	5	5	3	3/4	3/4	2	5	5	3/4	5	5
4	1	5	5	2	4	4	2	5	5	2	5	5
5	1	5	5	2	4	4	3	5	5	3	5	5
6	1	5	5	2	4	4	3/4	5	5	4	5	5

The washing fastness remained constant at 1 for all dye material concentration except at 2g/g and 3g/g dye material concentration where it was 2. No staining was observed on adjacent cotton and polyester fabrics.

The light fastness remained constant (3) with increase in dye material concentration upto 3g/g. Then it decreased slightly and again remained constant (2) on further increase in dye material concentration.

The wet rubbing fastness remained constant with increase in dye material concentration upto 3g/g except 2g/g where it decreased and then increased slightly. Thereafter it remained constant on further increase in dye material concentration. The dry rubbing fastness showed a decrease at 2g/g dye material concentration and then showed an increase with increase in dye material concentration and finally became constant. The rubbing fastness grades were in the range 2/3-4/5.

The alkaline perspiration fastness showed an initial increase with increase in dye material concentration. It decreased for 3g/g dye material concentration. Further increase in dye material concentration showed gradual increase in alkaline perspiration fastness. The acidic perspiration fastness remained constant with increase in dye material concentration upto 2g/g. It then showed a decrease for 4g/g and further increase in dye material concentration showed gradual increase in acidic perspiration

fastness. The perspiration fastness grades were in the range 2-4/5. No staining was observed on adjacent cotton and polyester fabrics.

#### 4.6.3.5 Effect of dyeing time

The washing, light, rubbing and perspiration fastness grades of Indian blackberry dye dyed on polyester for different time periods have been presented in Table 4.64.

**Table 4.64 Effect of dyeing time on fastness of Indian blackberry dye on polyester.**

Dye time (minutes)	WF			LF	RF			PF				
	CC	CSC	CSP	CC	W	D	CC	Al		Ac		
					CSC	CSC		CSP	CSW	CC	CSP	CSW
15	1	5	5	3	3	3	3	5	5	2/3	5	5
30	2	5	5	3	3	¾	3	5	5	2/3	5	5
45	2	5	5	3	3	¾	3	5	5	2/3	5	5
60	2	5	5	3	2/3	2/3	4	5	5	4/5	5	5
75	2	5	5	3	2/3	¾	4	5	5	¾	5	5
90	1	5	5	4	2/3	3	4	5	5	¾	5	5
105	1	5	5	3	3	4	4	5	5	¾	5	5

The washing fastness increased initially and remained constant with increase in dyeing time upto 75 minutes. With further increase in dyeing time it decreased and then again became constant. No staining was noticed on adjacent cotton and polyester fabrics.

The light fastness remained constant with increase in dyeing time except at 90 minutes where it increased slightly.

The wet rubbing fastness remained constant with increase in dyeing time upto 45 minutes. It then decreased slightly and remained constant upto

90 minutes. It showed a slight increase at 105 minutes again. The dry rubbing fastness showed an initial increase with increase in dyeing time. At 60 minutes it showed a decrease and again increased at 75 minutes. On further increase in dyeing time again decrease and then an increase in dry rubbing fastness was observed.

The alkaline perspiration fastness remained constant initially with increase in dyeing time. It increased slightly at 60 minutes and then remained constant with increase in dyeing time. The acidic perspiration fastness remained constant with increase in dyeing time till 45 minutes. It then increased slightly, decreased and became constant on further increase in dyeing time. No staining was observed on adjacent wool and polyester fabrics.

#### **4.6.3.6 Effect of mordanting method**

Table 4.57 depicts the washing, light, rubbing and perspiration fastness grades of Indian blackberry dye on polyester employing different mordant time the three mordanted methods with each mordant.

##### **4.6.3.6.1 Alum**

It is clear from the table that on mordanting with alum, the best fastness grade of washing was observed in simultaneous mordanted sample, followed by that of post-mordanted and pre-mordanted samples. Light fastness was the same with pre and simultaneous mordanting method and

was the best with post-mordanting method. The fastness grades of wet and dry rubbing were maximum in the case of post-mordanted sample, followed by that for pre and simultaneous mordanted samples. Maximum alkaline perspiration fastness grade was observed the case of post-mordanted sample, followed by that for simultaneous and pre-mordanted samples (in that order). Acidic perspiration fastness of post and simultaneous mordanted sample was the same and was minimum with pre-mordanting method.

#### **4.6.3.6.2 Chrome**

In the case of chrome treated samples, washing fastness was the best with simultaneous mordanting method, followed by that for post and pre-mordanting methods and fastness grades decreased with post and pre-mordanting methods. Light and wet rubbing fastness grades of pre and post-mordanted sample were the same and minimum in the case of simultaneous mordanting method. Dry rubbing fastness grades of simultaneous and post-mordanted sample were the same and was the best in the case of pre-mordanting method. Alkaline perspiration fastness grade was maximum in the case of simultaneous mordanting method, followed by the same fastness in the case of pre and post-mordanting methods. Acidic perspiration fastness was maximum for simultaneous mordanted sample, followed by that for pre and post-mordanting methods (in that order).

#### **4.6.3.6.3 Cupric sulphate**

The washing fastness was the best with post-mordanting method and was the same for pre and simultaneous mordanted samples. The light fastness grade was maximum for pre-mordanted sample, followed by that for simultaneous and post-mordanting methods. Wet rubbing fastness grades were the same for simultaneous and post-mordanted samples and minimum with pre-mordanting method. The dry rubbing and alkaline and acidic perspiration fastness grades were minimum for simultaneous mordanted samples and the best with pre and post-mordanting methods.

#### **4.6.3.6.4 Ferrous sulphate**

It can be seen from the table that on mordanting with ferrous sulphate the washing fastness grade was the best in the case of post-mordanted sample, followed by that for simultaneous and pre-mordanting methods. The same light fastness grades were obtained by pre and simultaneous mordanting methods, followed by that for post-mordanted sample. Wet rubbing fastness was maximum for post-mordanted sample, followed by that for simultaneous and pre-mordanting methods. Alkaline and acidic perspiration fastness grades were maximum in the case of post-mordanted sample, followed by that for pre and simultaneous mordanting methods.

No staining was noticed on adjacent fabrics of composite samples of washing and perspiration fastness for all of mordants.

#### **4.6.3.7. Effect of mordant concentration**

The washing, light, rubbing and perspiration fastness grades of polyester sample dyed with Indian blackberry dye using five different concentrations of four different mordants have been presented in Table 4.58.

##### **4.6.3.7.1 Alum**

It is clear from the table that on mordanting with alum the fastness grades of washing, light, wet and dry rubbing for change in colour decreased with increase in mordant concentration and became constant at 10, 15, 10 and 20 per cent concentration, respectively. The acidic and alkaline perspiration fastness grades for change in colour remained constant with increase in concentration of mordant. No staining was noticed on adjacent fabrics of composite samples of washing and perspiration fastness.

##### **4.6.3.7.2 Chrome**

In the case of chrome treated samples, the fastness grades of washing for change in colour increased with increase in concentration of mordant. Light fastness grades remained the same upto 4 per cent concentration except at 2 per cent concentration where it decreased. It again increased at

5 per cent concentration. Wet rubbing fastness increased with increase in concentration upto 4 per cent and then decreased at 5 per cent concentration while dry rubbing fastness grades after remaining constant upto 4 per cent concentration decreased at 5 per cent mordant concentration. The fastness grades of acidic and alkaline perspiration for change in colour remained constant with increase in mordant concentration.

No staining was noticed on adjacent fabrics of composite samples of washing and perspiration fastness.

#### **4.6.3.7.3 Cupric sulphate**

Washing fastness grades for change in colour were constant at 1, 2 and 4 per cent concentration, decreased slightly at 3 per cent concentration and increased slightly at 5 per cent concentration. Light fastness grades remained constant at different mordant concentrations except at 3 per cent concentration where the colour of dyed sample increased on exposure to light. The wet rubbing fastness decreased at 3 and 4 per cent concentration remaining constant at rest of mordant concentration whereas dry rubbing fastness remained unchanged at different mordant concentrations. The acidic and alkaline fastness grades for change in colour were equal which decreased at 2 per cent concentration, increased at 4 per cent concentration and then again decreased at 5 per cent concentration of mordant. No

staining was observed on adjacent fabrics of composite samples of washing and perspiration fastness.

#### **4.6.3.7.4 Ferrous sulphate**

It can be seen from the table that on mordanting with ferrous sulphate, the fastness grades of washing, wet and dry rubbing fastness decreased initially with increase in concentration of mordant upto certain level and then become constant on further increase in mordant concentration. Light fastness grades also decreased with increase in mordant concentration except at 1 and 5 per cent concentration where colour of dyed sample increased and changed, respectively on exposure to light. The acidic and alkaline fastness grades remained constant at different concentrations except acidic perspiration fastness at 3 per cent concentration where slight decrease was noticed in fastness grade.

Staining on adjacent fabrics of composite samples was not observed in both washing and perspiration fastness.

#### **4.6.3.8 Effect of the type of mordant**

The washing, light rubbing and perspiration fastness grade for the polyester sample dyed with Indian blackberry dye using different mordants under optimum conditions have been compared with those of control (unmordanted) sample in the Table 4.65.

**Table 4.65 Effect of mordant type on fastness of Indian blackberry dye on polyester.**

Mordant	WF			LF	RF		PF						
	CC	CSC	CSP	CC	W	D	Al			Ac			
					CSC	CSC	CC	CSP	CSW	CC	CSP	CSW	
Control	1	5	5	3	3	4	4	5	5	5	3/4	5	5
Alum	4	5	5	4	4	3/4	4/5	5	5	5	4/5	5	5
Chrome	4/5	5	5	4	3	2	4/5	5	5	5	4/5	5	5
Cupric sulphate	3/4	5	5	CI	2/3	2/3	4	5	5	5	4	5	5
Ferrous sulphate	4/5	5	5	CI	2/3	2/3	4/5	5	5	5	4/5	5	5

The fastness grades for light increased on application of alum and chrome while colour increased in the case of cupric sulphate and ferrous treated sample on exposure to light.

The dry rubbing fastness decreased on application of mordants while the wet rubbing fastness remained constant for chrome treated sample, increased for alum treated sample and decreased in the case of cupric sulphate and ferrous sulphate treated samples when compared to control sample.

The acidic and alkaline perspiration fastness increased on application of mordants except in the case of alkaline fastness grade for cupric sulphate treated sample where fastness was the same as compared to control sample.

No staining was observed on adjacent fabrics of composite samples used in washing and perspiration fastness. From the mean score of fastness

grades as given in Table 4.58, it is clear that alum was the best mordant followed by chrome, ferrous sulphate and cupric sulphate.

#### 4.6.4 Effect of mordants on CIE LAB values

The effect of different mordants on colour strength, chroma and intensity of the polyester samples dyed with Indian blackberry dye under optimum conditions has been presented in Table 4.66 in terms of K/S and CIE LAB values.

**Table 4.66 Effect of mordants on CIE LAB values of Indian blackberry dye on polyester.**

Mordant	L*	a*	b*	C*	K/S	Colour obtained
Control	60.516	5.628	9.170	10.759	1.73	Light café-au-lait
Alum	61.928	6.103	10.907	12.498	1.99	Café-au-lait
Chrome	61.325	5.812	10.685	12.163	1.99	Café-au-lait
Cupric sulphate	61.180	5.938	11.346	12.806	2.39	Yellowish café-au-lait
Ferrous sulphate	61.138	6.338	11.436	13.075	2.13	Dull café-au-lait

The higher L\* values for all the mordanted samples indicated the lighter shades of mordanted samples as compared to control sample. The values of a\*, b\* and C\* increased for all the mordanted samples showing an increase in redness, yellowness and brightness in the case of mordanted samples as compared to control sample.

The values of K/S increased for all the mordanted samples indicating increase in their colour strength as compared to control sample.



a



b



c



d



e

Golden drop



a



b



c



d



e

Indian blackberry

SS 3 : Polyester samples dyed with golden drop and Indian blackberry dyes under optimum dyeing conditions using different mordants (a - unmordanted sample, b - sample treated with ferrous sulphate, c - sample treated with cupric sulphate, d - sample treated with chrome, e - sample treated with alum).

The colour obtained with Indian blackberry dye under optimized conditions ranged from dull café-au-lait to café-au-lait.

A total of 67 shades of Indian blackberry dye were obtained in the process of optimization of different dyeing parameters.

#### **4.7 Dyeing with Onion dye**

##### **4.7.1 Optimum dyeing conditions without mordanting**

Details of the ranks for the optimization of each variable, namely carrier concentration, carrier treatment time, dye extraction pH, dye extraction time, dyeing pH, dye material concentration and dyeing time and their optimum values have been presented in Table 4.67.

###### **4.7.1.1 Optimum carrier concentration**

It can be seen from Table 4.67 that the colour of the dyed samples improved with the increase in the concentration of carrier, the optimum carrier concentration being 10 per cent. With further increase in concentration of carrier, the colour of dyed samples decreased.

###### **4.7.1.2 Optimum carrier treatment time**

It can be seen from the table that the dyeing improved with the increase in the time of carrier treatment, the optimum time being 60 minutes, beyond which the colour decreased again.

**Table 4.67 Optimization of carrier concentration, carrier treatment time, dye extraction pH, dye extraction time, dyeing pH, dye material concentration and dyeing time of onion dye on polyester on the basis of mean ranks given by a panel of judges.**

Variable	Rank	Optimum value
Carrier concentration (percent)		
5	2	
10	1	
15	5	10
20	3	
25	4	
Carrier treatment time (minutes)		
15	3	
30	4	
45	5	60
60	1	
75	2	
Dye extraction pH		
4	2	
5	1	
6	3	
7	4	5
8	5	
9	6	
10	7	
Dye extraction time (minutes)		
15	6	
30	7	
45	5	
60	4	90
75	3	
90	1	
105	2	
Dyeing pH		
4	1	
5	2	
6	3	
7	4	4
8	5	
9	6	
10	7	
Dye material concentration (g/g)		
0.5	7	
1	5.5	
2	5.5	
3	1	3
4	2	
5	3	
6	4	
Dyeing time (minutes)		
15	7	
30	6	
45	5	
60	3.5	105
75	3.5	
90	2	
105	1	

#### **4.7.1.3 Optimum dye extraction pH**

It can be seen from the Table 4.67 that the extraction of the dye from onion skin powder was the best at pH 5, i.e. in acidic conditions. The extraction decreased with increase in pH and was minimum at pH 10, i.e. in alkaline conditions.

#### **4.7.1.4 Optimum dye extraction time**

The table indicates that the extraction of dye increased with the increase in the duration of extraction. The optimum extraction was obtained in 90 minutes beyond which it decreased again. Similar observations were reported by Vednere et al (1990) for teak leaves.

#### **4.7.1.5 Optimum dyeing pH**

It is clear from the table that the dyeing was the best at pH 4, i.e. in acidic conditions. The dye uptake decreased with the increase in pH and was minimum at pH 10, i.e. in alkaline conditions.

#### **4.7.1.6 Optimum dye material concentration**

The table shows that the optimum was 3g per gram of polyester. The use of increased amount of dye resulted in reduced dye extraction and hence, reduced dye uptake.

#### **4.7.1.7 Optimum dyeing time**

The colour of polyester improved with increase in the duration of dyeing as depicted in Table 4.67 and the optimum dyeing time for onion dye was 105 minutes.

#### 4.7.2 Optimum mordanting conditions

The mordanting method and mordant concentration were optimized on the basis of overall fastness ratings of onion dye dyed polyester employing the carrier concentration, carrier treatment time, dye extraction pH, dye extraction time, dyeing pH, dye material concentration and dyeing time optimized earlier without mordanting. The fastness grades for optimization of mordanting method and mordant concentration are given in Tables 4.68 and 4.69, respectively. The optimum mordanting and mordant concentration for the four mordants have been presented in Table 4.70.

**Table 4.68 Optimization of mordanting method for onion dye on polyester on the basis of fastness grades.**

Mordant	Mor- danting method	WF			LF		RF			PF				MS	Rank
		CC	CSC	CSP	CC	W		D		Al		Ac			
						CSC	CSC	CC	CSP	CC	CSP	CC	CSP		
Alum	Pre	1	5	5	3	3	2	4	5	4/5	4	5	4/5	3.83	2
	Sim	1	5	5	1	2	2	4/5	5	5	2/3	5	5	3.81	3
	Post*	1	5	5	1	3	2/3	4/5	5	5	4/5	5	5	3.87	1
Chrome	Pre	1	5	5	3	1/2	2	4/5	5	5	4/5	5	5	3.83	3
	Sim	2/3	5	5	3	2	2/3	4/5	5	5	3	5	4/5	3.95	2
	Post*	2/3	5	5	3	3	3/4	4/5	5	5	3/4	5	5	4.16	1
Cupric sulphate	Pre	1	5	5	5	2	3	2	5	4/5	3	5	5	3.75	3
	Sim	1	5	5	5	2/3	3	2	5	5	2	5	4/5	3.79	2
	Post*	1	5	5	5	2	3	4	5	5	3/4	5	5	4.04	1
Ferrous sulphate	Pre*	1	5	5	5	2	2/3	4/5	5	4/5	4/5	5	5	4.08	1
	Sim	1	5	5	3	2/3	3/4	4	5	5	3/4	5	5	3.95	2
	Post	1/2	5	5	3	1/2	2/3	4	5	5	3/4	5	5	3.83	3

\*Optimum mordanting method

#### 4.7.2.1 Optimum mordanting method

The fastness ratings for different methods for mordanting have been presented in Table 4.68. The table reveals that the post-mordanting method of mordant application was the best for alum, chrome and cupric sulphate whereas in the case of ferrous sulphate, pre-mordanting method gave the best fastness grades.

**Table 4.69 Optimization of mordant concentration for onion dye on polyester on the basis of fastness grades.**

Mordant	Mordant concentration	WF			LF	RF		PF					MS	Rank	
		CC	CSC	CSP	CC	W	D	Al		Ac					
						CSC	CSC	CC	CSP	CSW	CC	CSP			CSW
Alum	5*	1	5	5	2	2/3	2/3	4/5	5	5	4/5	5	5	3.91	1
	10`	1	5	5	1	3	2/3	4/5	5	5	4/5	5	5	3.87	2
	15	1	5	5	1	2	2	4/5	5	5	4/5	5	5	3.75	4.5
	20	1	5	5	1	2	2	4/5	5	5	4/5	5	5	3.75	4.5
	25	1	5	5	1	3	2	4/5	5	5	4/5	5	5	3.83	3
Chrome	1*	4	5	5	5	2/3	2/3	5	5	5	4/5	5	5	4.45	1
	2	¾	5	5	5	2/3	2/3	4/5	5	5	4/5	5	5	4.16	4
	3	4	5	5	5	2/3	2/3	CI	5	5	4/5	5	5	4.40	3
	4	4	5	5	5	2/3	3	4	5	5	4/5	5	5	4.41	2
	5	4	5	5	1	2/3	3	4/5	5	5	4	5	5	4.08	5
Cupric sulphate	1*	1	5	5	5	2/3	2/3	4/5	5	5	¾	5	5	4.08	1
	2	1	5	5	5	2	3	4	5	5	¾	5	5	4.04	2
	3	1	5	5	5	2	2	3	5	5	¾	5	5	3.87	3.5
	4	1	5	5	5	2	2	3	5	5	3	5	5	3.83	5
	5	1	5	5	5	2	2	4	5	5	2/3	5	5	3.87	3.5
Ferrous sulphate	1	1	5	5	1	2/3	2/3	5	5	5	5	5	5	3.91	4.5
	2	1	5	5	5	2	2/3	4/5	5	4/5	4/5	5	5	4.08	2.5
	3	½	5	5	4	2/3	2/3	4	5	5	4/5	5	5	4.08	2.5
	4*	½	5	5	5	2/3	2/3	4	5	5	4/5	5	5	4.16	1
	5	1	5	5	5	2/3	2/3	3	5	5	3	5	5	3.91	4.5

\*Optimum mordant concentration

#### 4.7.2.2 Optimum mordant concentration

The fastness grades for different concentrations for mordants have been given in Table 4.69. The table makes it clear that the overall fastness grades decreased with increase in the concentration in the case of alum, chrome and cupric sulphate. The fastness grades increased upto certain level and then decreased with in increase in concentration of ferrous sulphate. The optimum concentration of alum, chrome, cupric sulphate and ferrous sulphate were 5, 1, 1 and 4 per cent, respectively (Table 4.70).

**Table 4.70 Optimum mordanting conditions for dyeing polyester with onion dye.**

Mordant	Mordanting method	Mordant concentration (percent)
Alum	Post	5
Chrome	Post	1
Cupric sulphate	Post	1
Ferrous sulphate	Pre	4

#### 4.7.3 Effect of dyeing parameters on the fastness of onion dye

##### 4.7.3.1 Effect of carrier concentration

The washing fastness, light fastness, rubbing fastness and perspiration fastness grades of the onion dye on polyester at different carrier concentrations have been presented in Table 4.71.

The washing fastness remained the same (1) with increase in carrier concentration, i.e. no effect of carrier concentration on washing fastness.

No staining was observed on adjacent fabrics of composite samples.

**Table 4.71 Effect of carrier concentration on fastness of onion dye on polyester.**

Carrier concentration (percent)	WF			LF	RF		PF					
	CC	CSC	CSP	CC	W	D	Al			Ac		
					CSC	CSC	CC	CSP	CSW	CC	CSP	CSW
5	1	5	5	2	2/3	3/4	4	5	5	4	5	5
10	1	5	5	2	2/3	3	4	5	5	4	5	5
15	1	5	5	1	2/3	3	3	5	5	3	5	5
20	1	5	5	1	2/3	3	3	5	5	3/4	5	5
25	1	5	5	1	2/3	3	3	5	5	2/3	5	5

The light fastness decreased at 15 per cent from grade 2-1 and then remained constant (1) with increase in carrier concentration.

The wet rubbing fastness remained constant (2/3) with increase in carrier concentration. The dry rubbing fastness decreased slightly with initial increase in carrier concentration and thereafter remained constant.

The alkaline and acidic perspiration fastness decreased at 15 per cent and thereafter remained constant with increase in carrier concentration. The acidic perspiration fastness decreased at 15 per cent carrier concentration. It then increased followed by a decrease with further increase in carrier concentration. No staining was observed on adjacent fabrics of composite samples.

#### 4.7.3.2. Effect of carrier treatment time

Table 4.72 shows the washing, light, rubbing and perspiration fastness of onion dye on polyester samples treated with carrier for five different time periods.

**Table 4.72 Effect of carrier treatment time on fastness of onion dye on polyester.**

Carrier treatment time (minutes)	WF			LF	RF			PF				
	CC	CSC	CSP	CC	W	D	AI		Ac			
					CSC	CSC	CC	CSP	CSW	CC	CSP	CSW
15	½	5	5	1	2	2	4	5	4/5	4	4/5	4/5
30	½	5	5	1	2/3	2/3	3	5	4/5	3	5	4/5
45	2/3	5	5	5	2	2/3	4	5	4/5	4	5	5
60	1	5	5	2	2/3	3	¾	5	5	¾	5	5
75	1	5	5	2	2/3	3	¾	5	5	¾	5	5

The washing fastness increased initially at 45 minutes and decreased with further increase in carrier treatment time. The washing fastness grades were in the range ½-2/3. No staining was found on adjacent cotton and polyester fabrics.

The light fastness increased with increase in carrier treatment time with an exception at 45 minutes where it showed excellent light fastness with grade 5.

The wet rubbing fastness increased and decreased in alternating manner with increase in carrier treatment time upto 60 minutes and finally became constant. The dry rubbing fastness showed a gradual increase from 2-3 with increase in carrier treatment time. Slight staining was observed on wool fabric of composite samples in the case of dyed samples given carrier treatment for 45 minutes or less duration while no staining was observed on polyester fabrics of composite samples for perspiration fastness.

### 4.7.3.3 Effect of dyeing pH

The fastness grades of washing, light, rubbing and perspiration of onion dye on polyester at different pH values are given in table 4.73.

**Table 4.73 Effect of dyeing pH on fastness of onion dye on polyester.**

Dyeing pH	WF			LF	RF			PF				
	CC	CSC	CSP	CC	W	D	CC	Al		Ac		
					CSC	CSC		CSP	CSW	CC	CSP	CSW
4	1	5	5	1	3	$\frac{3}{4}$	3	5	5	3	5	5
5	1	5	5	4	$\frac{2}{3}$	$\frac{3}{4}$	4	5	5	$\frac{2}{3}$	5	5
6	1	5	5	4	$\frac{2}{3}$	$\frac{3}{4}$	$\frac{3}{4}$	5	5	$\frac{3}{4}$	5	5
7	1	5	5	2	3	$\frac{3}{4}$	$\frac{3}{4}$	5	5	$\frac{3}{4}$	5	5
8	1	5	5	2	3	$\frac{3}{4}$	$\frac{2}{3}$	5	5	2	5	5
9	1	5	5	2	3	3	1	5	5	2	5	5
10	1	5	5	2	3	3	1	5	5	$\frac{1}{2}$	5	5

The washing fastness remained the same with increase in dyeing pH, i.e. remained unaffected. No staining was found on adjacent cotton and polyester fabrics.

The light fastness showed an initial increase at pH 5 and 6 and then decreased with increase in dyeing pH. It became constant with further increase in dyeing pH. The light fastness grades were in the range 1-4.

The wet rubbing fastness remained the same for all dyeing pH except at pH 5 and pH 6 where it decreased slightly. The dry rubbing fastness remained constant with increase in dyeing pH upto pH 8. It then decreased slightly with further increase in dyeing pH. The rubbing fastness grades were in the range 2-3.

The alkaline perspiration fastness showed an increase initially at pH 5. It then decreased gradually with increase in dyeing pH upto 8. The acidic perspiration fastness decreased initially at pH 5. It showed a slight increase at pH 6 and then decreased with further increase in dyeing pH. The perspiration fastness of the dyed samples was poor to very good. No staining was noticed on adjacent wool and polyester fabrics.

#### 4.7.3.4 Effect of dye material concentration

Table 4.74 indicates the fastness grades of onion dye on polyester for washing, light, rubbing and perspiration for different concentrations of dye material.

**Table 4.74 Effect of dye material concentration on fastness of onion dye on polyester.**

Dye concentration (g/g)	WF			LF	RF			PF				
					W	D	Al			Ac		
	CC	CSC	CSP	CC	CSC	CSC	CC	CSP	CSW	CC	CSP	CSW
0.5	1	5	5	2	2/3	3	2/3	5	5	2/3	5	5
1	1	5	5	2	2	3	3	5	5	2	5	5
2	1	5	5	2	2/3	3	3/4	5	5	3/4	5	5
3	1	5	5	4	2/3	3	3/4	5	5	3	5	5
4	1	5	5	4	2/3	3	3/4	5	5	3	5	5
5	1	5	5	4	2/3	2/3	2/3	5	5	2	5	5
6	1	5	5	2	2/3	3	3	5	5	2	5	5

The washing fastness remained the same (1) with increase in dye material concentration. No staining was observed on adjacent cotton and polyester fabrics.

The light fastness remained constant at initial concentrations and increased at 3g/g of dye material concentration. It then remained constant till 5g/g and decreased with further increase in dyeing concentration. The light fastness grades were in the range 2-4.

The wet rubbing fastness remained constant for all dye material concentrations except at 1g/g where it decrease slightly. The dry rubbing fastness also remained constant except at 5g/g dye material concentration where it decreased slightly. The rubbing fastness of the dyed sample was fair to good.

The alkaline perspiration fastness showed a gradual increase till 4g/g of dye material concentration. It then decreased and then again increased with further increase in dyeing material concentration. The acidic perspiration fastness showed an initial decrease, followed by increase and decrease at 2g/g and 3g/g concentration of dye, respectively and thereafter remained constant. The perspiration fastness grades were in the range 2-3/4. No staining was observed on adjacent wool and polyester fabrics.

#### **4.7.3.5 Effect of dyeing time**

The washing, light, rubbing and perspiration fastness grades of onion dye dyed on polyester for different time periods have been presented in Table 4.75.

**Table 4.75 Effect of dyeing time on fastness of onion dye on polyester.**

Dyeing time (minutes)	WF			LF	RF			PF				
	CC	CSC	CSP	CC	W	D	CC	Al		Ac		
					CSC	CSC		CSP	CSW	CC	CSP	CSW
15	1	5	5	2	2	3	½	5	5	½	5	5
30	½	5	5	2	2	2/3	2	5	5	2	5	5
45	1	5	5	2	2	2/3	2	5	5	2	5	5
60	1	5	5	2	2/3	3	3	5	5	¾	5	5
75	1	5	5	2	2/3	2/3	3	5	5	3	5	5
90	1	5	5	2	2	2/3	3	5	5	3	5	5
105	1	5	5	2	2	2	3	5	5	2/3	5	5

The washing fastness remained constant (1) with increase in dyeing time except at 30 minutes where it increased slightly (2/3). No staining was observed on adjacent cotton and polyester fabrics.

The light fastness remained the same (2) with increase in dyeing time.

The wet rubbing fastness remained the same (2) till 45 minutes and then increased to 2/3 with increase in duration of dyeing. It again decreased at 90 minutes. The dry rubbing fastness showed a decrease with increase in dyeing time except at 60 minutes where it increased slightly.

The alkaline perspiration fastness showed an increase with increase in dyeing time and became constant after 60 minutes. The acidic perspiration fastness increased upto 60 minutes and decreased on further increase in dyeing time. The perspiration fastness grades were in the range ½-¾. No staining was noticed on adjacent wool and polyester fabrics.

#### 4.7.3.6 Effect of mordanting method

Table 4.68 depicts the washing, light, rubbing and perspiration fastness grades of onion dye on polyester employing different mordants and the three mordanting methods with each mordant.

##### 4.7.3.6.1 Alum

It is clear from the table that on mordanting with alum no change in fastness grades of washing was observed with different mordanting methods. Light fastness grades were the same (1) for post and simultaneous mordanted samples and was maximum in the case of pre-mordanted sample. Dry rubbing fastness of pre and simultaneous mordanted samples was the same (2) and increased slightly (2/3) with post-mordanting method. Wet rubbing fastness grades were the same (3) for pre and post-mordanted samples and was minimum (2) for simultaneous mordanted sample. Alkaline perspiration fastness was the same (4/5) for simultaneous and post-mordanted samples and was minimum at 4 for pre-mordanted sample. Acidic perspiration fastness was maximum (4/5) for post-mordanted sample, followed by that for pre and simultaneous mordanted samples. Slight staining was observed only on wool fabric of composite sample of pre-mordanted perspiration fastness.

#### **4.7.3.6.2 Chrome**

In the case of chrome treated samples no change in the fastness grades of light (1) and acidic perspiration (4/5) was seen with different mordanting methods. Washing fastness remained the same for simultaneous and post-mordanted samples and was minimum (1) for pre-mordanted sample. Dry and wet rubbing fastness grades were the best for post-mordanted samples, i.e. 3 and  $\frac{3}{4}$ , respectively, followed by that for simultaneous and pre-mordanted samples. The fastness grades of acidic perspiration was maximum (4/5) with pre-mordanted sample, followed by that for post and simultaneous mordanted samples (in that order). No staining was observed on adjacent fabrics of composite samples in washing and perspiration fastness.

#### **4.7.3.6.3 Cupric sulphate**

The fastness grades of washing (1), light (5) and dry rubbing fastness (3) remained the same for different mordanting methods. Wet rubbing fastness grade was the best (2/3) for simultaneous mordanted sample, followed by that for pre and post-mordanted samples which had the same fastness. Alkaline perspiration fastness was the best at 4 for post-mordanted sample and was the same at 2 for pre and simultaneous mordanted sample. Acidic perspiration fastness grade was maximum (3/4) in the case of post-mordanted sample, followed by that for pre and

simultaneous mordanted samples. No staining was noticed on adjacent fabrics of composite samples of washing and perspiration fastness except slight staining on wool fabric of composite sample for acidic perspiration of simultaneous mordanted sample.

#### **4.7.3.6.4 Ferrous sulphate**

It can be seen from the table that on mordanting with ferrous sulphate, washing fastness of pre and simultaneous mordanted samples was the same (1) and was maximum in the case of post-mordanted samples. The fastness grades of light (3), alkaline perspiration (4) and acidic perspiration (3/4) remained the same for post and simultaneous mordanted samples and increased to 5, 4/5 and 4/5, respectively with pre-mordanting method. Wet and dry rubbing fastness was maximum for simultaneous mordanted sample at 2.3 and 3.4, respectively, followed by for that pre and post-mordanting methods. No staining was observed on adjacent fabrics of composite samples of washing and perspiration fastness except wool fabric of composite sample for alkaline perspiration fastness in the case of pre-mordanted sample.

#### **4.7.3.7 Effect of mordant concentration**

The washing, light, rubbing and perspiration fastness grades polyester sample dyed with onion dye using five different mordant concentration have been presented in Table 4.69.

#### **4.7.3.7.1 Alum**

On increasing the concentration of mordant, the washing and acidic and alkaline perspiration fastness grades for change in colour remained the same. Light fastness grades decreased at 10 per cent and remained unchanged on further increase in concentration of mordant. Wet rubbing fastness increased at 10 per cent, decreased at 15 per cent and then increased at 25 per cent concentration of mordant while dry rubbing fastness after remaining same upto 10 per cent concentration decreased at 15 per cent and then remained constant with increase in concentration of mordant. No staining was noticed on adjacent fabrics of composite samples used in washing and perspiration fastness.

#### **4.7.3.7.2 Chrome**

It is clear from the table that on mordanting with chrome washing fastness grades remained the same with increase in mordant concentration except at 2 per cent where it decreased slightly. Light and acidic perspiration fastness remained constant with increase in mordant concentration but marked decrease in light fastness grade and slight decrease in acidic perspiration fastness grade was observed at 25 per cent concentration. Wet rubbing fastness grades were constant with increase in mordant concentration, whereas dry rubbing fastness grades after

remaining constant upto 3 per cent concentration increased slightly at 20 per cent and remained the same at 25 per cent mordant concentration.

Alkaline perspiration fastness grades decreased with increase in mordant concentration and increased at 2 per cent mordant concentration and colour of dyed samples showed an increase at 3 per cent mordant concentration. No staining was noticed on the adjacent fabrics of composite samples of washing and perspiration fastness.

#### **4.7.3.7.3 Cupric sulphate**

In the case of cupric sulphate the washing and light fastness grades remained constant on increase in concentration of mordant. Wet and dry fastness decreased at 2 and 3 per cent concentration, respectively and then remained the same on further increase in concentration of mordant. Acidic and alkaline perspiration fastness grades for change in colour decreased with increase in concentration of mordants except alkaline perspiration fastness grade at 5 per cent concentration of mordant where it increased by a unit. No staining was observed on the adjacent fabrics of composite sample of both washing and perspiration.

#### **4.7.3.7.4 Ferrous sulphate**

It can be seen from the table that on mordanting with ferrous sulphate the fastness grades of washing increased at 3 and 4 per cent concentration and remained the same at 1, 2 and 5 per cent mordant

concentration. A marked increase in the light fastness was observed at 2 per cent concentration which remained constant on further increase in mordant concentration except at 3 per cent concentration where slight decrease in fastness was observed. Both wet and dry rubbing fastness grades improved with increase in the concentration of the mordant except wet rubbing fastness grade at 2 per cent concentration where slight decrease in fastness was noticed. Both acidic and alkaline perspiration fastness decreased with increase in mordant concentration. No staining was noticed on adjacent fabrics of composite samples of washing and perspiration fastness.

#### 4.7.3.8 Effect of the type of mordant

The washing, light rubbing and perspiration fastness grade for the polyester sample dyed with onion dye using different mordants under optimum conditions have been compared with those of control (unmordanted) sample in the Table 4.76.

**Table 4.76 Effect of mordant type on fastness of onion dye on polyester.**

Mordant	WF			LF	RF		PF					
	CC	CSC	CSP	CC	W	D	Al		Ac			
					CSC	CSC	CC	CSP	CSW	CC	CSP	CSW
Control	1	5	5	2	2	2	3	5	5	2/3	5	5
Alum	1	5	5	2	2/3	2/3	4/5	5	5	4/5	5	5
Chrome	4	5	5	5	2/3	2/3	5	5	5	4/5	5	5
Cupric sulphate	1	5	5	5	2/3	2/3	4/5	5	5	3/4	5	5
Ferrous sulphate	1/2	5	5	5	2/3	2/3	4	5	5	4/5	5	5

The fastness grades of light, wet and dry rubbing and alkaline and acidic perspiration increased on application of different mordants except the light fastness grade of alum treated sample. The light fastness grade 5 was also observed by Gulrajani et al (1992b) in the case of chrome, cupric sulphate and ferrous sulphate treated onion dyed wool.

The washing fastness showed a marked increase on application of chrome, slight increase on application of ferrous sulphate and remained constant on application of alum and cupric sulphate as mordants when compared with control sample. Gulrajani et al (1992b) also reported very good washing fastness of chrome treated onion dyed wool.

No staining was noticed on adjacent fabrics of composite samples used in washing perspiration fastness.

It can be seen from the mean score of fastness grades as given in Table 4.69 that chrome was the best mordant followed by ferrous sulphate, cupric sulphate and alum.

#### **4.7.4 Effect of mordants on CIE LAB values**

The effect of different mordants on colour strength, chroma and intensity of the polyester samples dyed with onion dye under optimum conditions has been presented in Table 4.77 in terms of K/S and CIE LAB values.

**Table 4.77 Effect of mordants on CIE LAB values of onion dye on polyester.**

Mordant	L*	a*	b*	C*	K/S	Colour obtained
Control	55.262	3.254	17.860	18.154	3.84	Sallow
Alum	56.904	3.728	23.321	23.617	4.97	Greyish fawn
Chrome	50.535	6.473	13.481	14.955	3.42	Drab/Mouse grey
Cupric sulphate	49.953	4.850	14.948	15.715	3.15	Café-au-lait
Ferrous sulphate	47.386	5.032	10.564	11.701	3.52	Hair brown

The lower L\* values for all the mordanted samples except in the case of alum treated sample indicated that the mordanted samples were darker than control sample except alum treated sample. The higher a\* values of all the mordanted samples in comparison to control sample showed an increase in redness of all the mordanted samples. The b\* and C\* values increased only in case of alum treated samples and hence, the yellowness and brightness increased only in the case of alum treated sample.

The colour strength also increased only in alum treated sample while other mordanted samples exhibited lower colour strength as seen from their K/S values in Table 4.77.

The colour obtained with onion dye under optimized conditions ranged from sallow to greyish fawn.

A total of 67 shades of onion dye were obtained in the process of optimization of different dyeing parameters.

## **4.8 Dyeing with Pine dye**

### **4.8.1 Optimum dyeing conditions without mordanting**

Details of the ranks for the optimization of each parameter, namely carrier concentration, carrier treatment time, dye extraction pH, dye extraction time, dyeing pH, dye material concentration and dyeing time and their optimum values have been presented in Table 4.78.

#### **4.8.1.1 Optimum carrier concentration**

It can be seen from Table 4.78 that the colour of the dyed samples improved with the increase in the concentration of carrier, the optimum carrier concentration being 10 per cent. With further increase in concentration of carrier, the colour of dyed samples decreased.

#### **4.8.1.2 Optimum carrier treatment time**

It can be seen from table that the best colour on polyester was obtained when the carrier treatment was given for 45 minutes after which the colour of dyed samples decreased again. Thus, the optimum carrier treatment time was 45 minutes.

#### **4.8.1.3 Optimum dye extraction pH**

It can be seen from the Table 4.78 that extraction of dye from bark powder was the best at pH 5, i.e. in acidic conditions beyond which it

**Table 4.78 Optimization of carrier concentration, carrier treatment time, dye extraction pH, dye extraction time, dyeing pH, dye material concentration and dyeing time of pine dye on polyester on the basis of mean ranks given by judges.**

Variable	Rank	Optimum value
Carrier concentration (percent)		
5	3.5	
10	1	
15	5	10
20	2	
25	3.5	
Carrier treatment time (minutes)		
15	3	
30	5	
45	1	45
60	4	
75	2	
Dye extraction pH		
4	2	
5	1	
6	3	
7	4.5	5
8	4.5	
9	6	
10	7	
Dye extraction time (minutes)		
15	4	
30	7	
45	6	
60	5	90
75	3	
90	1	
105	2	
Dyeing pH		
4	5	
5	4	
6	2	
7	1	7
8	3	
9	6	
10	7	
Dye material concentration (g/g)		
0.5	1	
1	2	
2	3	
3	4	0.5
4	5	
5	6	
6	7	
Dyeing time (minutes)		
15	7	
30	6	
45	4.5	
60	4.5	105
75	3	
90	2	
105	1	

decreased with increase in pH. The extraction of dye was minimum at pH 10, i.e. in alkaline conditions.

#### **4.8.1.4 Optimum dye extraction time**

The table indicates that the extraction of dye increased with the increase in the duration of extraction. The optimum extraction was obtained in 90 minutes beyond which it decreased again. Similar observations were reported by Dayal et al (2001) for *Pinus roxburghii* bark dye on silk, wool and cotton.

#### **4.8.1.5 Optimum dyeing pH**

It is clear from the table that as the pH increased, the dyeing improve and was the best at neutral pH. Further increase in pH resulted in duller colours. The neutral pH, i.e. pH 7 was, therefore, adjudged optimum for application of ashoka dye.

#### **4.8.1.6 Optimum dye material concentration**

The table showed that the optimum dye material concentration, was 0.5 g per gram of polyester. The dyeing decreased with further increase in concentration of dye material. Bahl and Gupta (1988) also reported use of 0.5 g/50 ml cutch dye on silk.

#### **4.8.1.7 Optimum dyeing time**

The colour of polyester improved with increase in the duration of dyeing as depicted in Table 4.78 and the optimum dyeing time for pine dye

was 105 minutes. Dayal et al (2001) reported similar dyeing time for *Pinus roxburghii* bark dye on silk, wool and cotton.

#### 4.8.2 Optimum mordanting conditions

The mordanting method and mordant concentration were optimized on the basis of overall fastness ratings of pine dye dyed polyester employing the carrier concentration, carrier treatment time, dye extraction pH, dye extraction time, dyeing pH, dye material concentration and dyeing time optimized earlier without mordanting. The fastness grades for optimization of mordanting method and mordant concentration are given in Tables 4.79 and 4.80, respectively. The optimum mordanting and mordant concentration for the four mordants have been presented in Table 4.81.

**Table 4.79 Optimization of mordanting method for pine dye on polyester on the basis of fastness grades.**

Mordant	Mor- danting method	WF			LF	RF			PF				MS	Rank	
		CC	CSC	CSP	CC	W		CC	Al		Ac				
						CSC	CSC		CSP	CSP	CSW	CC			CSP
Alum	Pre	4/5	4/5	4/5	CI	2/3	2/3	4	4/5	4/5	3/4	4/5	4/5	4.0	3
	Sim	4/5	5	5	CI	2	1/2	3	5	5	5	5	5	4.18	2
	Post*	CI	4/5	5	CC	2/3	2	4/5	5	5	4	5	5	4.25	1
Chrome	Pre	4	4/5	4/5	CI	2/3	3	3	4/5	4/5	2	4/5	4/5	3.77	3
	Sim	4/5	5	5	CI	1/2	1/2	3	5	5	2/3	4/5	5	3.86	2
	Post*	4	5	5	CI	3/4	2/3	4/5	5	5	3/4	5	5	4.36	1
Cupric sulphate	Pre	4/5	4/5	4/5	CI	2	2	4	4/5	4/5	2	4/5	4/5	3.77	3
	Sim	4/5	5	5	CI	3	2	3	5	5	4	5	5	4.22	2
	Post*	3/4	5	5	CI	2/3	2	4/5	5	5	4/5	5	5	4.27	1
Ferrous sulphate	Pre	4	4/5	4/5	CI	1/2	1	4	5	4	4/5	4	5	3.81	2
	Sim*	4	5	5	CI	2/3	2/3	3/4	5	5	4	5	5	4.31	1
	Post	3/4	5	5	1/2	1/2	1/2	3/4	4/5	5	2/3	5	5	3.62	3

\*Optimum mordanting method

### 4.8.2.1 Optimum mordanting method

The fastness ratings for different methods of mordanting have been presented in Table 4.79. The table reveals that the post-mordanting method was the best (highest mean score) for alum, chrome and cupric sulphate whereas in the case of ferrous sulphate, simultaneous mordanting method gave the best method to obtain best fastness grades.

**Table 4.80 Optimization of mordant concentration for pine dye on polyester on the basis of fastness grades.**

Mordant	Mordant concentration	WF			LF		RF			PF				MS	Rank
		CC	CSC	CSP	CC	W		D		Al		Ac			
						CSC	CSC	CC	CSP	CSW	CC	CSP	CSW		
Alum	5	4/5	5	5	CC	2/3	2	4	5	5	3/4	5	5	4.22	5
	10	CI	5	5	CC	2/3	2	4/5	5	5	4	5	5	4.25	4
	15	CI	5	5	CC	2/3	2	4/5	5	5	4	5	5	4.30	2.5
	20*	CI	5	5	CC	3	2/3	4	5	5	4	5	5	4.35	1
	25	CI	5	5	CC	3	2/3	4	5	5	3/4	5	5	4.30	2.5
Chrome	1	CC	5	5	1	3/4	3/4	3	5	4/5	3/4	5	5	4.00	5
	2*	4	5	5	CI	2/3	3/4	4/5	5	5	3/4	5	5	4.31	1
	3	4	5	5	CC	2/3	3	4	5	5	3/4	5	5	4.27	2
	4	4/5	5	5	1	2/3	2/3	4	5	5	4/5	5	5	4.08	4
	5	4	5	5	CC	2/3	2/3	3/4	5	5	3/4	5	5	4.18	3
Cupric sulphate	1	4	5	5	CC	3	2	4	4/5	4/5	4	4/5	4/5	4.09	3
	2*	3/4	5	5	CI	2/3	2	4/5	5	5	4/5	5	5	4.27	1
	3	CI	5	5	CC	2/3	1	4	5	5	4	5	5	4.15	2
	4	4/5	5	5	1	2/3	1	3/4	4/5	4/5	3/4	4/5	4/5	3.66	5
	5	4/5	5	5	CC	2/3	1	3/4	4/5	4/5	4	4/5	4/5	3.95	4
Ferrous sulphate	1	4	5	5	CC	3	1/2	3/4	5	4/5	5	5	4/5	4.13	4
	2	4	5	5	CI	2/3	2/3	3/4	5	5	4	5	5	4.31	2
	3*	CI	5	5	CI	3	2	4/5	5	5	4	5	5	4.35	1
	4	CI	5	5	CC	3	2	4/5	5	4/5	4	5	4/5	4.25	3
	5	CI	5	5	CC	3	2	CI	5	4/5	CC	5	4/5	4.12	5

\*Optimum mordant concentration

#### 4.8.2.2 Optimum mordant concentration

The fastness grades for different concentrations of mordants have been given in Table 4.80. The table makes it clear that the overall fastness grades increased with increase in mordant concentration upto certain level and then decreased with increase in the concentration in the case of all the mordants. The optimum concentration of alum, chrome, cupric sulphate and ferrous sulphate were 20, 2, 2 and 3 per cent, respectively (Table 4.81).

**Table 4.81 Optimum mordanting conditions for dyeing polyester with pine dye.**

Mordant	Mordanting method	Mordant concentration (percent)
Alum	Post	20
Chrome	Post	2
Cupric sulphate	Post	2
Ferrous sulphate	Sim	3

#### 4.8.3 Effect of dyeing parameters on the fastness of pine dye

##### 4.8.3.1 Effect of carrier concentration

The washing, light, rubbing and perspiration fastness grades of pine dye on polyester at different carrier concentrations have been presented in Table 4.82.

**Table 4.82 Effect of carrier concentration on fastness of pine dye on polyester.**

Carrier concentration (percent)	WF			LF	RF		PF						
	CC	CSC	CSP	CC	W	D	Al			Ac			
					CSC	CSC	CC	CSP	CSW	CC	CSP	CSW	
5	5	5	5	CC	2/3	2/3	5	5	5	5	5	5	5
10	4/5	5	5	CC	2/3	2/3	4	5	5	4	5	5	5
15	CC	5	5	CC	2/3	3	3	5	5	4	5	5	5
20	CI	5	5	CC	2/3	2/3	4	5	5	4/5	5	5	5
25	CI	5	5	CC	2/3	2/3	4	5	5	4	5	5	5

Washing fastness grades decreased initially with increase in concentration of carrier but at 15 per cent concentration the colour of dyed samples changed and increased on further increase in carrier concentration. Washing fastness of dyed samples was very good. No staining was noticed on adjacent cotton and polyester fabrics of complete samples.

The colour of dyed samples changed on exposure to light at different carrier concentrations.

Wet and dry rubbing grades also remained the same (2/3) for different carrier concentrations except dry rubbing fastness at 15 per cent where slight increase was observed in fastness grades.

Alkaline and acidic perspiration fastness grades decreased initially and then remained constant (4) except alkaline perspiration fastness which decreased at 15 per cent and acidic perspiration fastness which increased at 20 per cent carrier concentration. No staining was observed on adjacent wool and polyester fabrics of composite samples.

**Table 4.83 Effect of carrier treatment time on fastness of pine dye on polyester.**

Carrier treatment time (minutes)	WF			LF	RF			PF				
	CC	CS	PS	CC	W	D	CC	Al		Ac		
					CS	CS		PS	WS	CC	PS	WS
15	4	5	5	2	3	3	3/4	5	5	3	5	5
30	CC	5	5	2	3	4	3	5	5	5	5	5
45	4/5	5	5	2	2/3	2/3	3/4	5	5	4	5	5
60	4/5	5	5	CC	2/3	2/3	4	5	5	4	5	5
75	CC	5	5	CC	2/3	2/3	4	5	5	3/4	5	5

### 8.3.2 Effect of carrier treatment time

Table 4.83 shows the washing, light, rubbing and perspiration fastness of pine dye on polyester samples treated with carrier for five different time periods.

Washing fastness of dyed samples was very good and was the same at different carrier treatment time periods except at 30 minutes and 75 minutes where colour of dyed samples changed on washing. No staining was noticed on adjacent fabrics of composite samples.

Light fastness was poor and fastness grades were the same except at 60 minutes and 75 minutes of carrier treatment time where the colour of dyed sample changed on exposure to light.

Wet rubbing fastness decreased with increase in duration of carrier treatment remaining the same upto 60 minutes of carrier treatment. Dry rubbing fastness increased at 30 minutes, decreased at 45 minutes and remained the same on further increase in duration of carrier treatment. Rubbing fastness was fair to very good with fastness grades in the range 2/3 to 4.

The alkaline perspiration fastness showed an increase with increase in carrier treatment time except at 30 minutes where it decreased. The acidic perspiration fastness grades increased initially with increase in carrier treatment time upto 60 minutes. With further increase in carrier

treatment time it decreased. The perspiration fastness of the dyed samples was good to excellent. No staining was observed on adjacent wool and polyester fabrics.

#### 4.8.3.3 Effect of dyeing pH

The fastness grades of washing, light, rubbing and perspiration of pine dye on polyester at different pH values are given in 4.84.

**Table 4.84 Effect of dyeing pH on fastness of pine dye on polyester.**

Dyeing pH	WF			LF	RF		PF					
	CC	CSC	CSP	CC	W	D	CC	Al		Ac		
					CSC	CSC		CSP	CSW	CC	CSP	CSW
4	CI	5	5	CI	3	3	4	5	4/5	3/4	5	5
5	4/5	5	5	CI	3	3/4	4	5	4/5	3/4	5	4/5
6	4	5	5	CC	3	3	4	5	4/5	3/4	5	4/5
7	4/5	5	5	CC	2/3	2/3	4	5	5	4	5	4/5
8	CC	5	5	1	3	3/4	4	5	5	3	5	4/5
9	3	5	5	1	3	3	4	5	5	3	5	4/5
10	3/4	5	5	2	3	4	4	5	5	3	5	4/5

Washing fastness grades showed increase and decrease with every alternating pH values and fastness of dyed samples was good to very good with fastness grades in range 3 to 4/5. The adjacent cotton and polyester fabrics showed no staining.

Light fastness was poor at higher pH values, i.e. in alkaline conditions. The colour of dyed samples changed in terms of hue at pH 6 and 7 whereas increase in colour of dyed samples was noticed at pH 4 and 5, i.e. in acidic conditions.

Wet rubbing fastness grades remained the same with increase in dyeing pH except at pH 7 where slight decrease was seen. Dry rubbing fastness grades showed increase and decrease in alternating manner with increase in the pH of dyeing. Rubbing fastness of dyed samples was fair to very good with fastness grades in the range 2/3 to 4.

Alkaline perspiration fastness grades were the same (4) even on increasing pH of dyeing whereas acidic perspiration fastness grades increased at neutral pH, then decreased and remained the same (3) on further increase in pH value of dyeing. Slight staining was noticed on wool fabric at pH values below 7 in case of alkaline perspiration and at all pH values except pH 4 in the case of acidic perspiration fastness.

**Table 4.85 Effect of dye material concentration on fastness of pine dye on polyester.**

Dye concentration (g/g)	WF			LF	RF			PF				
					W	D	Al			Ac		
	CC	CSC	CSP	CC	CSC	CSC	CC	CSP	CSW	CC	CSP	CSW
0.5	2	5	5	CC	2	1/2	4	5	5	4	5	5
1	4	5	5	CC	2/3	2/3	4	5	5	4	5	5
2	4/5	5	5	CC	2/3	2/3	4	5	5	4	5	5
3	4	5	5	CC	3/4	3/4	3/4	5	5	3/4	5	5
4	4	5	5	1	3/4	4	3/4	5	5	3/4	5	5
5	3/4	5	5	1	3	3	3	5	5	3	5	5
6	3/4	5	5	1	3	3	3	5	5	3	5	5

#### 4.8.3.4 Effect of dye concentration

Table 4.85 indicates the fastness grades of pine dye on polyester for washing, light, rubbing and perspiration for different concentrations of dye material.

The washing fastness grades initially increased and then decreased with increase in concentration of dye material concentration. The washing fastness of dyed samples was fair to very good with fastness grades in range 2 to 4/5.

Light fastness of the dyed samples was poor at different dye material concentrations with fastness grade 1 at 4 g/g concentration and above. In the case of concentrations below 4 g/g the change in the colour of dyed samples was observed.

Wet and dry rubbing fastness grades increased with increase in concentration of dye material concentration upto 4 g/g and decreased on further increase in concentration of dye material. Rubbing fastness of dyed samples was poor to very good with fastness grades in range  $\frac{1}{2}$  to 4.

Alkaline and acidic perspiration fastness grades decreased to  $\frac{3}{4}$  with increase in concentration of dye material after remaining constant (4) upto 2 g/g concentration of dye material. No staining was noticed on adjacent wool and polyester fabrics of composite samples.

#### 4.8.3.5 Effect of dyeing time

The washing, light, rubbing and perspiration fastness grades of pine dye dyed on polyester for different time periods have been presented in Table 4.86.

**Table 4.86 Effect of dyeing time on fastness properties of pine dye on polyester.**

Dyeing time (minutes)	WF			LF	RF			PF				
	CC	CSC	CSP	CC	W	D	Al		Ac			
					CSC	CSC	CC	CSP	CSW	CC	CSP	CSW
15	CC	5	5	CI	3/4	3/4	3/4	5	5	3	5	5
30	CC	5	5	CC	3	3	3/4	5	5	3	5	5
45	4	5	5	CI	3	2/3	3/4	5	5	3/4	5	5
60	4/5	5	5	CC	2/3	2/3	4	5	5	4	5	5
75	2/3	5	5	CI	2/3	2	4	5	5	4	5	4/5
90	2	5	5	CC	2	2	4	5	5	4	5	4/5
105	2	5	5	CC	2	2	4	5	5	4	5	4/5

Washing fastness grades decreased with increase in duration of dyeing upto 60 minutes. The fastness grade was decreased slightly at 45 minutes and change in the colour of dyed samples at 15 minutes and 30 minutes. Washing fastness was fair to very good. No staining was observed on adjacent cotton and polyester fabrics.

The colour of dyed sample increased and changed in terms of hue in alternating manner on increase in the duration of dyeing.

Wet and dry rubbing fastness grades showed decrease on increase in duration of dyeing and was fair to good with grades in range 2 to 3/4.

Alkaline and acidic perspiration fastness grades showed an increase with increase in the duration of dyeing and remained the same for samples dyed for 60 minutes or for more duration.

Wool fabric of composite sample showed slight staining in the case of samples dyed for longer duration while no staining was observed on polyester fabrics of composite samples.

#### **4.8.3.6. Effect of mordanting method**

Table 4.79 depicts the washing, light, rubbing and perspiration grades of pine dye on polyester employing different mordants and the three mordanting methods with each mordant.

##### **4.8.3.6.1 Alum**

It can be seen from the table that on mordanting with alum, the washing fastness grades were the same at 4/5 for both pre and simultaneous mordanted samples and in the case of post mordanted sample, the colour of dyed sample increased on washing. On exposure to light, colour of dyed samples increased in the case of pre and simultaneous mordanting methods and changed in the case of post-mordanting method. Wet rubbing fastness grades at 2/3 were the same for pre and post-mordanted samples, followed by that for simultaneous mordanted sample. Dry rubbing fastness was maximum (2/3) in the case of pre-mordanting method, followed by that for post and simultaneous mordanting methods. Alkaline fastness grade was

maximum 4/5 in the case of post-mordanting method, followed by that for pre and simultaneous mordanted samples. Acidic perspiration fastness grade was maximum at 5 in the case of simultaneous mordanting method, followed by that for post and pre-mordanting methods. Slight staining was observed on cotton fabrics of pre and post-mordanted samples and polyester fabric of pre-mordanted sample in the case of washing fastness. In the case of perspiration fastness, staining was noticed on polyester and wool fabrics of pre-mordanted samples for both acidic and alkaline perspiration fastness.

#### 4.8.3.6.2 Chrome

In the case of chrome, the increase in colour of dyed samples was observed on exposure to light all the mordanting methods. The fastness grades of washing were the same (4) for pre and post-mordanted samples and was the best (4/5) for simultaneous mordanted sample. Wet rubbing fastness grade was the best (3/4) in the case of post-mordanting method, followed by that for pre and simultaneous mordanting methods. Alkaline perspiration fastness grades was maximum (4/5) for post-mordanted sample and remained the same (3) for pre and simultaneous mordanted samples (in that order). Acidic perspiration fastness was the best (3/4) in the case of post-mordanting method, followed by that for simultaneous and pre-mordanting methods. Staining was observed on cotton and polyester fabrics of pre-mordanted samples in the case of washing fastness. In the case of

perspiration fastness, staining was noticed on wool and polyester fabrics of pre-mordanted samples for both acidic and alkaline perspiration fastness and slight staining on wool fabric only was observed in simultaneous mordanted sample for acidic perspiration fastness.

#### **4.8.3.6.3 Cupric sulphate**

The increase in colour of dyed samples was observed on exposure to light in the case of all mordanting methods. The washing fastness grades remained the same (4/5) in pre and simultaneous mordanting methods and was minimum in the case of post-mordanting method. Wet rubbing fastness was maximum (3) in the case of simultaneous mordanting method, followed by that for post and pre-mordanting methods. Dry rubbing fastness grades remained the same at 2 with different mordanting method. Alkaline and acidic perspiration fastness was maximum at 4/5 in the case of post-mordanting method, followed by that for in the case of other two methods. Staining was observed on both adjacent fabric of pre-mordanted samples for both washing and perspiration fastness.

#### **4.8.3.6.4 Ferrous sulphate**

It is clear from the table that on mordanting with ferrous sulphate, the washing fastness remained the same at 4 for pre and simultaneous mordanted samples and was minimum ( $\frac{3}{4}$ ) for post-mordanted sample. Increase in colour of dyed sample was observed in the case of pre and

simultaneous mordanted samples on exposure to light. The light fastness was poor ( $\frac{1}{2}$ ) with post-mordanting method. Wet and dry rubbing fastness grades were maximum at  $\frac{2}{3}$  in simultaneous mordanting method, followed by that for pre and post-mordanting methods. Alkaline perspiration fastness remained the same ( $\frac{3}{4}$ ) in post and simultaneous mordanting methods and was the best (4) in the case of pre-mordanting method. Acidic perspiration fastness grade was maximum at  $\frac{4}{5}$  with pre-mordanting method, followed by that for simultaneous (4) and post ( $\frac{2}{3}$ ) mordanted samples. Staining was noticed on cotton and polyester fabrics of pre-mordanted sample of washing fastness. In the case of perspiration fastness, polyester fabrics showed staining for pre-mordanted sample of acidic perspiration fastness and post-mordanted sample of alkaline perspiration fastness.

#### **4.8.3.7 Effect of mordant concentration**

The washing, light, rubbing and perspiration fastness grades of polyester sample dyed with pine dye using five different mordant concentrations have been presented in Table 4.80.

##### **4.8.3.7.1 Alum**

The colour of the dyed samples increased on washing except at 5 per cent concentration at which very good fastness was observed. The colour of dyed samples changed on exposure to light at all the concentrations of mordant. Wet and dry rubbing fastness remained the same upto 15 per cent,

then increased and remained the same at 20 and 25 per cent concentrations of mordant. The alkaline and acidic perspiration fastness grades increased initially, then remained the same and decreased with further increase in the concentration of mordant. No staining was noticed on adjacent fabrics of composite samples of washing and perspiration fastness.

#### **4.8.3.7.2 Chrome**

It can be seen from the table that on mordanting with chrome the washing fastness remained the same on increasing the concentration of mordant except at 4 per cent where it increased slightly. Light fastness remained the same at 1 and 4 per cent concentrations and the colour of the dyed samples either changed or increased at rest of the concentrations of mordant. Wet rubbing fastness decreased with increase in concentration while dry rubbing fastness decreased at 2 per cent concentration and then remained the same even on increase in concentration of mordant. Alkaline perspiration fastness increased initially and then decreased with increase in concentration of mordant. The acidic fastness grades remained the same on increase in mordant concentration except at 4 per cent where fastness showed an increase. No staining was noticed on fabrics of composite samples of washing and perspiration fastness except slight staining on wool fabric at 1 per cent concentration in the case of alkaline perspiration fastness.

#### **4.8.3.7.3 Cupric sulphate**

In the case of cupric sulphate washing fastness grades increased with increase in mordant concentration except 2 per cent where fastness grade decreased slightly and 3 per cent where the colour of dyed sample increased on washing. The colour of dyed samples either increased or changed at different concentrations of mordant on exposure to light. Wet and dry fastness grades decreased initially and then remained constant on further increase in the concentration of mordant. The acidic and alkaline perspiration fastness grades increased initially at 2 per cent concentration and then decreased on further increase in mordant concentration. No staining on adjacent cotton and polyester fabrics was noticed in the case of washing fastness samples whereas slight staining was observed on both wool and polyester fabrics at 1, 4 and 5 per cent mordant concentrations in the case of perspiration fastness samples.

#### **4.8.3.7.4 Ferrous sulphate**

Washing fastness remained the same upto 2 per cent concentration after which the colour of dyed samples increased on washing. The colour of dyed samples either changed or increased at different mordant concentrations on exposure to light. Wet rubbing fastness was the same at different mordant concentrations except at 2 per cent where it decreased slightly. Dry rubbing fastness increased initially at 2 per cent, decreased at

3 per cent and then remained the same on further increase in mordant concentration. Alkaline perspiration fastness increased with increase in mordant concentration whereas acidic perspiration fastness decreased at 2 per cent and then remained the same on further increase in mordant concentration. At 5 per cent concentration, the colour of dyed sample increased and changed in alkaline and acidic perspiration fastness, respectively. No staining was observed on cotton and polyester fabrics in the case of wash fastness samples while slight staining was noticed on wool fabric at 4 per cent concentration in both acidic and alkaline perspiration fastness samples.

#### 4.8.3.8 Effect of the type of mordant

The washing, light rubbing and perspiration fastness grade for the polyester sample dyed with pine dye using different mordants under optimum conditions have been compared with those of control (unmordanted) sample in the Table 4.87.

**Table 4.87 Effect of mordant type on fastness of pine dye on polyester.**

Mordant	WF			LF	RF		PF					
	CC	CSC	CSP	CC	W	D	Al			Ac		
					CSC	CSC	CC	CSP	CSW	CC	CSP	CSW
Control	2	5	5	CC	2	2	4	5	4/5	4	5	4/5
Alum	CI	5	5	CC	2/3	2/3	4	5	5	4	5	5
Chrome	4	5	5	CI	3/4	2/3	4/5	5	5	3/4	5	5
Cupric sulphate	3/4	5	5	CI	2/3	2	4/5	5	5	4/5	5	5
Ferrous sulphate	CI	5	5	CI	3	2	4/5	5	5	4	5	5

The colour of dyed samples either changed or increased when exposed to light on application of different mordants. The colour of samples increased on washing when alum and ferrous sulphate were applied. The washing fastness grades showed marked increase when chrome and cupric sulphate were applied. The wet rubbing fastness improved in the case of all the mordanted samples while dry rubbing fastness improved on application of alum and chrome and remained the same on application of cupric sulphate and ferrous sulphate.

The alkaline perspiration fastness grades improved for chrome, cupric sulphate and ferrous sulphate treated samples and remained the same for alum treated sample whereas the acidic perspiration fastness grades improved in the case of cupric sulphate treated sample, remained the same for alum and ferrous sulphate treated samples and decreased in the case of chrome treated sample.

No staining was observed on adjacent fabrics of composite samples of washing and perspiration fastness except for slight staining on wool fabric in the case of acidic perspiration fastness of control sample. On the basis of the mean scores given in Table 4.80 it is clear that alum treated sample had best overall fastness followed by chrome and ferrous sulphate treated samples. The cupric sulphate treated sample had least fastness.

#### 4.8.4 Effect of mordants on CIE LAB values

The effect of different mordants on colour strength, chroma and intensity of the polyester samples dyed with pine dye under optimum conditions has been presented in Table 4.88 in terms of K/S and CIE LAB values.

**Table 4.88 Effect of mordants on CIE LAB values of pine dye on polyester.**

Mordant	L*	a*	b*	C*	K/S	Colour obtained
Control	48.388	12.918	9.804	16.217	3.16	Light violet brown
Alum	52.366	14.907	14.053	20.487	3.31	Brownish rosewood
Chrome	48.893	12.788	10.388	16.476	3.77	Greyish madeira
Cupric sulphate	51.448	13.265	13.265	19.017	4.47	Light brownish rosewood
Ferrous sulphate	49.165	12.190	11.468	16.737	8.10	Light greyish madeira

The higher L\* values than that of control sample value indicated the lighter shade of mordanted samples. The redness, as depicted by a\* values increased only in case of alum and cupric sulphate treated sample. The yellowness showed an increase in case of all the mordanted samples as can be seen by higher b\* values. Due to increase in yellowness, all the mordanted samples became brighter as supported by higher C\* values as compared control sample value.

The increase in K/S value of all the mordanted samples indicated increased colour strength of all the mordanted sample as compared to



a



b



c



d



e

Onion



a



b



c



d



e

Pine

Fig 4: Polyester samples dyed with onion and pine dyes under optimum dyeing conditions using different mordants (a - unmordanted sample, b - sample treated with ferrous sulphate, c - sample treated with cupric sulphate, d - sample treated with chrome, e - sample treated with alum).

colour strength of control sample. The colour obtained with pine dye under optimized conditions ranged from light violet brown to brownish rosewood.

A total of 67 shades of pine dye were obtained in the process of optimization of different dyeing parameters.

#### **4.9 Dyeing with Rubber plant dye**

##### **4.9.1 Optimum dyeing conditions of rubber plant dye without mordanting**

Details of the ranks for the optimization of each parameter, namely carrier concentration, carrier treatment time, dye extraction pH, dye extraction time, dyeing pH, dye material concentration and dyeing time and their optimum values have been presented in Table 4.89.

###### **4.9.1.1 Optimum carrier concentration**

It can be seen from the Table 4.89 that the colour of the dyed polyester improved with the increase in the concentration of carrier, the optimum carrier concentration being 10 per cent, beyond which it decreased.

###### **4.9.1.2 Optimum carrier treatment time**

It can be seen from the table that the dyeing improved with increase in the time of carrier treatment and the optimum time was 75 minutes.

###### **4.9.1.3 Optimum dye extraction pH**

It can be seen from the Table 4.89 that extraction of dye from leaves power was the best at pH 6, i.e. in slight acidic conditions. The extraction

**Table 4.89 Optimization of carrier concentration, carrier treatment time, dye extraction pH, dye extraction time, dyeing pH, dye material concentration and dyeing time of rubber plant dye on polyester on the basis of mean ranks given by a panel of judges.**

Variable	Rank	Optimum value
Carrier concentration (percent)		
5	3	
10	4	
15	5	20
20	1	
25	2	
Carrier treatment time (minutes)		
15	5	
30	4	
45	2	75
60	3	
75	1	
Dye extraction pH		
4	6	
5	2	
6	1	6
7	3	
8	4	
9	5	
10	7	
Dye extraction time (minutes)		
15	3	
30	5	
45	2	
60	1	60
75	4	
90	6	
105	7	
Dyeing pH		
4	7	
5	4	
6	3	
7	2	8
8	1	
9	5.5	
10	5.5	
Dye material concentration (g/g)		
0.5	7	
1	6	
2	3.5	
3	1	3
4	2	
5	3.5	
6	5	
Dyeing time (minutes)		
15	7	
30	6	
45	5	
60	4	75
75	1	
90	3	
105	2	

of dye decreased with increase in pH and was minimum at pH 10, i.e. in alkaline conditions.

#### **4.9.1.4 Optimum dye extraction time**

The table indicates that the extraction of dye increased with the increase in the duration of extraction 60 minutes was adjudged the best times for maximum extraction of dye. Extraction for a longer period was not found helpful in increasing the dye extraction. Similar results were reported by Katyayini and Jacob (1998) and Kumar and Bharat (1998) for mesta calyx and *Mangifera indica* dyes, respectively.

#### **4.9.1.5 Optimum dyeing pH**

It is clear from table that as the pH increased, the dyeing improved and was the best at pH 8, i.e. in slight alkaline conditions. The dyeing was minimum at pH 4, i.e. in acidic conditions.

#### **4.9.1.6 Optimum dye material concentration**

The table shows that the optimum dye concentration was 3g per gram of polyester. The dyeing decreased with further increase in concentration of dye material due to reduced dye extraction. Similar observations were reported by Katyayini and Jacob (1998) for mesta calyx dye on cotton.

#### **4.9.1.7 Optimum dyeing time**

The colour of polyester samples improved with increase in the duration of dyeing as depicted in Table 4.89 and the optimum dyeing time

for rubber plant dye was 75 minutes beyond which the colour decreased again.

#### 4.9.2 Optimum mordanting conditions

The mordanting method and mordant concentration were optimized on the basis of overall fastness ratings of rubber plant dye dyed polyester employing the carrier concentration, carrier treatment time, dye extraction pH, dye extraction time, dyeing pH, dye material concentration and dyeing time optimized earlier without mordanting. The fastness grades for optimization of mordanting method and mordant concentration are given in Tables 4.90 and 4.91, respectively. The optimum mordanting and mordant concentration for the four mordants have been presented in Table 4.92.

**Table 4.90 Optimization of mordanting method for rubber plant dye on polyester on the basis of fastness grades.**

Mordant	Mor- danting method	WF			LF		RF		PF				MS	Rank	
		CC	CSC	CSP	CC	W		D		Al		Ac			
						CSC	CSC	CC	CSP	CSW	CC	CSP			CSW
Alum	Pre	1	5	5	3	2/3	3	2/3	5	5	2/3	5	5	3.70	3
	Sim	2	5	5	2	3/4	4	4	5	5	2/3	5	5	4.00	2
	Post*	3	5	5	2	3	3/4	4	5	5	4	5	5	4.12	1
Chrome	Pre	1/2	5	5	2	2	3	2/3	5	5	1	5	5	3.50	3
	Sim	2	5	5	2	3/4	4	4	5	5	3/4	5	5	4.08	2
	Post*	3	5	5	2	3	3/4	3/4	5	5	4	5	5	4.16	1
Cupric sulphate	Pre	1/2	5	5	CI	2/3	2/3	2/3	5	5	2/3	5	5	3.77	3
	Sim	1/2	5	5	3	3	3	2	5	5	3	5	5	3.79	2
	Post*	3/4	5	5	2	3/4	2/3	4	5	5	4	5	5	4.12	1
Ferrous sulphate	Pre	2/3	5	5	3	2	2/3	3	5	5	3	5	5	3.83	2
	Sim	1/2	5	5	2	3	3/4	2	5	5	2	5	5	3.66	3
	Post*	3	5	5	3	2/3	3	4	5	5	4	5	5	4.12	1

\*Optimum mordanting method

### 4.9.2.1 Optimum mordanting method

The fastness ratings for different methods of mordanting have been presented in Table 4.90. The table reveals that the post-mordanting method of mordant application was the best for all the mordants.

**Table 4.91 Optimization of mordant concentration for rubber plant dye on polyester on the basis of fastness grades.**

Mordant	Mor- danting method	WF			LF		RF		PF			MS	Rank		
		CC	CSC	CSP	CC	W	D	CC	CSP	CSW	CC			CSP	CSW
Alum	5*	3/4	5	5	2	3/4	3/4	4	5	5	4	5	5	4.20	1
	10'	3	5	5	2	3	3/4	4	5	5	4	5	5	4.12	2
	15	3	5	5	1	3	2/3	4	5	5	4	5	5	3.95	3
	20	3	5	5	1	3	2	4	5	5	4	5	5	3.91	4.5
	25	3	5	5	1	3	2	4	5	5	4	5	5	3.91	4.5
Chrome	1	4	5	5	1	3	3	3/4	5	5	4/5	5	5	4.08	2.5
	2*	3	5	5	3	3	3/4	3/4	5	5	4	5	5	4.16	1
	3	3	5	5	1/2	2/3	2	4/5	5	5	4	5	5	3.95	4
	4	3	5	5	1/2	2/3	2	4/5	5	5	3/4	5	5	3.91	5
	5	3	5	5	1/2	2/3	2	5	5	5	5	5	5	4.08	2.5
Cupric sulphate	1	3	5	5	1/2	2/3	1/2	3/4	5	5	3/4	5	5	3.79	5
	2	3/4	5	5	2	3/4	2/3	4	5	5	4	5	5	4.12	2
	3	3/4	5	5	2	3	1/2	4/5	5	5	4/5	5	5	4.08	3
	4*	4	5	5	CI	2/3	1/2	4/5	5	5	4	5	5	4.22	1
	5	3	5	5	2	2/3	1/2	4	5	5	4	5	5	3.91	4
Ferrous sulphate	1	2/3	5	5	2	2/3	2	4	5	5	CI	5	5	3.90	4
	2*	3	5	5	3	2/3	3	4	5	5	4	5	5	4.12	1
	3	3	5	5	2	2/3	2/3	4	5	5	4	5	5	4.00	2
	4	3	5	5	1/2	2/3	1/2	4	5	5	4	5	5	3.87	5
	5	3/4	5	5	2	2/3	1/2	5	5	5	5	5	5	3.95	3

\*Optimum mordant concentration

### 4.9.2.2 Optimum mordant concentration

The fastness grades for different concentrations of mordants have been given in Table 4.91. The table makes it clear that the overall fastness grades decreased with the increase in concentration of alum. The fastness

grades initially increased upto certain level of mordant concentration beyond which it decreased in the case of chrome, cupric sulphate and ferrous sulphate. The optimum concentrations of alum, chrome, cupric sulphate and ferrous sulphate were 20, 2, 4 and 2 per cent, respectively (Table 4.92).

**Table 4.92 Optimum mordanting conditions for dyeing polyester with rubber plant dye.**

Mordant	Mordating method	Mordant concentration (percent)
Alum	Post	5
Chrome	Post	2
Cupric sulphate	Post	4
Ferrous sulphate	Post	2

#### 4.9.3 Effect of dyeing parameters on the fastness of rubber plant dye

##### 4.9.3.1 Effect of carrier concentration

The washing, light, rubbing and perspiration fastness grades of rubber plant dye on polyester at different dyeing time are given have been presented in Table 4.93

**Table 4.93 Effect of carrier concentration on fastness of rubber plant dye on polyester.**

Carrier concentration (percent)	WF		LF			RF			PF			
	CC	CSC	CSP	W		D	Al		Ac			
				CC	CSC	CSC	CC	CSP	CSW	CC	CSP	CSW
5	3	5	5	2	3	¾	4	5	5	¾	5	5
10	½	5	5	1	2/3	2/3	2	5	5	2	5	5
15	3	5	5	3	3	4	4	5	5	4	5	5
20	2	5	5	3	¾	4	4	5	5	4	5	5
25	2	5	5	4	¾	¾	4	5	5	4	5	5

Washing fastness grades decreased with increase in concentration of carrier except at 10 per cent where marked decrease was observed. The washing fastness was poor to good with grades in the range  $\frac{1}{2}$  to 3. No staining was observed on cotton and polyester fabrics of composite samples.

Light fastness grades increased with increase in concentration of carrier except at 10 per cent where decrease in fastness grades was noticed. Light fastness was poor to good with grades in the range 1 to 4.

Wet rubbing fastness increased with increase in carrier concentration except at 10 per cent where slight decrease was observed. Dry rubbing fastness grade decreased at 10 per cent, increased at 15 per cent and again decreased at 25 per cent concentration of carrier. The rubbing fastness was fair to very good with grades in the range  $\frac{2}{3}$  to 4.

Alkaline perspiration fastness was the same at all carrier concentration except at 10 per cent where decrease in fastness was noticed. Acidic perspiration fastness decreased at 10 per cent, increased at 15 per cent and thereafter remained constant on further increase in carrier concentration. Perspiration fastness was good to very good with grades in the range  $\frac{3}{4}$  to  $\frac{4}{5}$ . No staining was observed on adjacent wool and polyester fabrics of composite samples of perspiration fastness.

### 4.9.3.2 Effect of carrier treatment time

Table 4.94 shows the washing, light, rubbing and perspiration fastness of rubber plant dye on polyester samples treated with carrier for five different time periods.

**Table 4.94 Effect of carrier treatment time on fastness of rubber plant dye on polyester.**

Carrier treatment time (minutes)	WF			LF	RF			PF				
	CC	CSC	CSP	CC	W	D	CC	Al		Ac		
					CSC	CSC		CSP	CSW	CC	CSP	CSW
15	2/3	5	5	4	3	4	4	5	5	3/4	5	5
30	3	5	5	1	3/4	4	4/5	5	5	4/5	5	5
45	3	5	5	1	3/4	4	4	5	5	4/5	5	5
60	2	5	5	3	3/4	4	4	5	5	4	5	5
75	2	5	5	1	3	3	4/5	5	5	4/5	5	5

Washing fastness grades increased at 30 minutes, decreased at 60 minutes and remained the same on further increase in duration of carrier treatment. Washing fastness was fair to good with grades in the range 2 to 3.

Light fastness was poor at different time periods of carrier treatment except 15 and 60 minutes where marked increase in fastness was observed with 4 and 3 grades, respectively.

Wet rubbing fastness remained the same at different time periods of carrier treatment except at 15 minutes and 75 minutes where slight decrease was seen. Dry rubbing fastness remained the same at different durations of carrier treatment except at 75 minutes where decrease in grade

was noted. Rubbing fastness of dyed samples was good to very good with fastness grades in the range 3 to 4.

Alkaline fastness was the same at different time periods of carrier treatment except at 30 minutes and 75 minutes where slight increase was noticed. Acidic perspiration fastness grades were also same at different carrier treatment time except at 15 and 60 minutes where decrease in grades was noticed. Perspiration fastness was good to very good with grades in the range  $\frac{3}{4}$  to  $\frac{4}{5}$ . No staining was observed on adjacent fabrics of composite samples of perspiration fastness.

#### 4.9.3.3 Effect of dyeing pH

The fastness grades of washing, light, rubbing and perspiration of rubber plant dye on polyester at different pH values are given in Table 4.95.

**Table 4.95 Effect of dyeing pH on fastness of rubber plant dye on polyester.**

Dyeing pH	WF			LF	RF		PF					
					W	D	Al			Ac		
	CC	CSC	CSP	CC	CSC	CSC	CC	CSP	CSW	CC	CSP	CSW
4	$\frac{1}{2}$	5	5	4	$\frac{3}{4}$	$\frac{2}{3}$	3	5	5	4	5	5
5	$\frac{1}{2}$	5	5	5	$\frac{3}{4}$	$\frac{3}{4}$	4	5	5	$\frac{4}{5}$	5	5
6	2	5	5	2	$\frac{3}{4}$	$\frac{3}{4}$	4	5	5	4	5	5
7	2	5	5	3	$\frac{3}{4}$	4	4	5	5	4	5	5
8	1	5	5	2	3	4	4	5	5	$\frac{4}{5}$	5	5
9	1	5	5	2	4	$\frac{4}{5}$	$\frac{2}{3}$	5	5	$\frac{2}{3}$	5	5
10	1	5	5	2	$\frac{3}{4}$	$\frac{4}{5}$	3	5	5	$\frac{2}{3}$	5	5

Washing fastness increased at pH 6, decreased at pH 8 and remained the same on further increase in dyeing pH. Washing fastness of dyed

sample was poor to fair with grades in the range 1-2. No staining was observed on adjacent cotton and polyester fabrics of composite samples of washing fastness.

Light fastness grades increased and decreased in alternating manner with rise in the pH of dyeing upto pH 8 after which fastness remained the same on further increase in pH of dyeing. Light fastness was poor to very good with grades in the range 2 to 5.

Wet rubbing fastness grades remained the same upto pH 7 after which increase and decrease in alternating manner was noticed at higher pH value. Dry rubbing fastness showed an increase with increase in pH. Rubbing fastness of the dyed samples was fair to very good with grades in the range 2/3 to 4/5.

Alkaline perspiration fastness increased at pH 5, decreased at pH 9 after remaining the same at in between pH values, and increased again at dyeing pH 10. Acidic perspiration fastness increased and decreased in alternating manner with rise in pH of dyeing. Perspiration fastness was fair to very good with fastness grades in the range 2/3 to 4.5. No staining was observed on adjacent wool and polyester fabrics of composite samples of perspiration fastness.

#### 4.9.3.4 Effect of dye material concentration

Table 4.96 indicates the fastness grades for rubber plant dye on polyester for washing, light, rubbing and perspiration for different concentrations of dye material.

**Table 4.96 Effect of dye material concentration on fastness of rubber plant dye on polyester.**

Dye concentration (g/g)	WF			LF	RF		PF					
	CC	CSC	CSP	CC	W	D	Al			Ac		
					CSC	CSC	CC	CSP	CSW	CC	CSP	CSW
0.5	1/2	5	5	2	3	3	4/5	5	5	3/4	5	5
1	2	5	5	2	3	3	4	5	5	3/4	5	5
2	2	5	5	3	3/4	4	4	5	5	4	5	5
3	2/3	5	5	2	2/3	2/3	4	5	5	4	5	5
4	3/4	5	5	2	2/3	2/3	4/5	5	5	4/5	5	5
5	3	5	5	2	2/3	3	3/4	5	5	4	5	5
6	2	5	5	1	3	3/4	3	5	5	4	5	5

The washing fastness grades increased with increase in dye material concentration upto 4g/g concentration and decreased on further increase in dye material concentration. Washing fastness was poor to good. No staining was observed on adjacent cotton and polyester fabric of composite samples of washing fastness.

Light fastness increased at 2g/g concentration, decreased at 3g/g concentration remaining the same at rest of concentrations of dye material. Light fastness was poor to fair with grades in the range 1/2 to 3/4.

Wet rubbing fastness increased at 2g/g concentration, then decreased and remained the same upto 5g/g concentration after which increase was

noticed with increase in dye material concentration. Dry rubbing fastness increased, at 2g/g concentration, then decreased and remained the same upto 4g/g concentration after which an increase was observed with increase in dye material concentration. Rubbing fastness of dyed samples was fair to good with fastness grades in the range 2/3 to 4.

Alkaline perspiration fastness grades decreased with increase in dye material concentration at 1g/g, increased at 4g/g concentration and then decreased on further increase in concentration of dye material. Acidic perspiration fastness grades remained the same at different dye material concentration except at 0.5g/g and 1g/g concentrations of dye material where it remained the same before increasing and at 4g/g concentration where slight increase in fastness was observed. Perspiration fastness of the dyed samples was good to very good with grades in the range 3 to 4/5. No staining was observed on wool and polyester fabrics of composite samples of perspiration fastness.

#### **4.9.3.5 Effect of dyeing time**

The washing, light, rubbing, and perspiration fastness grades of rubber plant dye dyed on polyester for different time period have been presented in table 4.97.

After remaining the same upto 30 minutes, fastness grades of washing increased at 45 minutes and remained the same upto 75 minutes

after which it decreased. Washing fastness was poor to fair with fastness grades in the range 1-2. No staining was observed on adjacent cotton and polyester fabrics of composite samples of perspiration fastness.

**Table 4.97 Effect of dyeing time on fastness of rubber plant dye on polyester.**

Dyeing time (minutes)	WF			LF	RF		PF					
					W	D	Al			Ac		
	CC	CSC	CSP	CC	CSC	CSC	CC	CSP	CSW	CC	CSP	CSW
15	1	5	5	3	3	3	2	5	5	4	5	5
30	1	5	5	2	3	3	2	5	5	4	5	5
45	2	5	5	2	2/3	3	3	5	5	3	5	5
60	2	5	5	3	3/4	4	4	5	5	4	5	5
75	2	5	5	1	2/3	3	4	5	5	4	5	4/5
90	1	5	5	2	2	3	4	5	5	4	5	4/5
105	1	5	5	3	2	3	2	5	5	2/3	5	4/5

Light fastness grades increased and decreased in alternating manner upto 75 minutes after which an increase was noted in fastness grades on increase in duration of dyeing. Light fastness of dyed samples was poor to fair with grades in the range 1 to 3.

Wet rubbing fastness grades decreased with increase in duration of dyeing except at 60 minutes where an increase was noticed. Dry rubbing fastness remained the same for samples dyed for different time periods except at 60 minutes where an increase was noticed. Rubbing fastness of dyed samples was fair to very good with fastness grades in the range 2 to 4. Alkaline perspiration fastness grades increased with increase in duration of dyeing upto minutes and decreased at 105 minutes after remaining the same

for in between values of dyeing time. Acidic perspiration fastness grades were the same for samples dyed for different time periods except at 45 minutes and 105 minutes where a decrease in fastness was noticed. Perspiration fastness of dyed sample was fair to very good with fastness grades in the range 2 to 4. No staining was observed on adjacent fabrics except on wool fabrics in acidic perspiration fastness samples for longer duration of dyeing.

#### **4.9.3.6 Effect of mordanting method**

Table 4.90 depicts washing, light, rubbing and perspiration fastness grades of rubber plant dye on polyester employing different mordants and the three mordanting methods with each mordant.

##### **4.9.3.6.1 Alum**

It can be seen from the table that on mordanting with alum the washing fastness was 1, 2 and 3 with pre, simultaneous and post-mordanting methods, respectively. Light fastness grades were the same at 2 in the case of simultaneous and post-mordanting method and was maximum at 3 with pre-mordanting method. Dry and wet rubbing fastness grades were maximum with simultaneous mordanting method. Alkaline perspiration fastness was the same (4) with pre and simultaneous mordanting method and was minimum at 2/3 with post-mordanting method.

Acidic perspiration fastness grade was maximum (4) with post-mordanting method and was the same (2/3) with other two mordanting methods.

#### 4.9.3.6.2 Chrome

In the case of chrome, washing fastness  $\frac{1}{2}$ , 2 and 3 with pre, simultaneous and post-mordanting methods, respectively. Light fastness grades were the same (2) in the case of all the mordanting methods. Dry and wet rubbing and alkaline perspiration fastness grades were maximum for simultaneous mordanted samples, followed by that for post and pre-mordanted samples (in that order). Acidic perspiration fastness grade was maximum (4) for post-mordanted sample, followed by that for simultaneous ( $\frac{3}{4}$ ) and pre-mordanted ( $\frac{1}{2}$ ) samples.

#### 4.9.3.6.3 Cupric sulphate

The fastness grades of washing were the same ( $\frac{1}{2}$ ) for pre and simultaneous mordanted samples and the best ( $\frac{3}{4}$ ) in the case of post-mordanting method. Colour of dyed sample increased on exposure in the case of pre-mordanting method, followed by that of simultaneous (3) and post-mordanting (2) methods. Wet rubbing fastness grade was maximum at  $\frac{3}{4}$  in the case of post-mordanting method, followed by that for simultaneous (3) and pre-mordanting ( $\frac{2}{3}$ ) methods. Dry rubbing fastness grades were the same at  $\frac{2}{3}$  in the case of pre and post-mordanting methods and the best (3) with simultaneous mordanting method. Acidic perspiration

fastness grade was maximum at 4 in the case of post-mordanting method, followed by that for pre and simultaneous mordanting methods. In the case of fastness grades of alkaline perspiration, the best result was that of post-mordanted sample, followed by that for simultaneous and pre-mordanted samples at 3 and 2/3, respectively (in that order).

#### **4.9.3.6.4 Ferrous sulphate**

It is clear from the table that on mordanting with ferrous sulphate the washing fastness was maximum (3) in the case of post-mordanting method, followed that for pre-mordanting (2/3) and simultaneous mordanting (1/2) methods. Light fastness grades were the same (3) for pre and post-mordanting methods and in the case of simultaneous mordanting method fastness was minimum (2).

Dry and wet rubbing fastness grades were maximum in the case of simultaneous mordanting method, followed by that for post and pre-mordanting methods but dry rubbing fastness was slightly higher than wet rubbing fastness.

Alkaline and acidic perspiration fastness grades were similar being maximum (4) in the case of post-mordanting method, followed by that for pre and simultaneous mordanting methods.

No staining was seen on adjacent fabrics of the composite samples of washing and perspiration fastness in the case of all the mordants.

#### **4.9.3.7 Effect of mordant concentration**

The washing, light, rubbing and perspiration fastness grades of polyester samples dyed with rubber plant dye using five different mordant concentrations have been presented in Table 4.91.

##### **4.9.3.7.1 Alum**

In the case of alum both acidic and alkaline perspiration fastness remained unchanged on increasing the concentration of mordants. Wet rubbing, washing and light fastness decreased initially and then remained unchanged on further increase in concentration of mordants. Dry rubbing fastness decreased with increase in concentration of mordants. No staining was observed on adjacent fabrics of composite samples used in washing and perspiration fastness.

##### **4.9.3.7.2 Chrome**

Washing fastness remained the same at different concentrations after decreasing initially at 2 per cent. Light fastness increased at 2 per cent and marked decrease in fastness was observed at 3 per cent which remained the same on further increase in concentration of mordant. Wet rubbing fastness decreased and remained the same at 3 per cent concentration onwards. Dry rubbing fastness increased initially at 2 per cent concentration, then decreased at 3 per cent and remained the same on further increase in mordant concentration. Alkaline perspiration fastness increased with

increase in concentration of mordant. Acidic perspiration fastness decreased with increase in concentration of mordant upto 4 per cent and then marked increase was observed at 5 per cent concentration of mordant. No staining was observed on adjacent fabrics of composite samples of both washing and perspiration fastness.

#### **4.9.3.7.3 Cupric sulphate**

It is clear from the table that on mordanting with cupric sulphate, washing fastness increased with increase in the concentration of mordant upto 4 per cent and then decreased at 5 per cent concentration. Light fastness increased at 2 per cent concentration and remained the same on further increase in concentration. Wet rubbing fastness increased at 2 and 3 per cent concentration while dry rubbing fastness increased only at 2 per cent concentration and remained the same on other concentrations. Alkaline perspiration fastness increased upto 4 per cent and decreased at 5 per cent concentration whereas acidic perspiration fastness increased upto 3 per cent and then decreased and remained the same at 4 and 5 per cent concentrations. No staining was observed on adjacent fabrics of composite samples of the washing perspiration fastness.

#### **4.9.3.7.4 Ferrous sulphate**

In the case of ferrous sulphate washing fastness grades showed an increase with increase in the concentration of mordant. Light fastness grades remained the same at 1, 3 and 5 per cent, increased at 2 per cent and

decreased at 4 per cent concentration of mordant. Wet rubbing fastness remained unchanged with increase in concentration of mordant. Dry rubbing fastness increased at 2 per cent concentration and then decreased with increase in concentration of mordant. Alkaline and acidic perspiration fastness remained unchanged upto 4 per cent and increased at 5 per cent concentration. Also the colour of dyed sample increased at 1 per cent concentration on treatment with acidic perspiration solution. No staining was noticed on adjacent fabrics of the composite sample of both washing and perspiration fastness.

#### 4.9.3.8 Effect of mordant type

The washing, light rubbing and perspiration fastness grade for the polyester sample dyed with rubber plant dye using different mordants under optimum conditions have been compared with those of control (unmordanted) sample in the Table 4.98.

**Table 4.98 Effect of mordant type on fastness of rubber plant dye on polyester.**

Mordant	WF			LF	RF		PF					
	CC	CS	PS	CC	W	D	Al			Ac		
					CS	CS	CC	PS	WS	CC	PS	WS
Control	2	5	5	1	2/3	3	4	5	5	4	5	5
Alum	¾	5	5	2	¾	¾	4	5	5	4	5	5
Chrome	3	5	5	3	3	¾	¾	5	5	4	5	5
Cupric sulphate	4	5	5	CI	2/3	½	4/5	5	5	4	5	5
Ferrous sulphate	3	5	5	3	2/3	3	4	5	5	4	5	5

A marked increase was seen in washing fastness grades of all the mordanted samples as compared to grade of control sample.

Light fastness grades also increased with application of mordants and colour increased in the case of cupric sulphate treated sample on exposure to light.

Wet rubbing fastness of alum and chrome treated samples increased but remained the same as that of control sample when cupric sulphate and ferrous sulphate treatments were applied. Dry rubbing fastness increased for alum and chrome treated samples, decreased for cupric sulphate treated sample and remained constant for ferrous sulphate treated samples.

The acidic perspiration fastness remained the same even on application of different mordants while the alkaline perspiration fastness grades were the same for alum and ferrous sulphate treated samples, decreased for chrome treated sample and increased for cupric sulphate treated sample when compared with control sample.

No staining was noticed on adjacent fabrics of composite samples of both washing and perspiration fastness.

The mean score of fastness grades as given in Table 4.91 suggests that cupric sulphate was the best mordant followed by alum, chrome and ferrous sulphate.

#### 4.9.4 Effect of mordants on CIE LAB values

The effect of different mordants on colour strength, chroma and intensity of the polyester samples dyed with rubber plant dye under optimum conditions has been presented in Table 4.99 in terms of K/S and CIE LAB values.

**Table 4.99 Effect of mordants on CIE LAB values of rubber plant dye on polyester.**

Mordant	L*	a*	b*	C*	K/S	Colour obtained
Control	69.488	4.211	2.169	4.737	0.73	Light purplish grey
Alum	70.002	3.660	3.921	5.364	0.83	Light greyish fawn
Chrome	69.018	3.977	1.367	4.205	0.70	Ruby grey
Cupric sulphate	70.494	2.342	4.296	4.893	1.03	Reddish lilac grey
Ferrous sulphate	71.901	3.668	7.863	8.676	1.44	Light purplish grey

The higher L\* values for all the mordanted samples except for chrome treated sample indicated the lighter mordanted samples as compared to control sample. The redness of all the mordanted samples was lower but yellowness of all mordanted samples except chrome treated sample increased as shown by a\* and b\* values. Hence, all the mordanted samples except chrome treated sample were brighter as compared to control sample as depicted by C\* values.

The K/S values also increased in the case of all the mordanted samples except chrome treated sample. So the dye uptake was higher in the case of all mordanted samples except chrome treated sample when

compared with control sample. The colour obtained with rubber plant dye under optimized conditions ranged from light purplish grey to ruby grey.

A total of 67 shades of rubber plant dye were obtained in the process of optimization of different dyeing parameters.

#### **4.10 Dyeing with Turmeric dye**

##### **4.10.1 Optimum dyeing conditions without mordanting**

Details of the ranks for the optimization of each parameter, namely carrier concentration, carrier treatment time, dye extraction pH, dye extraction time, dyeing pH, dye material concentration and dyeing time and their optimum values have been presented in Table 4.100.

###### **4.10.1.1 Optimum carrier concentration**

It can be seen from the Table 4.100 that the colour of the dyed polyester improved with the increase in the concentration of carrier, the optimum carrier concentration being 10 per cent, beyond which it decreased.

###### **4.10.1.2 Optimum carrier treatment time**

It can be seen from the table that the dyeing improved with the increase in carrier treatment, the optimum time being 45 minutes, beyond which the coloured decreased again.

###### **4.10.1.3 Optimum dye extraction pH**

It can be seen from the Table 4.100 in that the extraction of dye from rhizome powder was the best at neutral pH, i.e. pH 7. The extraction of dye

**Table 4.100 Optimization of carrier concentration, carrier treatment time, dye extraction pH, dye extraction time, dyeing pH, dye material concentration and dyeing time of turmeric on polyester on the basis of mean ranks given by a panel of judges.**

Variable	Rank	Optimum value
Carrier concentration (percent)		
5	3	
10	1	
15	2	10
20	5	
25	4	
Carrier treatment time (minutes)		
15	4	
30	5	
45	1	45
60	2	
75	3	
Dye extraction pH		
4	6	
5	4	
6	2	
7	1	7
8	3	
9	5	
10	7	
Dye extraction time (minutes)		
15	4	
30	3	
45	1	
60	5	45
75	2	
90	6	
105	7	
Dyeing pH		
4	3	
5	2	
6	1	
7	4	6
8	5	
9	6	
10	7	
Dye material concentration (g/g)		
0.5	7	
1	6	
2	5	
3	4	4
4	1	
5	2	
6	3	
Dyeing time (minutes)		
15	2	
30	1	
45	3.5	
60	3.5	30
75	5.5	
90	6.5	
105	7	

decreased in either acidity, i.e. towards pH 4 or alkalinity, i.e. towards pH 10 of the extraction medium.

#### **4.10.1.4 Optimum dye extraction time**

The table indicates that a duration of 45 minutes was adjudged the best for maximum extraction of the dye. Extraction for longer period was not found helpful in increasing the dye extraction. This may be because after a particular time some substances other than dye also start to leach out from the plant material which may hinder the dye absorption. Similar observations were made by Singh et al (1993a) in the case of madder dye.

#### **4.10.1.5 Optimum dyeing pH**

It is clear from the table that the best dyeing was obtained at pH 6, i.e. in slight acidic condition. The dyeing was minimum at pH 10, i.e. in alkaline condition. Lokhande et al (1998) also reported acidic medium as optimum for dyeing nylon with turmeric dye employing high temperature high pressure method.

#### **4.10.1.6 Optimum dye material concentration**

The table shows that the optimum dye material concentration was 4 g per gram of polyester. The dyeing decreased with further increase in concentration of dye material due to reduced dye extraction.

#### **4.10.1.7 Optimum dyeing time**

The colour of the dyed polyester improved with increase in dyeing time upto 30 minutes as depicted in Table 4.100. On further increase in

duration of dyeing the colour of the dyed samples decreased. Thus, the optimum dyeing time was 30 minutes.

#### 4.10.2 Optimum mordanting conditions

The mordanting method and mordant concentration were optimized on the basis of overall fastness ratings of turmeric dye dyed polyester employing the carrier concentration, carrier treatment time, dye extraction pH, dye extraction time, dyeing pH, dye material concentration and dyeing time optimized earlier without mordanting. The fastness grades for optimization of mordanting method and mordant concentration are given in Tables 4.101 and 4.102, respectively. The optimum mordanting and mordant concentration for the four mordants have been presented in Table 4.103.

**Table 4.101 Optimization of mordanting method for turmeric dye on polyester on the basis of fastness grades.**

Mordant	Mor- danting method	WF			LF			RF			PF			MS	Rank
		CC	CSC	CSP	CC	CSC	CSC	CC	CSP	CSW	CC	CSP	CSW		
Alum	Pre	CC	5	5	1	3	2/3	4	4/5	3/4	3/4	5	4/5	3.77	2
	Sim	CC	5	5	1	2/3	3/4	2	5	3/4	3/4	5	3	3.5	3
	Post*	CC	5	5	1	3	2/3	4/5	4/5	4/5	4/5	4/5	4/5	3.95	1
Chrome	Pre	CC	5	5	1	2/3	3	3/4	4/5	3	4/5	5	3/4	3.6	2
	Sim	CC	5	5	1	2/3	4	3	4	2/3	3	4/5	2	3.3	3
	Post*	4	5	5	1	3/4	3/4	4/5	5	4/5	4/5	5	4/5	4.16	1
Cupric sulphate	Pre	1	5	5	1	1/2	2	3	5	3/4	2	4/5	3	3.04	2
	Sim	1	5	5	1	2/3	4	1/2	5	2	1	4/5	1/2	2.83	3
	Post*	4	4/5	5	1	2/3	2	4	4/5	4	4	4/5	3	3.58	1
Ferrous sulphate	Pre	CC	5	5	1	2	2	3	4/5	3	2	4/5	2	3.09	3
	Sim	CC	5	5	1	3/4	4	1	5	4/5	1	5	3/4	3.50	2
	Post*	CC	5	4/5	1	2/3	2	4/5	5	4/5	3	4	4	3.63	1

\*Optimum mordanting method

#### 4.10.2.1 Optimum mordanting method

The fastness ratings for different methods of mordanting have been presented in Table 4.101. The table reveals that the post-mordanting method of mordant application was the best (highest mean score) method for all the mordants.

**Table 4.102 Optimization of mordant concentration for turmeric dye on polyester on the basis of fastness grades.**

Mordant	Mordant concentration	WF			LF	RF		PF					MS	Rank		
		CC	CSC	CSP	CC	W	D	CC	Al	CSP	CSW	CC			Ac	CSP
Alum	5	CC	5	5	1	2	2	4/5	4/5	4/5	4/5	4/5	4/5	4/5	3.81	5
	10*	CC	5	5	1	3	2/3	4/5	4/5	4/5	4/5	4/5	4/5	4/5	3.95	1
	15	CC	5	5	1	2/3	2/3	4/5	4/5	4/5	4/5	4/5	4/5	4/5	3.90	3
	20	CC	5	5	1	2/3	2	4/5	4/5	4/5	4/5	4/5	4/5	4/5	3.90	3
	25	CC	5	5	1	2/3	2	4/5	4/5	4/5	4/5	4/5	4/5	4/5	3.90	3
Chrome	1	3/4	5	5	1	3/4	3/4	4	5	4/5	4/5	5	4/5	4.08	4	
	2*	4	5	5	1	3/4	3/4	4/5	5	4/5	4/5	5	4/5	4.16	1	
	3	4	5	5	1	3	2/3	4/5	5	4/5	4/5	5	4/5	4.12	2	
	4	3/4	5	5	1	3	2/3	4/5	5	4/5	4/5	5	4/5	4.08	4	
	5	3/4	5	5	1	3	2/3	4/5	5	4/5	4/5	5	4/5	4.08	4	
Cupric sulphate	1*	4/5	4/5	5	1	2/3	2	4/5	4/5	4/5	4/5	4/5	3	3.87	1	
	2	4	4/5	5	1	2/3	2	4	4/5	4	4	4/5	4	3.58	5	
	3	4/5	4/5	5	1	2/3	2	4/5	4/5	4/5	4/5	4/5	4/5	3.83	3	
	4	4	4/5	5	1	2/3	2	4/5	4/5	4/5	4/5	4/5	4/5	3.83	3	
	5	4	4/5	5	1	2/3	2	4/5	4/5	4/5	4/5	4/5	4/5	3.83	3	
Ferrous sulphate	1	CC	5	5	1	2/3	2	4/5	5	4/5	4	5	4	3.90	2.5	
	2	CC	4/5	5	1	2/3	2	4/5	5	4/5	3	4	4/5	3.63	5	
	3*	CC	5	5	1	2/3	2	4/5	5	4/5	4/5	5	4/5	3.95	1	
	4	CC	5	5	1	2	2	4/5	5	4/5	4/5	5	4/5	3.90	2.5	
	5	CC	5	5	1	2	2	4	5	4/5	4	5	4/5	3.81	4	

\*Optimum mordant concentration

#### 4.10.2.2 Optimum mordant concentration

The fastness grades for different concentrations of mordants have been given in Table 4.102. The table makes it clear that the overall fastness

grades decreased with increase in concentration of cupric sulphate. In the case of alum, chrome and ferrous sulphate, the overall fastness grades increased with increase in mordant concentration upto certain level. Beyond that concentration fastness ratings decreased. The optimum concentration of alum, chrome, cupric sulphate and ferrous sulphate were 10, 2, 1 and 3 per cent, respectively (Table 4.103).

**Table 4.103 Optimum mordanting conditions for dyeing polyester with turmeric dye.**

Mordant	Mordanting method	Mordant concentration (percent)
Alum	Post	10
Chrome	Post	2
Cupric sulphate	Post	1
Ferrous sulphate	Post	3

#### 4.10.3. Effect of dyeing parameters on the fastness of turmeric dye

##### 4.10.3.1 Effect of carrier concentration

The washing fastness, light fastness, rubbing fastness and perspiration fastness grades of Turmeric dye on polyester at different carrier concentrations have been presented in Table 4.104.

**Table 4.104 Effect of carrier concentration on fastness of turmeric dye on polyester.**

Carrier concentration (percent)	WF			LF	RF			PF				
	CC	CSC	CSP		CC	W	D	Al			Ac	
				CSC		CSC	CC	CSP	CSW	CC	CSP	CSW
5	1	5	5	1	2/3	2/3	3	4	3	3	4	3
10	1	5	5	1	3/4	4/5	4	4	3	4	4	3
15	1	5	5	1	3/4	4	3/4	4	3	4	4	3
20	1	5	5	1	4	4	3	4	3	4	4	3
25	1	5	5	1	3	3	4	4	3	3	4	3

Washing and light fastness grades remained the same (1) at all the concentrations of the carrier. No staining was observed on adjacent cotton and polyester fabrics. Wet rubbing fastness grades increased with increase in carrier concentration upto 20 per cent and decreased on 25 per cent. Dry rubbing fastness grades increased at 10 per cent and decreased on further increase in concentration of carrier. Washing fastness of the dyed samples was fair to very good. Alkaline perspiration fastness grades increased at 10 per cent, then decreased upto 20 per cent concentration and increased again at 25 per cent concentration of carrier. Acidic perspiration fastness grades were the same (4) except at 5 and 25 per cent concentrations where decrease in grades was observed. The perspiration fastness was good to very good with grades in the range of 2/3 to 4/5. Staining was observed on wool and polyester fabrics of composite samples of perspiration fastness.

**Table 4.105 Effect of carrier treatment time on fastness of turmeric dye on polyester.**

Carrier treatment time (minutes)	WF			LF	RF			PF				
	CC	CSC	CSP	CC	W	D	Al			Ac		
					CSC	CSC	CC	CSP	CSW	CC	CSP	CSW
15	2/3	5	5	1	4/5	4	3/4	4/5	3/4	4/5	4/5	3
30	2/3	5	5	1	4/5	4	4	4/5	3/4	3/4	4/5	3/4
45	2	5	5	1	3/4	3	4	4	3	4	4/5	3
60	1	5	5	1	3/4	4/5	3/4	4	3	4	4	3
75	1	5	5	1	4/5	4/5	3	4	3	4	4	3

#### 4.10.3.2. Effect of carrier treatment time

Table 4.105 shows the washing, light, rubbing and perspiration fastness of turmeric dye on polyester samples treated with carrier for five different time periods.

Washing fastness decreased with increase in duration of carrier treatment. The washing fastness of dyed samples was poor to fair with grades in range 1-2/3. No staining was observed on adjacent cotton and polyester fabrics of composite samples of washing fastness.

Light fastness of dyed samples was poor with fastness grade 1 which remained the same at different durations of carrier treatment.

Wet and dry rubbing fastness grades decreased initially upto 45 minutes and then decreased on further increase in time periods of carrier treatment. Rubbing fastness was good to very good having grades in the range 3 to 4/5.

Alkaline perspiration fastness increased at 30 minutes and then decreased with increase in duration of carrier treatment after remaining the same at 45 minutes. Acidic perspiration fastness grades decreased initially and remained the same on further increase in duration of carrier treatment after increasing at 45 minutes. The perspiration fastness of the dyed samples was good to very good with grades in range 3 to 4/5. Staining was

noticed clearly on wool and polyester fabrics of composite samples of perspiration fastness.

#### 4.10.3.3 Effect of dyeing pH

The fastness grades of washing, light, rubbing and perspiration of turmeric dye on polyester at different pH value have been presented in Table 4.106.

**Table 4.106 Effect of dyeing pH on fastness of turmeric dye on polyester.**

Dyeing pH	WF			LF	RF		PF					
	CC	CSC	CSP	CC	W	D	AI			Ac		
					CSC	CSC	CC	CSP	CSW	CC	CSP	CSW
4	1	5	5	1	2	4/5	3	4	3	3	4	3
5	1	5	5	1	2	4/5	3	4	3	3	4	3
6	1	5	5	1	2/3	4/5	3	4	3	4	4	3
7	1	5	5	1	¾	4/5	¾	4	3	4	4	3
8	1	5	5	1	4	4/5	4	4/5	4	4/5	5	4/5
9	1/2	5	5	1	4/5	4/5	4	5	4/5	4	5	4/5
10	1	5	5	1	4/5	4/5	4	5	5	4	5	5

The fastness grades of washing remained the same at all dyeing pH except at pH 9 where slight increase was noticed. The washing fastness of dyed samples was poor with grades 1 and ½. No staining was noticed on adjacent cotton and polyester fabrics of composite samples of washing fastness.

Light fastness was poor for dyed samples which remained the same at different dyeing pH.

Wet rubbing fastness grades increased with increase in pH of dyeing. The wet rubbing fastness of dyed samples was fair to very good with

grades in the range 2 to 4/5. The dry rubbing fastness of the dyed samples was very good at 4/5 and remained the same at different pH values.

Alkaline fastness grades showed an increase with increase in the pH of dyeing remaining the same at pH 4 and 6 and again at pH 8 and 10. Acidic perspiration fastness grades also increased with increase in the pH of dyeing upto pH 8 and then decreased and remained the same at pH 9 and 10. The perspiration fastness of dyed samples was good to very good. Both wool and polyester fabrics of composite samples showed staining for both acidic and alkaline perspiration fastness except polyester fabrics of composite samples at higher pH.

**Table 4.107 Effect of dye material concentration on fastness of turmeric dye on polyester.**

Dye concentration (g/g)	WF			LF	RF			PF				
	W		D	CC	Al		Ac					
	CC	CSC	CSC		CC	CSP	CSW	CC	CSP	CSW		
0.5	3	5	5	1	4/5	4/5	5	5	5	5	5	5
1	CC	5	5	1	4/5	4/5	4/5	5	4/5	4/5	5	4/5
2	1	5	5	1	3/4	4/5	3/4	4	3	4	4	3
3	1	5	5	1	3	4/5	3/4	4	3	4	4	3
4	1	5	5	1	3	4	2	4	2	3	4	2/3
5	1	5	5	1	3	3/4	2	4	2	2	4	2
6	1	5	5	1	2/3	3/4	2	4	2	2	4	2

#### 4.10.3.4. Effect of dye material concentration

Table 4.107 indicates the fastness grades of turmeric dye on polyester for washing, light, rubbing and perspiration for different concentrations of dye material.

On washing change in colour was observed at 1g/g concentration and washing fastness remained the same at 2 to 6g/g dye material concentration. Washing fastness of dyed samples was poor (1) except at 0.5g/g dye material concentration where it was good (3). No staining was noticed on adjacent cotton and polyester fabrics of composite samples.

Both wet and dry rubbing fastness decreased with increase in concentration of dye material and was fair to very good with grade in the range 2/3 to 4/5.

Alkaline and acidic perspiration fastness grades decreased with increase in the concentration of dye material after and remained the same at higher concentrations. The perspiration fastness was fair to excellent with grades in the range 2 to 5. Staining was noticed on wool and polyester fabrics of composite samples of both acidic and alkaline perspiration fastness except both adjacent fabrics at 0.5g/g and only polyester fabrics at 1g/g dye material concentration where no staining was observed.

#### **4.10.3.5 Effect of dyeing time**

The washing, light, rubbing and perspiration fastness grades of turmeric dye dyed on polyester for different time periods have been presented in Table 4.108.

The washing and light fastness of the dyed samples was poor with fastness grade 1 which remained the same for different dyeing time

periods. No staining was noticed on adjacent cotton and polyester fabrics of composite samples of washing fastness.

**Table 4.108 Effect of dyeing time on fastness of turmeric dye on polyester.**

Dyeing time (minutes)	WF			LF	RF			PF				
	CC	CSC	CSP	CC	W	D	Al			Ac		
					CSC	CSC	CC	CSP	CSW	CC	CSP	CSW
15	1	5	5	1	$\frac{3}{4}$	4/5	3	4	3	2	4	3
30	1	5	5	1	$\frac{3}{4}$	4/5	3	4	3	3	4	3
45	1	5	5	1	$\frac{3}{4}$	4/5	3	4	3	$\frac{3}{4}$	4	3
60	1	5	5	1	$\frac{3}{4}$	4/5	$\frac{3}{4}$	4	3	4	4	3
75	1	5	5	1	4	4/5	3	4	4	3	5	4/5
90	1	5	5	1	4	4/5	4	4/5	4/5	4	5	5
105	1	5	5	1	$\frac{3}{4}$	4/5	4	4/5	4/5	4	5	5

Dry rubbing fastness was very good at 4/5 which also remained the same for different dyeing time period. Wet rubbing fastness was good to very good with grades  $\frac{3}{4}$ -4 and grades remained the same for different dyeing time periods except at 75 and 90 minutes where slight increase was observed.

Alkaline perspiration fastness increased with increase in duration of dyeing after remaining the same upto 45 minutes. Acidic perspiration fastness grades increased with increase in duration of dyeing upto 60 minutes. It decreased at 75 minutes, increased at 90 minutes and then remained the same at 105 minutes. Perspiration fastness was fair to very good with grades ranging from 2 to 4. Staining was noticed on both wool and polyester fabrics of composite samples of perspiration fastness except

wool and polyester fabrics in the case of acidic perspiration fastness at 90 and 105 minutes.

#### **4.10.3.6 Effect of mordanting method**

Table 4.101 depicts the washing, light, rubbing and perspiration fastness grades of turmeric dye on polyester employing different mordants and the three mordanting methods with each mordant.

##### **4.10.3.6.1 Alum**

It is clear from the table that on mordanting with alum the colour of the dyes samples changes on washing in the case of all the three mordanting methods. No staining was observed on cotton and polyester fabrics of composite samples of washing fastness. Light fastness (1) also remained the same in all the three mordanting methods. Wet rubbing fastness grades were the same (3) in the case of pre and post-mordanting methods and was minimum (2/3) with simultaneous mordanting method whereas dry rubbing fastness grade was maximum with simultaneous mordanting method and remained the same (3) with pre and post-mordanting methods. Alkaline perspiration fastness grade was maximum (4/5) in the case of post-mordanted sample, followed by that for pre (4) and simultaneous (2) mordanted samples. The staining was observed on polyester (slight) and wool (more) fabrics of composite samples of perspiration fastness.

#### 4.10.3.6.2 Chrome

The colour of dyed samples changed on washing in the case of pre and simultaneous mordanting method and fastness grade of 4 was obtained in the case of post-mordanting method. No staining was observed on adjacent fabrics of composite samples. Light fastness grades remained the same treated with different mordanting methods. Best wet drying fastness grade of  $3/4$  was obtained in case of post-mordanting method and the same fastness grades ( $2/3$ ) were obtained in the case of pre and simultaneous mordanting methods. Dry rubbing fastness grade was maximum (4) in the case of simultaneous mordanting method, followed by that for post ( $3/4$ ) and pre-mordanting (3) methods. Alkaline fastness grade was maximum at  $4/5$  in the case of post-mordanting method, followed by that for pre ( $3/4$ ) and simultaneous (3) mordanting methods. Acidic perspiration fastness was the same at  $4/5$  in the case of pre and post-mordanting methods and was minimum (3) with simultaneous mordanting method. Slight staining was observed only on wool fabrics of composite sample in the case of post-mordanting method. Slight staining on polyester and heavy staining on wool fabric of composite samples of perspiration fastness was noticed for both pre and simultaneous mordanting methods.

#### **4.10.3.6.3 Cupric sulphate**

In the case of cupric sulphate, washing fastness was the same (1) with pre and simultaneous mordanting methods and was the best with post-mordanting method. Slight staining was noticed on cotton fabric in post-mordanted samples. Light fastness grades remained unchanged at 1 in the case of all the mordanting methods. Wet rubbing fastness remained the same (2/3) in post and simultaneous mordanting methods and was minimum (1/2) with pre-mordanting method whereas dry rubbing fastness grades were the same (2) in pre and post-mordanting methods and the best (4) with simultaneous mordanting method. Alkaline and acidic perspiration fastness grades were maximum (4) in the case of post-mordanting method, followed by that for pre and simultaneous mordanting methods. Slight staining on polyester and heavy staining was noticed in the case of all the mordanting methods and in both alkaline and acidic perspiration fastness.

#### **4.10.3.6.4 Ferrous sulphate**

It is clear from the table that on mordanting with ferrous sulphate the colour of the dyed samples changed on washing in the case of all three mordanting methods. Slight staining was observed on polyester fabric in case of post-mordanted sample. Light fastness grades were the same (1) in the case of all the mordanting methods. Wet rubbing fastness grade was maximum (3/4) in the case of simultaneous mordanting method, followed by

that for post (2/3) and pre-mordanting (2) methods. Dry rubbing fastness grades were the same (2) in the case of pre and post-mordanting methods and was maximum (4) in the case of simultaneous mordanting method. Alkaline and acidic perspiration fastness grades were maximum in the case of post-mordanting method, followed by that for pre and simultaneous mordanting methods. Staining was observed on polyester and wool fabrics in the case of all mordanting methods except polyester fabric of simultaneous mordanted samples in both acidic and alkaline perspiration fastness and polyester fabric of post-mordanted sample in alkaline perspiration fastness.

#### **4.10.3.7 Effect of mordant concentration**

The washing, light, rubbing and perspiration fastness grades of polyester samples dyed with turmeric dye using five different mordant concentrations have been presented in Table 4.102.

##### **4.10.3.7.1 Alum**

On increasing the concentration, the washing (CC), light (1) and alkaline and acidic perspiration (4/5) fastness remained unchanged. Wet rubbing fastness was maximum (3) at 10 per cent and remained unchanged after decreasing slightly on further increase in concentration of mordant. Dry rubbing fastness increased at 10 and 15 per cent concentrations and remained the same (2) at 5, 20 and 25 per cent concentrations. No staining

was noticed on cotton and polyester fabrics in washing fastness test while slight staining was observed on both wool and polyester fabrics of composite samples of acidic and alkaline perspiration fastness.

#### **4.10.3.7.2 Chrome**

Light and acidic perspiration fastness grades remained the same at all the concentrations of the mordant. Alkaline perspiration fastness increased slightly at 2 per cent and remained the same on further increase in the concentration of mordant. Wet and dry rubbing fastness grades remained the same upto 2 per cent, decreased at 3 per cent and then remained constant on further increase in the concentration of mordant. Washing fastness grades increased slightly at 2 and 3 per cent concentrations and remained same on rest of concentrations of the mordant. No staining was observed on adjacent fabrics of composite samples of washing fastness but slight staining was observed on adjacent wool fabrics of composite samples of perspiration fastness.

#### **4.10.3.7.3 Cupric sulphate**

It is clear from the table that on mordanting with cupric sulphate light and wet and dry rubbing fastness remained the same at different concentrations of the mordant. Acidic and alkaline fastness reduced slightly at 2 per cent concentration and remained the same on rest of

concentration of the mordant. Slight staining was observed on adjacent wool and polyester fabrics of composite samples of perspiration fastness.

The washing fastness grades increased slightly at 1 and 3 per cent concentrations and remained the same on rest of concentrations of mordants. Slight staining was noticed only on cotton fabrics of composite samples of washing fastness.

#### **4.10.3.7.4 Ferrous sulphate**

In the case of ferrous sulphate, washing (CC), light (1) and dry rubbing (2) fastness grades remained unchanged with different concentrations of mordant. Slight staining was observed only on cotton fabric at 2 per cent concentration in the case of washing fastness.

Wet rubbing fastness remained unchanged 2/3 upto 3 per cent concentration but decreased slightly and remained the same (2) at 4 and 5 per cent concentrations of mordant. Alkaline perspiration fastness grades remained the same upto 4 per cent concentration and decreased slightly at 5 per cent concentration of mordant whereas acidic perspiration fastness decreased at 2 per cent concentration, increased at 3 and 4 per cent concentration and remained the same (4) at 1 and 5 per cent concentrations. Staining was noticed on wool fabric in the case of both acidic and alkaline perspiration fastness and on polyester fabric slight staining was also observed at 2 per cent concentration in acidic perspiration fastness.

#### 4.10.3.8 Effect of the type of mordant

The washing, light rubbing and perspiration fastness grade for the polyester sample dyed with turmeric dye using different mordants under optimum conditions have been compared with those of control (unmordanted) sample in the Table 4.109.

**Table 4.109 Effect of mordant type on fastness of turmeric dye on polyester.**

Mordant	WF			LF	RF		PF					
	CC	CSC	CSP	CC	W	D	Al			Ac		
					CSC	CSC	CC	CSP	CSW	CC	CSP	CSW
Control	1	5	5	1	$\frac{3}{4}$	4/5	3	4	3	3	4	3
Alum	CC	5	5	1	3	2/3	4/5	4/5	4/5	4/5	4/5	4/5
Chrome	4	5	5	1	$\frac{3}{4}$	$\frac{3}{4}$	4/5	5	4/5	4/5	5	4/5
Cupric sulphate	4/5	4/5	5	1	2/3	2	4/5	4/5	4/5	4/5	4/5	4/5
Ferrous sulphate	CC	5	5	1	2/3	2	4/5	5	4/5	4/5	5	4/5

The washing grades increased on application of chrome and cupric sulphate while the colour of dyed samples changed on washing in the case of alum and ferrous sulphate treated samples. In case of washing fastness, staining was observed on cotton fabric only in the case of cupric sulphate treated sample.

The light fastness grades remained unchanged even on application of mordants. Both acidic and alkaline perspiration fastness grades improved while wet and dry rubbing fastness grades reduced on application of mordants when compared with control sample.

In the case of perspiration fastness, slight staining was noticed on all wool and polyester fabrics of composite sample except on polyester fabrics of composite samples in the case of chrome and ferrous sulphate treated samples.

It can be seen from mean scores of fastness grades as given in Table 4.102 the best mordant was chrome followed by alum, ferrous sulphate and cupric sulphate.

#### 4.10.4 Effect of mordants on CIE LAB values

The effect of different mordants on colour strength, chroma and intensity of the polyester samples dyed with turmeric dye under optimum conditions has been presented in Table 4.110 in terms of K/S and CIE LAB values.

**Table 4.110 Effect of mordants on CIE LAB values of turmeric dye on polyester.**

Mordant	L*	a*	b*	C*	K/S	Colour obtained
Control	70.146	-1.328	65.945	65.958	14.64	Dark mimosa (yellow)
Alum	70.051	2.444	69.877	69.920	16.38	Olive yellow
Chrome	61.224	1.954	53.143	53.179	7.96	Golden brown
Cupric sulphate	51.358	17.642	45.818	49.097	13.77	Mimosa (Yellow)
Ferrous sulphate	61.164	2.946	53.858	53.939	14.12	Vivid yellow/Chrome yellow/Primary yellow

The lower L\* values of the all the mordanted samples as compared to control sample indicated that mordanted samples were darker than the

control sample. The negative value of  $a^*$  indicated greenness in the control sample and this greenness decreased in mordanted samples as depicted by positive and higher values of  $a^*$ . All the mordanted samples except one treated with alum exhibited less yellowness and were duller as compared to control sample as depicted by  $b^*$  and  $C^*$  values.

The increase in K/S value of alum treated sample indicated increase in dye uptake in this sample. In the case of samples mordanted with other mordants dye uptake was less as shown by lower K/S values than that of control sample value. The colour obtained with turmeric dye under optimized conditions ranged from vivid yellow to golden brown.

A total of 67 shades of turmeric dye were obtained in the process of optimization of different dyeing parameters.

#### **4.11 Total number of shades of natural dyes developed**

In the process of optimization of different dyeing parameters, namely carrier concentration, carrier treatment time, dye extraction pH, dye extraction time, dyeing pH, dye material concentration, dyeing time, mordanting method and mordant concentration, a total of 67 shades were developed for each dye. Thus, for 10 dyes 670 shades of natural dyes were developed.



a



b



c



d



e

Rubber plant



a



b



c



d



e

Turmeric

**SS 5 : Polyester samples dyed with rubber plant and turmeric dyes under optimum dyeing conditions using different mordants (a - unmordanted sample, b - sample treated with ferrous sulphate, c - sample treated with cupric sulphate, d - sample treated with chrome, e - sample treated with alum).**

## CHAPTER V

### SUMMARY

The present study was conducted to optimize the processes of dyeing polyester with selected natural dyes applying synthetic mordants. The effect of synthetic mordants on various parameters of dyeing was investigated and the colour fastness properties of the dyed polyester were evaluated.

For this study, ten natural dyes were selected, namely ashoka leaves (*Polyalthia longifolia*), banyan leaves (*Ficus bengalensis*), beefwood bark (*Casuarina equisetifolia*), flame of forest bark (*Butea monosperma*), golden drop root (*Onosma echioides*), Indian blackberry bark (*Syzygium cumini*), onion skin (*Allium cepa*), pine bark (*Pinus roxburguii*), rubber plant leaves (*Ficus elastica*) and turmeric rhizome (*Curcuma domestica*). Four synthetic mordants, namely alum (Potassium aluminium sulphate), chrome (Potassium dichromate), cupric sulphate and ferrous sulphate, carrier dyeing method and benzoic acid as a carrier were used in the study.

Conditions of dyeing polyester with each of the ten dyes in combination with each of the four synthetic mordants were optimized, by

optimizing nine parameters of dyeing, namely carrier concentration, carrier treatment time, dye extraction pH, dye extraction time, dyeing pH, dye material concentration, dyeing time, mordanting method and mordant concentration. First seven parameters were optimized on the basis of the ranks given by a panel of judges considering the chroma and intensity of the dyed samples. Last two variables were optimized on the basis of mean scores for washing, light, rubbing and perspiration fastness grades.

Effect of seven dyeing parameters, namely carrier concentration, carrier treatment time, dyeing pH, dye material concentration, dyeing time, mordanting method and mordant concentration on washing, light, rubbing and perspiration fastness properties of the dye was evaluated by employing tests prescribed by BIS, India. Effect of different mordants on the hue, value and intensity of the colour of the dye was investigated by calculating K/S and CIELAB values.

The important findings of the study are discussed below:

### **5.1 Optimum dyeing conditions without mordanting**

Optimum carrier concentration for different dyes ranged from 10 to 25 per cent whereas optimum carrier treatment time ranged from 45 to 75 minutes.

The best extraction of the dyes was achieved in the pH range of 5 to 10, it being pH 5 for onion and pine dyes, pH 6 for beefwood and rubber plant dyes, pH 7 for banyan, golden drop and turmeric dyes, pH 8 for Indian blackberry dye and pH 10 for ashoka and flame of forest dyes.

The optimum extraction time for different dyes ranged from 30 to 90 minutes. The optimum extraction time was 30 minutes for banyan dye, 45 minutes for ashoka dye, beefwood, flame of forest, Indian blackberry and turmeric dyes, 60 minutes for rubber plant dye and in 90 minutes of golden drop, onion and pine dyes. Beyond the optimum extraction time the colour results were either constant or poorer. It may be because after a particular time some other substances other than the dye start to leach out of the plant material which may hinder in the dyeing process.

The optimum dyeing pH ranged from 4 to 10. Dyeing with banyan, flame of forest, golden drop, onion and turmeric dyes was the best in acidic medium, with ashoka, beefwood and Indian blackberry dyes at neutral pH and with rubber dye was the best in alkaline medium. The best dyeing of a dye at a particular pH was due to the fact that the dye was not soluble at that pH, so it had a tendency to leave aqueous phase and get adsorbed on fibre surface.

The optimum dye material concentration for different dyes ranged from 0.5 to 4g/g of polyester. Employing larger amounts of the dye

material in dyebath resulted in reduced extraction because the dyebath became saturated with the dye material even before complete extraction of dye due to lowering of liquor to dye material ratio.

Optimum dyeing time for the dyes ranged from 45 minutes to 105 minutes, whereas it was between 90 to 105 minutes in the case of most (seven) of the dyes because diffusion of dye into polyester fibre was slow and hence, better colour was obtained if dyeing was carried out for longer periods which helped better diffusion of dye into fibres. The optimum conditions of dyeing for all the dyes without mordanting are summarized in Table 5.1.

## **5.2 Optimum mordanting conditions**

Best mordanting method was found to be post-mordanting in 70 percent cases, followed by pre-mordanting and simultaneous mordanting methods in 15 percent cases each.

Optimum mordant concentration was found to range between 1 to 5 percent (o.w.f.) in the case of chrome, cupric sulphate and ferrous sulphate and 5 to 20 percent (o.w.f.) in the case of alum. The optimum mordanting conditions for all the mordants in combination with different dyes are summarized in Table 5.2.

**Table 5.1 : Optimum dyeing conditions for dyeing polyester with the ashoka, banyan, beefwood, flame of forest, golden drop, Indian blackberry, onion, pine, rubber plant and turmeric dyes without mordanting.**

Dye	Carrier		Dye extraction		Dyeing pH	Dye material concentration (g/g)	Dyeing time (minutes)
	concentration (per cent)	treatment time (minutes)	pH	time (minutes)			
Ashoka	20	60	10	45	7	1	105
Banyan	25	75	7	30	6	1	90
Beefwood	10	60	6	45	7	2	60
Flame of forest	15	60	10	45	4	3	90
Golden drop	10	60	7	90	6	3	90
Indian blackberry	10	60	8	45	7	2	105
Onion	10	60	5	90	4	3	105
Pine	10	45	5	90	7	0.5	105
Rubber plant	20	75	6	<b>60</b>	8	3	75
Turmeric	10	45	7	45	6	4	30

**Table 5.2 : Optimum mordanting conditions for dyeing polyester with ashoka, banyan, beefwood, flame of forest, golden drop, Indian blackberry, onion, pine, rubber plant and turmeric dyes.**

Dye	Mordanting Method						Mordant concentration (per cent)			
	Alum	Chrome	Cupric sulphate	Ferrous sulphate	Alum	Chrome	Cupric sulphate	Chrome	Cupric sulphate	Ferrous sulphate
Ashoka	Post	Sim	Sim	Sim	5	5	3	5	3	2
Banyan	Post	Pre	Post	Post	15	3	2	3	2	3
Beefwood	Post	Post	Post	Pre	10	2	2	2	2	5
Flame of forest	Pre	Pre	Post	Pre	20	5	1	5	1	1
Golden drop	Post	Post	Post	Sim	10	2	2	2	2	4
Indian blackberry	Post	Sim	Post	Post	5	5	3	5	3	1
Onion	Post	Post	Post	Pre	5	1	1	1	1	4
Pine	Post	Post	Post	Sim	20	2	2	2	2	3
Rubber plant	Post	Post	Post	Post	5	2	4	2	4	2
Turmeric	Post	Post	Post	Post	10	2	1	2	1	3

### **5.3 Effect of dyeing parameters on fastness properties of the dyes**

#### **5.3.1 Effect of carrier concentration**

Perspiration fastness was generally found to have inverse relationship with carrier concentration whereas the washing, light and rubbing fastness were found to be either unaffected or decreased with increase in concentration of carrier.

#### **5.3.2 Effect of carrier treatment time**

Washing, rubbing and perspiration fastness were found to decrease with increase in time of carrier treatment, whereas the light fastness was found to be unaffected in about 60 per cent of the cases.

#### **5.3.3 Effect of dyeing pH**

Washing and perspiration fastness of the most of the dyes showed inverse relationship with dyeing pH whereas the rubbing fastness generally increased with increase in pH. The light fastness of different dyes did not show any specific trend.

#### **5.3.4 Effect of dye material concentration**

The washing, light, rubbing and perspiration fastness grades followed different trends with different dyes.

### **5.3.5 Effect of dyeing time**

The washing fastness was found to decrease with increase in dyeing time whereas the light and rubbing fastness grades did not show any specific trend. The perspiration fastness grades increased with increase in dyeing time.

### **5.3.6 Effect of mordanting method**

Different trends in washing, light, rubbing and perspiration fastness grades were observed in different dyes. However, the overall fastness was maximum with post-mordanting method in the majority of the dyes.

### **5.3.7 Effect of mordant concentration**

Different trends in washing, light, rubbing and perspiration fastness grades increased with increase in concentration of mordants to some extent, i.e. their mean scores were the best at 2 or 3 per cent mordant concentration in the case of chrome, cupric sulphate and ferrous sulphate and at 10 or 15 per cent mordant concentration in the case of alum, after which an inverse relation was found in most of the dyes.

### **5.3.7 Effect of the type of mordant**

Different mordants were found to be suitable for different dyes. In the case of ashoka, beefwood, golden drop, onion, pine and turmeric dyes the best fastness results were obtained with chrome while alum was found

to be the best for Indian blackberry and rubber plant dyes. Cupric sulphate was found to be the best for banyan dye while in the case of flame of forest dye all the mordants produced similar fastness results.

Mordants were found to have different effects on the different types of fastness in different dyes. In the case of ashoka, Indian blackberry, pine and turmeric dyes marked increase was observed in the washing fastness grades only. In the case of beefwood, flame of forest and onion dyes, no increase or slight increase in different fastness grades was noticed whereas in the case of banyan and golden drop dyes an increase was seen in all the fastness grades.

On application of alum, washing, light, rubbing and perspiration fastness grades increased in most of the dyes except few dyes where different fastness grades remained the same and washing and rubbing fastness grades decreased.

On application of chrome also washing, light, rubbing and perspiration fastness grades increased or the remained some in all the dyes except rubbing fastness where decrease was noticed in three dyes and perspiration fastness where decrease was observed in one dye.

Washing, light, rubbing and perspiration fastness grades increased or remained the same in all the dyes on application of cupric sulphate as

mordant except washing and perspiration fastness grades in one dye each and rubbing fastness grades in three dyes which showed a decrease.

Similarly, on application of ferrous sulphate as mordant, washing, light, and perspiration fastness grades either increased or remained the same in all the dyes whereas rubbing fastness grades decreased in four dyes and increased or remained the same in rest of the dyes.

#### **5.4 Comparison of fastness properties of different dyes**

##### **5.4.1 Washing fastness**

The colour of dyed samples changed in hue or increased on washing in the case of flame of forest, pine and turmeric dyes. Washing fastness grades of onion dye were minimum followed by ashoka, beefwood and golden drop dyes where washing fastness was fair to very good. The washing fastness was good to very good in the case of banyan, Indian blackberry and rubber plant dyes.

##### **5.4.2 Light fastness**

On exposure to light, the colour of dyed samples increased or changed in hue in the case of flame of forest and pine dyes. Light fastness was the best in the case of onion and Indian blackberry dyes, good in the case of banyan, beefwood and golden drop dyes, fair in ashoka and rubber plant dyes and poor in the case of turmeric dye.

### **5.4.3 Rubbing fastness**

Rubbing fastness grades of all the dyes were almost similar in the range 2 to  $\frac{3}{4}$  in beefwood and ashoka dyes where 4 and  $\frac{4}{5}$  grades were obtained. Thus, rubbing fastness of natural dyes on polyester was moderate to good in most of the cases.

### **5.4.4 Perspiration fastness**

Perspiration fastness of all the dyes on polyester was good to very good.

The change in colour on washing or on exposure to light was due to change in chemical structure of dye on reaction with alkali or due to photochemical degradation, respectively.

The increase in colour on exposure to light was due to dissolution of dye in heat of illumination. This occurs in the cases when dye aggregates in fibres which is characteristic of disperse dyes due to their insolubility in water.

## **5.5 Effect of mordants on CIE LAB values**

All the mordants helped in increasing dye uptake by the fibre in most of the cases as indicated by higher K/S values of the mordanted samples than that of control (unmordanted) sample.

Maximum colour strength was observed in the case of turmeric dye, followed by pine and onion dyes. Only in the case of flame of forest, golden drop and turmeric dyes, the K/S value of control sample was higher than that of mordanted samples.

Most of the mordanted samples were brighter than control samples in the case of beefwood, golden drop, Indian blackberry, pine and rubber plant dyes. In the case of ashoka, banyan, onion and turmeric dyes only one mordanted sample was brighter than control sample whereas all the mordanted samples were duller than control sample in the case of flame of forest dye.

### **Conclusions**

- The ten dyes used in the study had affinity for polyester and may be classified as natural disperse dyes since polyester can be dyed mainly by disperse dye.
- The optimum dyeing conditions for various dyes varied from dye to dye except a few optimum conditions which were similar in the case of few dyes.
- The optimum dyeing time for almost all the dyes was one hour or more. In the majority of the cases the post-mordanting was found to be the best to improve the overall fastness ratings.

- The synthetic mordants used helped in improving the fastness property of the natural dyes.
- The most suitable mordant for dyeing polyester varied from dye to dye.
- The synthetic mordants helped in increasing the dye uptake (colour strength) in the case of all the dyes.
- Different mordants modified the colours produced by different dyes. Thus, producing a variety of shades from a single dye.
- The optimization of dyeing condition of ten natural dyes employing four synthetic mordants also concluded in the development of 67 new shades for each dye totaling to 670 new shades for ten natural dyes.

### **Implications of the study**

- The present study has added new natural dyes for polyester to those being used in the textile processing at present. This will expand the shade range of the natural dyes available for polyester and thus, will help in their revival. This may also help in commercial production of these dyes for export.

- The study has been carried out using carrier dyeing method and this has given satisfactory fastness results. This can be used for dyeing polyester in small scale industry resulting in saving of energy.
- The knowledge generated by this study will be helpful to the traditional dyers in improving the colour fastness quality of the traditional textiles which will enhance their domestic and specially export marketability and thereby improve the economic condition of the dyers, thus helping them in continuing their profession and consequently preserving the textile heritage of India.
- The various dyeing processes developed through this study are simple to practice and will help the homemakers in dyeing their apparels and household textiles at home, thus enhancing their creativity and innovation.

#### **Suggestions for further studies**

- A study can be undertaken to find out the effect of the studied natural dyes on physical properties of the substrate.
- The work can be extended by investigating the application of various other bark, fruit, leaf, root and flower dyes on polyester.
- A comparative study can be done on synthetic fibres.

- A similar study can be conducted employing natural mordants instead of synthetic mordants.
- Another study can be carried out employing combination synthetic mordants or natural mordants or synthetic and natural mordants both.
- A study of the studied dyes can be conducted by using them in purified form for dyeing and investigating their chemical factors.
- The dyeing with natural dyes can also be studied by using different energy saving dyeing techniques, e.g. solar dyeing, ultrasonic dyeing, solvent assisted dyeing, supercritical fluid dyeing, etc.
- A study can be conducted on transfer printing with natural dyes on polyester.

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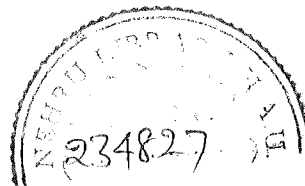
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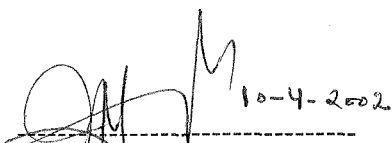
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### ABSTRACT

The present study was conducted to optimize the processes of dyeing polyester with ten natural dyes employing four synthetic mordants and carrier dyeing method. Nine parameters of dyeing were optimized and effect of seven of these parameters on the washing, light, rubbing and perspiration fastness properties was evaluated. Effect of different synthetic mordants on the colour of dyed samples was investigated in terms of CIE LAB and K/S values.

It was found that the optimum carrier concentration ranged from between 10 to 25 percent (o.w.f.) and optimum carrier treatment time ranged between 45 to 75 minutes for different dyes. The optimum pH values for extraction ranged between 5 to 10 and optimum extraction time ranged between 30 to 90 minutes for all the dyes. The optimum dyeing pH ranged between 4 to 10, optimum dye material concentration between 0.5 to 4 g/g of polyester and optimum dyeing time between 45 to 105 minutes for different dyes. Best mordanting method was found to be post-mordanting and optimum mordant concentration ranged between 1 to 20 percent (o.w.f.). Generally direct relationship was observed between rubbing and perspiration fastness and dyeing pH and dyeing time, respectively whereas inverse relationship was seen between washing and perspiration fastness and carrier treatment time and dyeing pH, respectively. No clear trend of increase or decrease in light fastness was observed with most of the dyeing parameters.

The most suitable mordant for each dye varied from dye to dye. All the mordants improved the fastness properties of the dyes, specially the light fastness, followed by washing, perspiration and rubbing fastness. These also enhanced the dye uptake in the case of all the dyes.

  
 10-4-2002  
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 Signature of Major Advisor

  
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 Signature of Student