APPLICATION OF NATURAL DYES FOR COTTON PRINTING

BY

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Dedicated

To

My Loving Family

CERTIFICATE-I

This is to certify that this dissertation entitled, **"Application of natural dyes for cotton printing"**, submitted for the degree of **Master of Science,** in the subject of **Clothing and Textiles** to the CCS Haryana Agricultural University, is a bonafide research work carried out by **Ms. Radhika Agrawal** 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.

MAJOR ADVISOR

CERTIFICATE-II

This is to certify that this dissertation entitled, **"Application of natural dyes for cotton printing"**, submitted by **Ms. Radhika Agrawal** to the CCS Haryana Agricultural University in partial fulfilment of the requirements for the degree of **Master of Science,** in the subject of **Clothing and Textiles**, has been approved by the Student's Advisory Committee after an oral examination on the same.

MAJOR ADVISOR

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DEAN, POST-GRADUATE STUDIES

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Radhika Agrawal

(RADHIKA AGARWAL)

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CHAPTER-I

INTRODUCTION

The art of dyeing and printing has played an important role in adding beauty to the textile world. Dyeing is the art of imparting particular hues and tints to thread, fabric and other material by employing colouring matter, whereas, in printing, design and colour forms an artistic expression to embellish the fabric. In other words, printed fabric are defined as the fabric which has a motif, pattern or design by application of dyes.

In India the dyeing and printing with natural dyes is an age old practice. Natural dyes have been part and parcel of men's life since time immemorial. *The fragments of madder dyed cloth found during the Indus valley excavation are the evidence of its great antiquity. The craft of printing also occupies a unique place in Indian civilization. It is believed that the process of printing with natural dyes has been in practice from as long as the 10th Century. The Moghul, rulers gave a fillip to this craft and it was enthusiastically patronised by the Rajput Kings (Lal, 1994).*

India has a rich tradition of using natural dyes and was the source of earliest natural dyes known to man. The art and craft to beautify the

textiles was being practised in every corner of our country by expert craft persons. This may be Bandha Sarees in Orissa and Rajasthan, Multani Sarees in Rajasthan, Gujrat, Madhya Pradesh, Kalamkari printing in Bihar and Andhra Pradesh, Sanganeri and Bagru printing in Rajasthan (Mishra, 2000 and Lal 1994).

With the growth of civilisation and technological advancement the needs of the people increased.To fulfill the requirement of the society, industrial revolution took place as a result there was an influx of synthetic dyes in 1856 which marked the first step in the decline of natural dyes. The bright shades and brilliance in colours of the new synthetic dyes drove the natural dyes in oblivion.

The unbridled use of synthetic dyestuff in textiles with full of chemicals increased day by day whose toxicity was unknown and neither questioned. Though there dyes makes our life gay, beautiful aesthetic and colourful, they also pose many problems as they release harmful amines which are carcinogenic and allergic in nature. Moreover the production of synthetic dyes involves many violent chemical reactions and the toxic by-products which are discharged in water or in the atmosphere leading to environmental pollution and health hazards. These ecological considerations prompted the German authorities to imposed a ban on those textiles dyed with azo-dyes or other such dyes that have carcinogenic effect.

Thus with the present national and international awareness on environment, natural dyes are back in vogue as these are non-hazardous, biodegradable and have better compatibility with the environment. They

also posses lower toxicity and allergeic properties (Vanker, 2000). Colours obtained from natural dyes have a harmonic-a-resonance-a depth that commercial dyes lacks. It is considered that natural dyes yield luster, the under glow of rich colour, aromatic smell, soft light and shadow soothing to human eyes (Chinchwade, 1997).

Today's scene of natural dye promotion is very optimistic and promising. The scientists and textile technologist of various institutions have started veering the sources of natural dyes as a possible means of producing ecologically sound products which would appeal to green minded consumers. A lot of information is available on dyeing with vegetable dyes, but very meagre information is available on printing with natural dyes which also play a very significant role in embellishment of the fabrics.

Keeping in mind the importance of printing with natural dyes, efforts have been made on the application of natural dyes for cotton printing with the following objectives :

i) To standardize the recipe of printing paste for printing cotton fabric with selected natural dyes.

ii) To study the effect of mordants on the prints obtained with selected natural dyes.

iii) To analyze the colour fastness of printed fabric.

CHAPTER-II

REVIEW OF LITERATURE

An attempt has been made here to present a brief resume of the available literature on the issues relevant to the present study under the following sub heads:

1. Traditional techniques of designing with natural dyes
2. Dyeing with natural dyes
3. Printing with natural dyes

1. TRADITIONAL TECHNIQUES OF DESIGNING WITH NATURAL DYES

India's traditional hand printed textiles are the oldest and famous for the exquisite work and designs. The art is assumed to have originated in the far east where the Hindus and the Chinese were known to have practised hand printing with wooden blocks. They were known to have printed cloth as early as 500 BC (Shenai, 1992). The following traditional techniques of designing with natural dyes are being practiced in India by few craft person.

I) Kalamkari :

Kalamkari is an exquisite ancient craft of painted and printed fabric with natural dyes. It derives its name from kalam means pen, and kari means work, literally Pen-work.

The colours used from natural sources are red from Indian madder root, yellow from the pomegranate seed or mango bark and black from myrobolan fruit. It demands a lot of treatment before and after printing. Depending upon the treatment of cloth or quality of the mordant, the colours change accordingly.

In Andhra Pradesh, both the Masulipatnam and Srikalahasti villages are recognized as major centers for kalamkari (Anonymous. Kalamkari-exquisite ancient craft. http://www.chennaionline.com/artscene/craftplce/hi.../kalamkari.as).

II) Sanganeri and Bagru prints :

Sanganeri and Bagru are two important places of printers concentration in Rajasthan. The ingredient used and the process of printing in sanganeri and Bagru are more or less the same.

Printing techniques :

The process of printing involves the following steps :

a) Pre treatment of fabric

Katia : Cutting of the cloth

Hari sarana : Designing, scouring and bleaching with the paste of goat, sheep, cow or buffalo dung.

Tapai : Spreading of fabric under bright sun and removal of dung paste by washing.

Harada karana or peela karana : Mordanting with myrobolan.

Peela Jharana : Removal of superfluously hold myrobolan.

b) Printing : The fabric is printed with the following dyes obtained from various vegetable source.

Vegetable sources	Colour obtained
Geru (Begar)	Red
Rusted iron and jaggery (Siahi)	Black
Pomegranate rinds	Mustard green
Babool bark	Orange brown colour
Ratanjot	Greenish mustard
Jackfruit and saw dust	Greenish yellow
Red sandal	Red brown
Kacha katha	Brown
Kashula flowers	Yellow brown
Chawal kodi	Red brown
Harshingar or babool bean	Yellow brown

III) Ajarakha

The most popular item in Badmer prints of Rajasthan, is a sheeting cloth printed in bold designs in red, blue and black, with a peculiar resist effect. Lime and tamarind seed powder are used to produce the resist effect.

IV) Ikat :

The warp and weft yarn are both tied and dyed before weaving using many natural dyes as well as indigo. In Orissa and Andhra Pradesh the yarn tie-dyed is known as ikat where as in Gujarat are know Patan Patola (double Ikat).

Teli Rumal :

Teli Rumal of Andhra were also traditionally woven using double ikat and natural dyes. The themes mainly consist of ginds and small geometric designs or figurative work. The colours, mostly red, pink or purple are made from alizarin, iron or indigo and numerous other vegetable sources.

2. DYEING WITH NATURAL DYES

A lot of work on application of natural dyes have been done in India but the studies conducted in recent past have been included in this chapter.

Daiwabo (1994) developed 'chemical free' dyed fabric made up of organic cotton. The chemical free methods included using food colour dyes and natural edible colours. Scouring agents included extracts from oranges, apples, sugar or coconut oil and gardenia and cochineal were used for pale yellow and pale pink dyes. Softening agent have also been developed from sugar, coconut oil and beef fat.

Dopoola *et al.* (1994) reported Extraction spectroscopic and colouring potential studies of the dye in ginger rhizome (*Zingiber*

officinale). The dye was extracted quantitatively using the solid-liquid extraction method and its physico-chemical parameters as well as colouring potential was studied. A wave length of maximum absorption was max-420 nm. The dyes gave a brilliant yellow colouration of light, wash and dry ironing ratings of 5, 3 and 4 respectively on bleached cotton.

Gurley (1995) developed a process for permanently dyeing cotton, linen or cellulosic fibers after pretreating the fibres at 110-170°F with a mordant comprising an aqueous colloidal suspension formed by adding soda ash and alum to warm water. The treated fabric were dyed in a aqueous solution using natural dye liquor.

Bhattacharya *et al.* (1996) investigated Eco-friendly dyeing of cotton fabric with the naturally occuring dyestuff *Katha* (*Acecia catechu*) which has been done in an automatic jigger at levels of 0.5 to 3%. The resulting brown fabrics, when assessed for colour strength, wash fastness, acidic and alkaline perspiration fastness, light fastness and rubbing fastness found that it was good with respect to the ISO and test wash method.

Saxena *et al.* (1997) standardized the method of application of lac dye on cotton using chitosan (a naturally occuring polymer) for pretreatment. Alum, ferrous sulphate and tannic acid were used as mordants. The dyed fabric had medium light fastness, good rubbing and perspiration fastness but poor wash fastness. The fastness to washing showed a marked improvement when a cross linking treatment with DMDHEU was given to the dyed fabrics.

Kalyani and Jacob (1998), studied the tapping dye from *gulmohr* for cotton. The dye material concentration was selected 3g. Out of four methods of extraction i.e. aqueous, alkaline, acidic and alcoholic, aqueous and alkaline mehod was found the best. Dye extraction time and dyeing time was 60 min and mordanting time was 30 minutes. Premordanting method gave better results. Copper sulphate (0.5%), potassium dichromate (0.5%) with aqueous dye extract and ferrous sulphate (0.4%), cobalt sulphate (0.6%) with alkaline dye extract were used to get colours like olive green, green, brownish black and biscuit. Samples have fair to excellant fastness to dry rubbing. Wet rubbing exhibited poor to good fastness. Samples exhibited poor to good washing fastness. To alkaline perspiration test, the samples showed poor to excellant and good to excellant fastness was shown with acidic prespiration. Sunlight fastness showed poor to fair fastness after 40 hrs of exposure.

Katyayini and Jacob (1998) optimized the extraction of the dye from mesta calyx. When extracted directly from mesta caylx showed better rests as compared to the powdered form. It produces different colours such as pink, pinkish brown and violet with alum, copper sulphate and stannous chloride respectively.

Kumar and Bharti (1998a) investigated that *Eucalyptus* yield dye. Aqueous extract of the hybrid *Eucalyptus* bark yield bright brown dye with media 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.

Padmaja and Jacob (1998a) investigated development of dye from *red hibiscus* flowers and its assessment of cotton. Four methods of extraction were tried on cotton in the pre-testing. Acidic media of dye extraction was found best. Among the three mordanting methods post mordanting method was found the best. Dye material concentration was kept 3 g. Dyeing time and extraction time was 60 min. and mordanting time was 30 min. Mordants used were Alum (0.3%) copper sulphate (0.5%), Lead acetate (0.7%), ferrous sulphate (0.5%), zinc chloride (0.4%), cobaltous sulphate (0.7%). Biscuit colour had good fastness to rubbing, but during washing showed fair to poor fastness to colour change, and good resistance to colour staining. Fastness to both acidic and alkaline prespiration as well as sunlight fastness were found to be good.

Prabhu and Raja (1998a) extracted a dye from African marigold (*Tagetes erecta*) for dyeing cotton fabric. In slightly acidic medium it gave dull yellow colour, which showed poor fastness properties, but in slightly alkaline medium, a deep yellow coloured fabric was produced with moderate fastness properties. Addition of sodium chloride and sodium sulphate as exhausting agent did not show much change in colour and the dye uptake.

Prabhu and Raja (1998b) dyed cotton with myrobolan using various metallic salts as mordants using for different dyeing times and assessed colourfastness properties. Though dyeing for 90 minutes showed no improvement in the depth of the colour over 75 minutes, but fastness ratings were very good.

Prabhu and Senthilkumar (1998) developed a dye from *Rosa indica* using tannic acid, myrobolan in combination with copper sulphate, aluminium sulphate, potassium dichromate, ferrous sulphate, stannous chloride and tartar emetic. The results revealed that the dye gave dull yellow colour and showed good to very good washing and light fastness.

Samy and Raja (1998) reported that cotton fabric could be dyed using dye extracted from Arecanut (*Areca catechu*). Desized, scoured and bleached woven cotton fabrics were dyed both with and without mordants and fastness properties were assessed. It was concluded that natural dyes gave a range of shades from beige to black with satisfactory fastness properties on dyed fabrics.

Devi *et al.* (1999) studied dyeing of cotton fabric with turmeric (*Curecuma longa*) using synthetic mordants for colour fastness properties, general fabric appearance : including luster; texture and fabric count, weight, thickness, breaking strength and elongation. Approximately 63 per cent of judges rated the general appearance of untreated samples as good, whereas 60 per cent evaluated the samples treated with alum as fair and samples treated with ferrous sulphate had a poor appearance.

Gaba (1999) extracted dyes from the flowers of *pili kaner* and *jatropha* to dye cotton fabric. The optimum values for *jatropha* and *pili kaner* were 3 per cent dye concentration, 90 and 75 minutes extraction times, respectively and 60 minutes dyeing time. Pre mordanting method was adjudged best. In both the dyes the tensile strength increased in

almost all the cases in warp direction but decreased in weft direction, it might be due to swelling of fibres, the loss was minimum. Elongation percentage showed increase in some cases while decrease in others. Flexural rigidity in all the dyed samples showed increase in both the directions.

Khanna and Dedhia (1999) dyed pineapple leaf fibres (PALF) with two natural dyes, namely *ratanjot* and *eucalyptus* leaves using mordants and their various combinations. When the pretreatment with *harda* and pomegranate rind were compared regarding the strength, reflectance and fastness properties, it was inferred that in some cases *harda* treatment was better whereas in others pomegranate rind. It was also found that the type of mordant or mordant combination had an effect on the reflectance, strength as well as fastness properties of PALF dyed.

Radhika and Jacob (1999) developed a dye from *jatropha* seeds for dyeing cotton pretreated with myrobalan and indicated that alkaline method was the best method of extraction with dye concentration 15 g and dyeing time and extraction time 60 minutes. It was found that *jatropha* seeds gave a range of bright, even and soft colours on cotton fabric using different mordants. All the dyed samples showed fair to excellent colourfastness to various agencies.

Vastrad *et al.* (1999) carried out a study to evaluate the colourfastness of cotton fabrics dyed with three naturally available sources, viz., African marigold (*Tagetes erecta*), Golden rods (*Solidago Canadensis*) and onion skin (*Allium cepa*). It was revealed that tin

imparted good wash fastness to samples dyed with golden rods; chrome for marigold dyeing and alum and tin for dyeing with onionskins. Irrespective of the dyeing material, alum showed good fastness to dry crocking and perspiration.

Ghorpade *et al.* (2000a) dye cotton in sonicator using eco-friendly mordants with red dye extracted from Sappan wood by techniques using ultrasound energy. This dye when dyed in sonicator showed medium fastness to washing, rubbing and perspiration. With the aid of tannic acid and eco-friendly metallic mordants improved the fastness properties that was earlier considered as fugitive.

Ghorpade *et al.* (2000) a one-bath process of dyeing using sonicator and simultaneous antimicrobial finish by methanolic extract of tulsi leaves (*Ocimum sanctum*) was investigated and very encouraging results were observed. One bath dyeing process showed very effective dyeing as evaluated by the fabrics' fastness properties. Similarly, the antimicrobial activity evaluated by soil burial test of both treated and untreated fabric exhibited the resistance of microbial growth on the treated fabric after soil burial for 3 days.

Ghorpade *et al.* (2000b) exploited a natural colouring material from canna flowers extract to dye cotton and concluded that dyed samples had good fastness properties. The colour range was from pinkish purple to dark purple by pre-mordanting in stannic chloride, sap green with alum, dark green and yellow mustard with ferrous sulphate with pre and post mordanting, respectively.

Rastogi *et al.* (2000) used *lac* dye on cationised cotton fabric. Maximum colour yield was obtained in the fabric treated with 5 per cent Discofix DBA at pH 4.0 and exhibited good fastness to washing and perspiration, fair to good fastness to light and excellent fastness to dry and wet.

Singh *et al.* (2000) studied the effect of *Jatropha integrima* on physical properties of cotton fabric. Samples were pre-mordanted with 1, 3, 5 per cent of tin and iron and dyed. Results revealed that fabric count, weight and thickness of all dyed samples had increased. Tensile strength increased except 1 per cent tin in warp direction and decreased in weft direction for all the samples. Elongation per cent decreased in 1 and 3 per cent of tin in warp and weft direction, whereas the fabric dyed with 1, 3 and 5 per cent of iron and 5 per cent of tin showed increase in elongation. Flexural rigidity increased in all cases of tin and iron except 3 per cent tin.

Tiwari *et al.* (2000a) carried out ultrasonic dyeing with *tulsi* leaves (*Ocimum sanctum*) for cotton using eco-friendly mordants. It was observed that the dye did not give much variation in colour; however, colour adherence to fabric was good. The colours obtained were light green, fluorescent green shades, dark green kakhi with stannic chloride, stannons chloride, alum and ferrous sulphate, respectively. All the dye samples exhibited fair to good fastness to rubbing and good fastness to light and washing.

Tiwari *et al.* (2000b) used aqueous extract of *bougainvillea* for dyeing cotton in sonicator. Natural, acidic and basic solution of the

dye extract showed a full gamut of colours from dark magenta in acidic medium to yellow in basic medium. Basic extract gave *mehandi* green with alum. The washing and light fastness of dyed samples were fairly good.

Vankar *et al.* (2000a) investigated newer natural colourants, ultrasonic energized dyeing of cotton fabric with deep purple flowers of cineraria was carried out. Colour ranged from purple to dull green depending upon the use of mordant. The results were excellent with stannic chloride; however, alum, ferrous sulphate and stannous choloride also gave fairly good fastness properties.

Vankar *et al.* (2000b) investigated the ultrasonic energized dyeing of cotton fabric with eucalyptus bark using eco-friendly mordants.Post-mordanting was better in terms of fastness properties. The colours obtained were ranging from light peach to brown, light green to dark olive green depending on the type and concentration of mordants. The colour was evaluated by means of L*, a*, b* colour coordinates. Also revealed that the bark after extraction of dye could be successfully used for removal of heavy metal from textile effluents.

Dayal *et al.* (2001) extracted a natural dye from *pinus roxburghii* bark and needles for dyeing cotton fabric could be used to isolated the dye in 4.8 and 5.0 per cent yield, respectively. Different colours ranging from light brown, brownish peach and dark brown were obtained. The dyed samples had very good wash fastness; good light, crocking and perspiration fastness except stannous chloride treated samples which had poor wash and light fastness.

Eom *et al.* (2001) cationized the cotton fabric before dyeing with natural colourant, namely Redwood, Gromwell,, Cochineal, Goldthread and *Amur* cork tree for improving its dye ability. The K/S value of cationized cotton fabric dyed with Redwood and Cochineal was higher then that of untreated one. In case of Gromwell, though the K/S value of the cationized fabric was higher than that of untreated one, the fabric showed poor levelness. Cationization of cotton had no effect on the dye ability of Goldthread and *Amur* cork tree. It was observed when the concentration of sodium hydroxide exceeds beyond a certain level NaOH hydrolyzes the cationizing agent and the K.S. value of dyed materials decreased.

Senguta *et al.* (2001a) conducted a study on dyeing cotton fabric with liquor with or without synthetic mordants. It was observed that the K/S values of the dyed samples increased with the increased depth of shade in all the cases, even with the sample dyed without mordant. All the dyed samples without mordant or with the addition of mordants exhibited good washing and rubbing fastness properties.

Singh *et al.* (2001) standardized the dyeing conditions for cotton with a floral dye *Rein wardtia* trigyana. Results of the study revealed that the suitable wave length was 440 nm and the optimum dye extraction time was 15 minutes. The maximum colour yield was obtained by 7 per cent dye material concentration when dyed for 45 minutes.

Tiwari *et al.* (2001) reported the dyeing properties of cotton fabric and hosiery material with *al* roots by using sonicator. A variety of shades

were produced ranging from peach to brown, light green to dark brown to black. Best results were obtained with stannic chloride as mordant for cotton as well as for hosiery. The dyed samples exhibited good to very good fastness properties to all agencies.

Tiwari and Vankar (2001) extracted a dye from alkanet root bark and dyed pretreated cotton with acetic acid in sonicator and microwave. The napthaquinone based chromophore dye exhibited high affinity for cotton and a variety of shades were obtained with different mordants. All the dyed samples showed good colourfastness.

Vankar *et al.* (2001b) carried out ultrasonic energized dyeing of cotton fabric with *Cassia fistula* bark (Amaltas). Colour obtained ranged from light peach to brown, light green to dark brown to black depending on type concentration of mordants. The colour of dyed samples had been evaluated in terms of L*, a*, b* values. All the dyed samples showed good dyeing properties and mordanting enhanced the colourfastness properties.

Yadav *et al.* (2001) conducted a study to optimize the dyeing parameters for cotton with barberry and *black kikar* bark. It was observed that optimum wave length for *barberry* was 410 nm and for *black kikar* it was 430 nm. The optimum dye material concentration was 8 per cent and 6 per cent, optimum extraction time was 60 and 75 minutes, for barberry and *black kikar* bark dye, respectively. Best shades were obtained when dyeing was carried out for 45 and 60 minutes for barberry and *black kikar* bark, respectively.

Bhattacharyya and Lohiya (2002) dyed cotton with four vegetable dyes viz. turmeric, pomergranate rind, catechu and Nova Red (mixture of *magistha* and betel nut) using conventional and HTHP method of dyeing. The colour data as well as wash, light and perspiration fastness characteristics were investigated. The results indicated improved fastness with most of the dyes when dyed with HTHP method as compared to conventional method.

Devi *et al.* (2002a) used a natural colourant annatto for dyeing cotton using metallic mordants. Annatto produced a very bright orange shade on cotton. The colour was found to be sensitive to alkali reagents. By observing the fastness properties of mordanted annatto dyed cotton it could be concluded that the dye was having very fair wash fastness, good to excellent crocking fastness and fair to good perspiration fastness.

Devi *et al.* (2002b) extracted a dye from pulp of *amaltas* (*Cassia fistula*) for dyeing cotton. Pre and post mordanting methods were found suitable for application of *amaltas* dye. Even though the absorption values were reduced in mordanted cotton, visually the shades produced were darker. All mordanted samples showed excellent to outstanding sunlight fastness, good to very good wash fastness, excellent resistant to crocking and good perspiration fastness.

Mahale *et al.* (2002) investigated the colourfastness properties of eco dyed cotton with marigold. Results of the study revealed that the sample pre mordanted with 3 per cent potassium dichromate showed good fastness properties for all agencies followed by 15 per cent potash

alum concentration. Among all the simultaneously dyed samples 1 per cent potassium dichromate showed highest grades for all the colourfastness properties. Samples post mordant with 2 per cent and 3 per cent potassium dichromate showed good colourfastness to washing rubbing, sunlight and perspiration.

3. PRINTING WITH NATURAL DYES

Shenai and Date (1979) in a study ain bark extract was used for printing on silk. Roller printing was done. The fabric was printed with a thickened solution of bark extract, followed by development in diazo salt solution. After printing it was dried and steamed at 5 psig pressure in a cottage steamer for 25 min. Good to very good all-round fastness properties were produced using orange GC, Red RBI, Kuma Blue BL, Kurma Red B, O-toluidine and sulphanilic acid. Faulty prints could be completely stripped by a treatment with sodium hypochloride solution.

Shenai and Rao (1979) studied the two methods. In first method cotton fabric was printed with thickened solution of the Ain bark extract, steamed and developed with a passage through diazo salt solution and reverse method was followed in second time i.e. padding the cloth with Ain bark extract solution, drying and printing with thickened solution of diazo salts. Better prints were obtained in case of first method. It was seen that washing fastness of all the prints obtained by first method was excellent, while light fastness was good.

Udaiyini and Jacob (1988) developed new vegetable dyes for kalamakai printing and assessed their colour fastness properties. Grey desized cotton material was used which was pre-treated for two days

prior to dyeing. Vegetable dyes Bixa orellena seeds, Indigo fera tinetoria leaves, orange red heartwood of coesalpnia soppan were used for dyeing orange, blue garnet and lavender colours. Results of colour fastness tests overall efficiency of blue was the best followed by orange, garnet and lavender.

Kaur (1995) studied different methods of mordanting using alum, ferrous sulphate and *Harda* with some commonly used natural dyes - *Marjith, Patang, Tessu, Dohi, Kusum, Anaar, Harada and Babul.* It was concluded that premordanting with $FeSO_4$ and printing with almost all the dyes produced a range of dark brown to black on cotton fabric that were very fast to washing and light. Though alum printed with various methods produced a variety of hues but no direct correlation with ferrous property was found.

Goel and Chauhan (1996) prepared printing paste for cotton using mordants and thickener. Three different concentrations (6, 8 and 10%) of printing paste were applied by blocks on desired area of fabric. After printing the fabric was dried, kept overnight and steamed for 45-60 min to fix the mordant. These samples were than dyed with natural dye extracted i.e. Manjistha. Finally, the fabric was given a cold rinse, dried and ironned in a slightly moist conditions. The results indicated that washing made the colour faster and brighter. It was also concluded that premordanting method was best for penetration of dye into the fabric.

Gupta and Goel (1997) conducted experiments to standardized the recipe for application of mordants for printing of silk using *tissu*

flower dye. The effect of mordants on colour development and their methods of applications were studied. One per cent to 10 per cent concentration of chrome gave light to dark reddish maroon colour. A wide range of shades were produced when using with other mordants such as stannous chloride and ferrous sulphate. Light to darker colour prints were obtained by varying the concentrations of mordants i.e. 2, 6 and 10 per cent. Excessive concentration upto 10% showed bleeding of colour and also fabric become brittle of harsh.

Goel and Goel (1997) investigation was carried out to explore the usefulness of kitchen waste material; onion skin, to obtain a wide range of colours on silk fabric with the help of different mordants. It was observed that low percentage of alum upto 4 per cent does not gave any print. About 6-10 per cent concentration of alum gave a light shade print. While 1-10 per cent concentration of chrome imparted light to dark reddish maroon colour, ferrous gave light to dark brown colours, copper produced coppery brown and stannous chloride gave an orange coloured prints. Combination of mordants improved the depth of colour, appearance, lustre and sharpness of printed fabric.

Kola (2001) made an attempt to dye and print cotton, silk and polyester with onion peels. Printing paste was prepared by adding 600 mg of ferrous sulphate in 60 ml of dye extract. CMC (2.8 gms) was added as a thickener and smooth paste was made by stirring. Printing was done by blocks and samples were treated in hot oven at by 70°C for 15-20 minutes, kept overnight and washed in running water. The colour fastness test of all the samples showed negligible or slightly

changed colour in case of cotton and silk fabric while in case of polyester, noticeable change in colour was observed. In printed polyester fabrics, staining of the background fabric was seen.

The review of literature reveals that a number of research studies have been conducted on dyeing with natural dyes. But very meagre studies have been done on printing with natural dyes.

CHAPTER-III

MATERIALS AND METHODS

The methodology has been explained under the following sections:

1. Selection of raw material
   - i) Textile material
   - ii) Dyes from vegetable sources
   - iii) Chemicals
   - iv) Thickener
2. Preparation of fabric for printing
3. Determination of preliminary data of fabric
   - i) Fabric count
   - ii) Weight per unit area
   - iii) Fabric thickness
4. Preparation of guar paste.
5. Preparation of screens for printing
6. Standardization of printing paste
   - i) Optimum conditions for dye extraction
   - ii) Optimization of dye extraction pH
   - iii) Optimization of dye paste
   - iv) Optimization of pH of guar paste
   - v) Optimization of dye paste and guar paste ratio
   - vi) Optimization of fixer concentration
   - vii) Selection of mordants
   - viii) Optimization of mordant concentration

7. Printing of cotton fabric
8. Determination of colour fastness properties of printed samples
   - i) Fastness against washing
   - ii) Fastness against sunlight
   - iii) Fastness against rubbing
     - a) Dry rubbing
     - b) Wet rubbing

1. SELECTION OF RAW MATERIAL

i) Textile material :

Pure cotton fabric was selected for the study.

ii) Dyes from vegetable sources :

On the basis of literature twenty locally available vegetable dyes were tried. After pilot study three sources were selected for the study which have been listed in Table 1 (Plate 1-3). The selection was made on the basis of printing affinity and colours obtained.

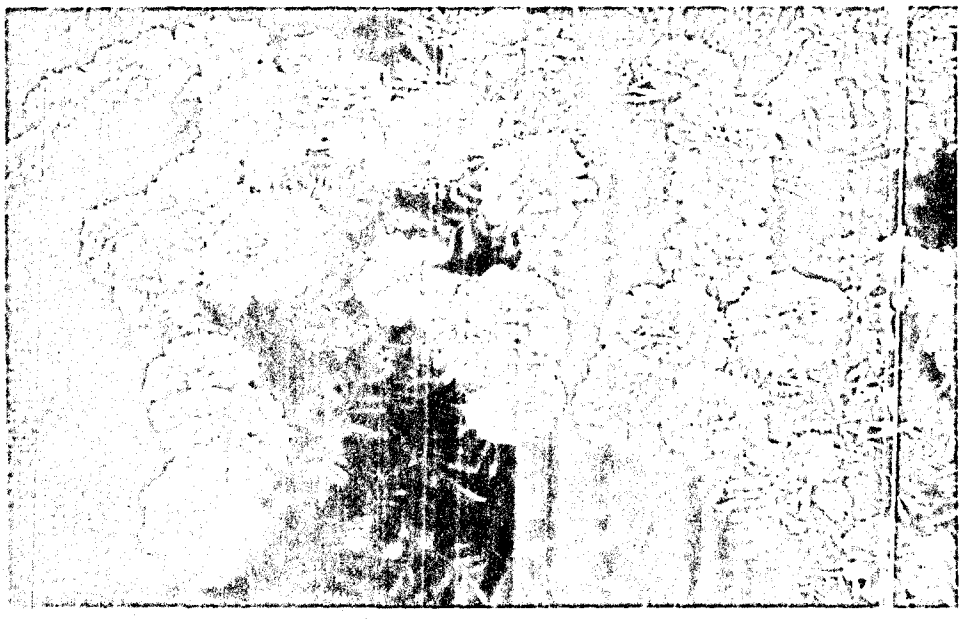
Table 1 : Dye materials used in the study.

English name	Local name	Botanical name	Parts used
Marigold	*Ganda*	*Tagetes erecta*	Flower
Rose	*Gulab*	*Rosa indica*	Flower
Peepal	*Pipal*	*Riligiosa ficus*	Bark

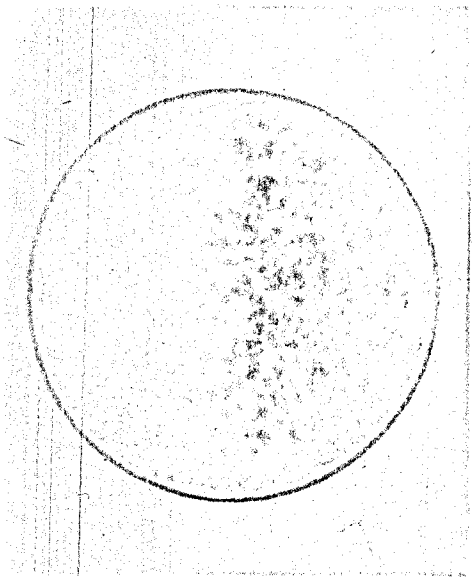
iii) Chemicals :

Laboratory grade chemicals were used for the present study and have been listed in Table 2.

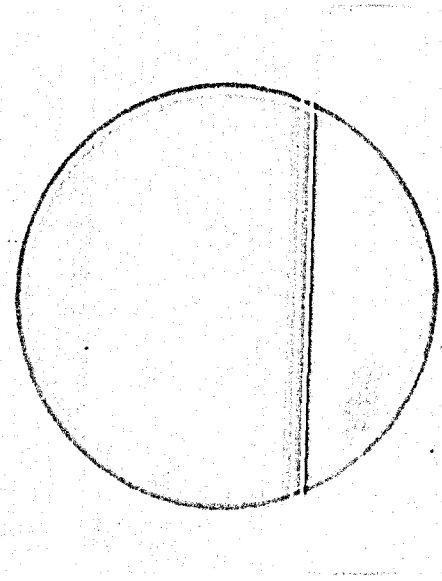
MARIGOLD



Flowers



Petals



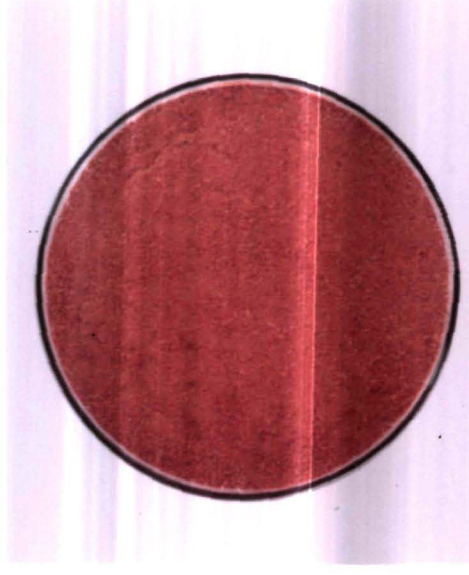
Dye Powder

Plate 1

MARIGOLD

Flowers

Petals



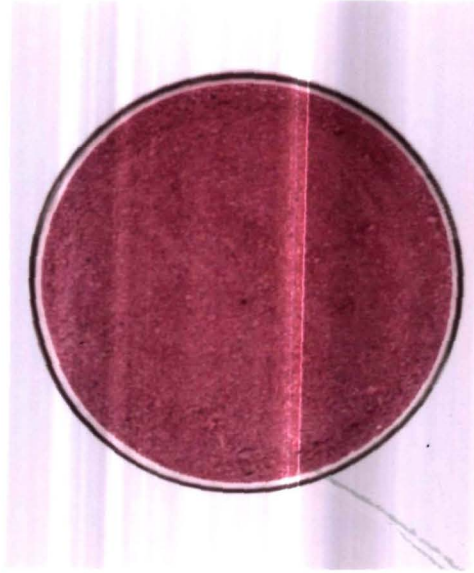
Dye Powder

Plate 1

ROSE

Flowers

Petals



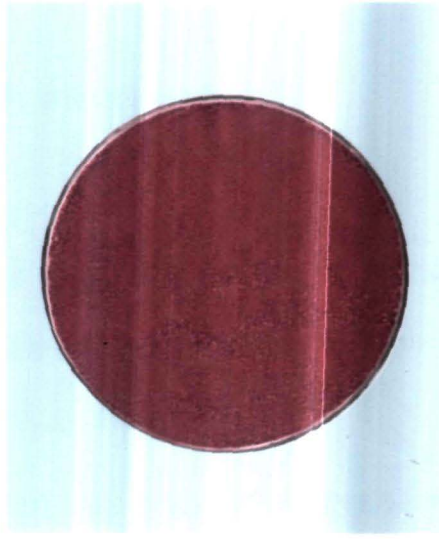
Dye Powder

Plate 2

PEEPAL

Tree

Bark



Dye Powder

Plate 3

Table 2 : Chemicals used in the study

	Chemical name	Purpose
1.	Sodium hydroxide	To control pH and for scouring.
2.	Hydrochloride acid	To control pH
6.	Neutral soap	For scouring and washing.
7.	Acrafix	Fixer
8.	Potassium dichromate	For mordanting
9.	Copper sulphate	For mordanting
10.	Zinc Chloride	For mordanting
11.	Ferrous sulphate	For mordanting

iv) Thickener :

Guar gum was selected to thicken the paste (Plate 4) for printing because it is a natural source and has maximum viscosity as compared to other thickener (Shenai, 1976).

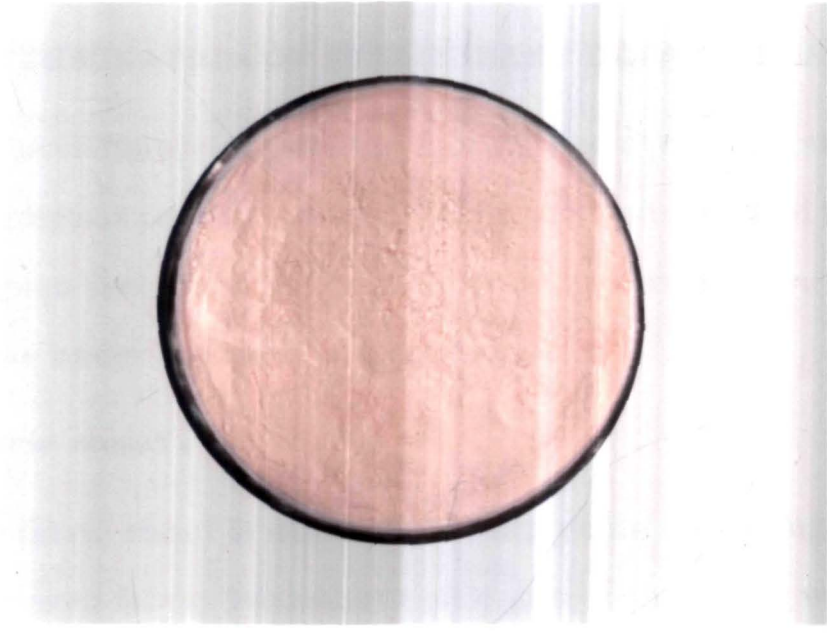
2. PREPARATION OF FABRIC FOR PRINTING

Scouring of cotton fabric :

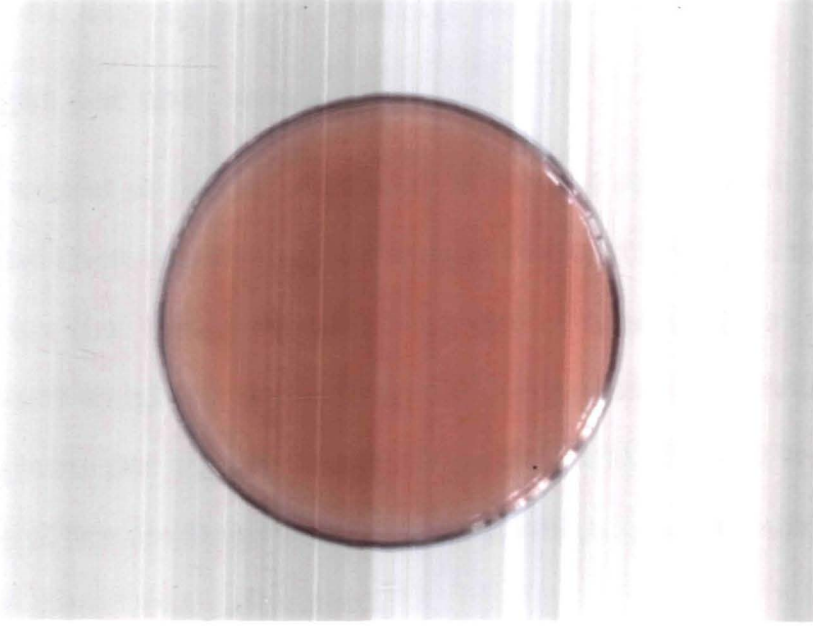
Scouring was done for removal of natural and added impurities like oils, fats, waxes and other adventitious dirt that might have been added to the fabric during manufacturing process.

The fabric was weighed and wetted out prior to impregnating in scouring bath. The fabric was squeezed thoroughly and was scoured in a solution containing sodium hydroxide (1 gm/litre) and neutral

THICKENER



Guar Powder



Guar Paste

Plate 4

soap (2 gm/liter) at boiling temperature (100°C) for 1 hr with material to liquor ratio 1:40. Then it was rinsed thoroughly and dried.

3. DETERMINATION OF PRELIMINARY DATA OF FABRIC

The preliminary data of the fabric to be dyed was taken under three parameters i.e. fabric count, weight per unit area and thickness. The samples were conditioned prior to determination of fabric dimensions under standard test conditions.

i) Fabric count :

The fabric count is numbers of warp yarns and filling yarns per inch in a woven fabric. Paramount pick glass with pointer was used to determined fabric count using ASTM-D 123 test method. It was determine by counting the number of threads per square inch in the warp and weft directions at five different places in the fabric using pick glass. An average of five readings was taken.

ii) Weight per unit area :

The weight of fabric is defined as weight of a known area of the material and then computing the weight per unit area. Samples of size 20 x 12.5 cm (i.e. template size) were cut at random from fabric. The samples were weighted separately on the paramount precision scale for GSM (grams per square meter) using ASTM D3776-90 test method. An average of five readings was taken and weight per unit area in grams per square meter was calculated.

iii) Fabric thickness

Thickness is defined as "the distance between two parallel surfaces while exerting a specified pressure on the material", the

pressure to be applied on the specimen by the pressure foot of the tester. Paramount thickness tester was used to determine the thickness of fabrics using ASTM-D1777-60 test method. A specimen was placed on flat surface below pressure foot of the instrument, without any folds. The pressure foot was lowered slowly upon the specimen until the pointer of the dial meter stopped moving further and the reading on the dial was recorded in mm. An average of five readings was calculated as the fabric thickness.

4. PREPARATION OF THE GUAR PASTE

To prepare thickener paste, 80 gm guar gum was soaked in one liter of luke warm water for 18 hours and then placed on preheated hot plate for 1 hour with constant stirring to get the homogenous mixture, which was finally strained through a nylon mesh (Shenai, 1976).

5. PREPARATION OF SCREEN FOR PRINTING

Screen printing technique was used for printing of cotton fabric. For the present study the screen was prepared using photo-chemical process.

Screen frame was made of wood (smooth, well seasoned and low water absorbancy) with nylon gauze. In photochemial method, screen was coated with sensitizing solution (mixture of gelatine and potassium dichromate). Coating was done in day light (not in direct sunlight) and dried. After complete drying, the screen was exposed under a transparent paper with ink in an exposure frame with close contact between the design and screen to the source of ultraviolet light for 5

minutes at a distance of nine inches from the lamp. After exposure the screen was washed in dark room by immersing in hot water without applying any friction to gauze. As soon as the design portion appeared quite open, the screen was washed again with cold water and allowed to dry.

6. STANDARDIZATION OF PRINTING PASTE

Variables for printing of cotton fabric has been explained in this section under following sub-section.

i) Optimum conditions for dye extraction :

All the three dyes selected for the study were extracted at optimum conditions on the basis of literature.

Table 3 : Optimum extraction conditions.

Name of the dye	Percent dye concentration	Extraction time (min.)	Source
Marigold flowers	6	45	Mahale (2000)
Rose flowers	10	15	Prabu (1999) and lab. experiment
Peepal bark	10	30	Karenjkar et al. (2000)

The raw dye material i.e. flowers and bark were collected, dried in shade and ground to make powder form. For dye extraction of selected dyes, optimum quantity of dye power (Table 3) was dissolved properly in aqueous medium. The solution was kept on pre-heated (boiling temperature) water bath and extracted for optimum time. During

extraction, the dye material was allowed to float freely in the water and the solution was stirred frequently. After removal from water bath solution was strained through nylon cloth and thus clear dye solution was obtained.

ii) Optimization of dye extraction pH :

The dye material was extracted at five different pH values (5,6,7,8,9). The dye solution thus obtained was used for printing samples, which were steamed and rinsed in cold water.

The printed samples were visually evaluated by five judges having experience of working with natural dyes. A tool for subjective evaluation was prepared to assess the colour of printed design visually on three point scale i.e. excellent, good and fair scoring 3, 2 and 1, respectively. Weighted mean score was calculated for selection of the best samples. Visual evaluation was got done to optimize pH for dye extraction, dye concentrate, pH of guar paste and selection of mordants.

iii) Optimization of dye paste :

Dye paste is vary important component of printing paste as it impart colour. The optimum amount of dye material was extracted at optimum pH for optimum time in three different extraction baths and strained. These extracted solutions (100 ml each) were reduced to three different volume i.e. 10 ml 'A', 7.5 ml 'B', 5 ml 'C' to make the dye paste. The samples were printed, steamed, rinsed and evaluated to find out the best dye paste [as mention in section 6 (ii)].

iv) Optimization of pH of guar paste :

Thickener paste i.e. guar gum paste was optimized using three different pH values i.e. 4, 6, 8. The samples were printed, steamed and rinsed. The printed samples were evaluated visually for optimization of pH for thickener paste [as per section 6(ii)].

v) Optimization of dye paste and guar paste ratio :

The main constituents of printing paste are dye and thickener. To standardize the printing paste, three different ratios of dye paste and thickener paste i.e. 1:4, 1:5 and 1:6 were tried. Optimum ratio for printing paste was find out on the basis of washing fastness of printed samples. The change in colour of printed design and staining on adjacent fabric was assessed using grey scale No. 1 and 2 respectively (ISO 105 method).

vi) Optimization of fixer concentration :

The third component of printing paste was fixer for which acrafix was used. The optimum ratio of dye paste and guar paste was taken as determined in section 6(v). Four different fixer concentration 0.5, 1.0, 1.5 and 2.0 per cent on the basis of weight of printing pastes (OWPP) were used and a without fixer sample was also printed. The printed samples were steamed and washed in the laundrometer. After washing the samples were got evaluated by the experts with grey scale No. 1 & 2 (ISO 105 methods).

vii) Selection of mordants :

Seven different mordants were tried for printing and on the basis of visual assessment and washing fastness two of them were selected.

viii) Optimization of mordant concentration :

The mordant concentration was optimized using 3 concentrations viz. 3, 5 and 7 per cent (OWPP) of all the selected mordants. Using optimum printing paste [Section 6(i-vi)] samples were printed, steamed and rinsed in cold water.

The amount of mordants was calculated on the basis of total weight of the printing paste as.

$$\text{Amount of mordant} = \frac{\text{Total weight of printing paste} \times \% \text{ of mordant}}{100}$$

Simultanous mordanting method was carried out to make the printing paste.

7. PRINTING OF COTTON FABRIC

For preparing the printing paste, the optimum amount of dye paste and guar paste was mixed and optimum amount of fixer and mordant was added on the basis of total printing paste and stirred vigorously to produce a uniform printing paste. The cotton fabric was printed with optimum printing paste using screen printing technique.

The fabric was then dried and after 24 hours it was steamed at 100°C for 45 minutes in a steaming chamber. The steamed samples were given a rinse in cold water so as to remove superficial printing paste (Gupta and Goel, 1997).

8. DETERMINATION OF FASTNESS PROPERTIES OF PRINTED SAMPLES

Fastness is the ability of dye to retain its colour after exposure to sun, washing or other colour destroying agents (Wingate, 1988).

In the present study the wash fastness, rubbing fastness and light fastness properties of the printed samples were studied by the methods prescribed by the Bureau of Indian Standards. Colour fastness was got assessed from judges.

i) Fastness against washing :

The printed samples were evaluated for washing fastness. For the present study, the washing fastness test was carried out as per recommendations of IS:3361-1979 method.

Preparation of samples :

A printed specimen measuring 10 cm x 4 cm of the material to be tested was cut out and placed between two pieces of plain undyed fabrics measuring 10 cm x 4 cm. One piece was of white cotton and other was wool fabric. The three pieces were stitched around the edges. The composite specimen were weighed and the required quantity of soap solution at the rate of 5 gm/l of water was prepared keeping material liquor ratio 1 : 50. One composite specimen was placed in each of the eight containers of launderometer containing soap solution.

The composite specimen were treated for 45 minutes at 50±2°C in the launderometer. The specimen were removed and rinsed in cold water and squeezed. The stitching along the two long sides and one

short side was removed. The samples were opened and dried in air with three pieces (one test specimen and two adjacent pieces). The visual difference was compared between the original and tested material with the difference was assessed with grey scale No. 1 as per the recommendations of ISO105 method.

Also the degree of staining on the undyed cloth was assessed with the help of grey scale No. 2 for staining as per the recommendations of ISO 105 method.

ii) Fastness to sunlight :

The apparatus used for testing of fastness to sunlight was the exposure rack. The fastness to sunlight was tested as per the test method IS : 686-1985 prescribed by the Bureau of Indian standards. The printed design to be tested was wrapped in parellel lengths closely on a card to make a test specimen of area not less than 1 cm x 6 cm.

The test specimen were mounted in a exposure rack in such a way that half of each specimen was covered and other half was exposed to light. The samples were exposed to day light every day from sunrise to sunset, keeping the exposure rack at an angle of 45^{0} and face the north east direction.

For evaluating the light fastness, test specimen were exposed for 48 hrs. The change in colour of the test specimen was assessed with the help of grey scale No. 1.

iii) Fastness against rubbing :

Fastness to rubbing means the resistance of textile material to every type of rubbing and staining from textiles in actual use. The

apparatus used for testing the rubbing fastness was the crockmeter manufactured by paramount. Two types of rubbing fastness tests, dry and wet was carried out.

The fastness of rubbing was carried out according to IS:766-1988 method as given below :

The printed samples of size 25 cm x 10 cm were taken for carrying out dry as well as wet rubbing tests.

a) Dry rubbing :

Took one test sample and fixed to the rubbing device of crockmeter. The undyed cotton sample of size 2 x 2 inches was fixed to the finger of rubbing device of crockmeter. The test specimen was rubbed to and fro with the undyed piece with a downward force of 900 g. in a straight line, along a track of 10 cms for 10 times in 10 seconds. The test specimens were got evaluated for the change in colour and staining using a grey scale.

b) Wet rubbing :

A fresh (dry) test piece was fixed to the rubbing device and soaked the fresh piece of undyed cotton sample of size 2 x 2 inches in distilled water, followed by squeezing (drawn between two rubber rollers, squeezed by means of rubber roller on a glass plate) to remove excessive water. Thus the samples were subjected to rubbing test. Dried the wet piece at room temperature. The evaluation for the degree of staining and change in colour was done in similar manner as described for the dry rubbing test.

CHAPTER-IV

RESULTS AND DISCUSSION

The results of this study have been explained under the following sub-sections.

1. Preliminary data of fabric
2. Standardization of printing paste
   - i) Optimization of dye extraction pH
   - ii) Optimization of dye paste
   - iii) Optimization of pH of guar paste
   - iv) Optimization of dye paste and guar paste ratio
   - v) Optimization of fixer concentration
   - vi) Selection of mordants
   - vii) Optimization of mordant concentration
   - viii) Optimum variables of printing paste
   - ix) Colours obtained with the printing
3. Colour fastness properties of printed samples

1. PRELIMINARY DATA OF FABRIC USED

The preliminary data of the cotton fabric used for the present study indicated that the fabric count was 62x57. Weight per unit area was 104 gm per sq. meter and thickness of fabric was 0.333 mm.

2. STANDARDIZATION OF PRINTING PASTE

The present investigation was carried out to explore the usefulness of three vegetable sources i.e. marigold flower, rose flower, peepal bark for printing cotton fabric and to obtain a wide range of colours using different mordants. Thus experiments were conducted to optimize the extraction pH, dye paste, pH of guar paste, dye paste and guar paste ratio, fixer concentration and mordant concentration. The data for the same is presented in Table 4-16 and Plate 5-19.

i) Optimization of dye extraction pH :

The pH which gives the best colours is the optimum pH for dye extraction. The optimum pH for dye extraction was found out by extracting selected dyes at 5 different pH values viz. 5,6,7,8,9 and samples were printed.

It is evident (Table 4) that the best colour for marigold flowers was obtained at pH 7 (3.0) followed by printed samples in the reducing order of pH 8 (2.6), pH 9 (2.4), pH 6 (2.2) and pH 5 (1.8). The samples printed with rose flowers extracted at pH 8 (3.0) was assessed best followed by pH 9 (2.4), pH 7 (2.0), pH 6 (1.8) and pH 5 (1.2). The results of peepal bark printed samples also highlighted that at pH 8 (3.0) the colour was excellent followed by pH 9 (2.8), pH 7 (2.0), pH 6 (2.0) and

Optimization of Extraction pH

pH	Marigold	Rose	Peepal
[5]			
[6]			
[7]			

Plate No. 5

Contd.....

Optimization of Extraction pH

pH	Marigold	Rose	Peepal
[8]			
[9]			

Plate No. 5

pH 5 (1.8). Hence marigold dye was found best when extracted at 7 pH, rose and peepal dye were adjudged best when extracted at 8 pH (Plate 5).

Table 4 : Optimization of dye extraction pH.

Sr. No.	Dye extraction pH	Weighted mean score		
		Marigold flowers	Rose flowers	Peepal bark
1.	5	1.8	1.2	1.8
2.	6	2.2	1.8	2.2
3.	7	**3.0**	2.0	2.0
4.	8	2.6	**3.0**	**3.0**
5.	9	2.4	2.4	2.8

It can thus be concluded that best colours were obtained at neutral pH or at slightly alkaline pH. Rose (2002), reported that the pH for extraction from all the selected materials dyes was alkaline.

II. Optimization of dye paste:

A dye paste was prepared in order to achieve printing with sharp outlines. The dye solutions were concentrated to 10 ml 'A', 7.5 ml 'B', 5 ml 'C' i.e. dye paste A, B and C and mixed with guar paste in ratio 1:5 to print the samples.

The results (Table 5) highlighted that the excellent sharp outlines were found when dye paste 'B' was used, scoring 3.0 followed by 'A' dye paste scoring 1.8 and 'C' dye paste scoring 1.6 for marigold. The outlines of the rose were excellent with 'A' paste (3.0) followed by 'B'

Optimization of Dye Paste

Dye Paste (ml)	Marigold	Rose	Peepal
[10]			
[7.5]			
[5]			

Plate No. 6

Optimization of pH of Guar Paste

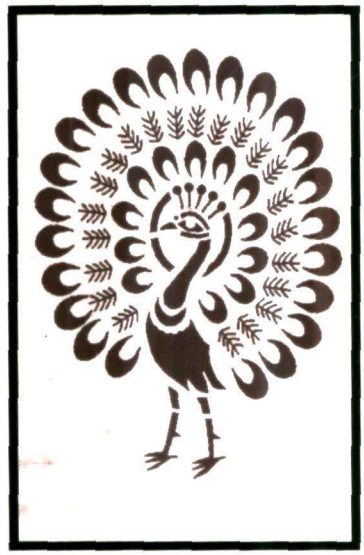
pH	Marigold	Rose	Peepal
[4]			
[6]			
[8]			

Plate No. 7

Optimization of Dye Paste & Guar Paste Ratio

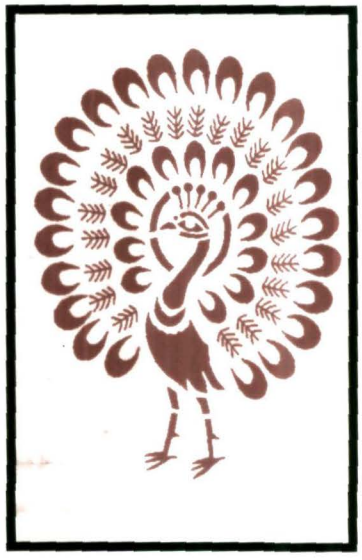
Ratio	Marigold	Rose	Peepal
[1:4]			
[1:5]			
[1:6]			

Plate No. 8

Optimization of Fixer Concentration

Fixer Conc. (%)	Marigold	Rose	Peepal
Vithout fixer			
[0.5]			
[1.0]			

Plate No. 9

Optimization of Fixer Concentration

Fixer Con. (%)	Marigold	Rose	Peepal
[1.5]			
[2.0]			

Plate No. 9

Selection of Mordants

Marigold

Alum

Chrome

Copper Sulphate

Ferrous Sulphate

Lead Acetate

Stannous

Zinc Chloride

Plate No. 10

Selection of Mordants

Peepal

Alum

Chrome

Copper Sulphate

Ferrous Sulphate

Lead Acetate

Stannous

Zinc Chloride

Plate No. 12

printed samples mordanted with other mordants ranged from 3/4-2/3 i.e. fairly good to poor.

The washing fastness of peepal dye with copper sulphate was good (4) and with zinc chloride and lead acetate was fairly good (3/4) but there was very little staining (4/5). The colour change with other mordants when printed with peepal ranged from 3/4-2/3 i.e. fairly good to poor.

The results of visual assessment and washing fastness depicted that for the marigold dye chrome and copper sulphate mordants were selected. The rose dye when mordanted with chrome and ferrous sulphate gave the best results.The printed samples of peepal dye with zinc chloride and copper sulphate, inferred the best (Plate 10, 11, 12).

There was a change in colour in printing with natural dyes with change of mordants. Like that of dyeing with natural dyes, wide range of colour obtained by using different mordant with a single natural dye as it has been reported by different authors (Bhattacharya 1999, Katyayini 1999, Kalyani 1999, Vastrad 1999, Vankar 2000, Moses 2000, Rose 2002). The fixation of colour was also improved with mordants in dyeing reported by Prabhu (1998), Moses (2000).

VII. Optimization of mordant concentrations :

To optimize the mordant concentration for each dye were tried at different concentrations viz. 3, 5 and 7 per cent.

Optimization of mordant concentration of marigold flowers :

The data (Table 11) divulged that the washing fastness for colour staining of marigold printed samples when mordanted with chrome

Optimization of Mordant Concentration

Marigold

Copper Sulphate (3%)

Chrome (3%)

Copper Sulphate (5%)

Chrome (5%)

Copper Sulphate (7%)

Chrome (7%)

Plate No. 13

and copper at 3 and 5 per cent were 5. So, the colour staining was negligible.

The washing fastness grades for colour change of marigold when mordanted with 3 and 5 per cent chrome indicated very good results (4/5), followed by 7 per cent chrome indicating 3/4 mordanting of the same paste with 3 per cent copper sulphate had very good fastness (4/5), followed by 3 and 7 per cent (4). Hence it has been inferred that optimum concentration of chrome and copper sulphate mordants were 3 and 5 per cent respectively (Plate 13).

Table 11 : Optimization of mordant concentration for marigold flower.

Sr. No.	Mordant	Percent mordant conc.	Washing fastness grades	
			CC	CS
1.	Chrome	**3**	**4/5**	**5**
		5	4/5	5
		7	3/4	5
2.	Copper sulphate	3	4	4/5
		5	**4/5**	**5**
		7	4	5

CC : Colour change; CS : Colour staining

Optimization of mordant concentration for rose flower

The results in Table 12 indicated that the washing fastness for colour staining of rose printed samples when mordanted with chrome showed no staining (5) and mordanted with copper sulphate exhibited little staining (4-4/5).

Optimization of Mordant Concentration

Rose

Chrome (3%)

Ferrous Sulphate (3%)

Chrome (5%)

Ferrous Sulphate (5%)

Chrome (7%)

Ferrous Sulphate (7%)

Plate No. 14

Table 12 : Optimization of mordant concentration for rose flower.

Sr. No.	Mordant	Percent mordant conc.	Washing fastness grades	
			CC	CS
1.	Chrome	3	4	5
		5	**4/5**	**5**
		7	4	5
2.	Ferrous sulphate	**3**	**4**	**4**
		5	3/4	4/5
		7	2/3	4/5

CC : Colour change; CS : Colour staining

The washing fastness grade for colour change of rose when mordanted with 5 per cent chrome depicted very good fastness (4/5) followed by 3 and 7 per cent chrome (4). The samples when mordanted with 3 per cent ferrous sulphate indicated good fastness (4), followed by 5 and 7 per cent showing fair to fairly good fastness (3/4-2/3). Thus it has been concluded that the optimum concentration of chrome and ferrous sulphate mordants were 5 and 3 per cent respectively (Plate 14)

Optimization of mordant concentration for peepal bark

The Table 13 indicates that the colour staining of peepal printed sample when mordanted with zinc sulphate and copper sulphate at all three concentration ranged from 4-4/5.

The data elucidated that the washing fastness for colour change of peepal bark when mordanted with 5 and 7 per cent zinc chloride showed fairly good results (3/4) followed by 3 per cent mordant fair (3). The mordanting with 7 per cent copper sulphate depicted good

Optimization of Mordant Concentration

Peepal

Zinc Chloride (3%)

Copper Sulphate (3%)

Zinc Chloride (5%)

Copper Sulphate (5%)

Zinc Chloride (7%)

Copper Sulphate (7%)

Plate No. 15

results (4) followed by 5 and 3 per cent as it ranged from 3-3/4. The results depicted that the optimum concentration of zinc chloride and copper sulphate were 5 and 7 per cent (Plate 15).

The results are in accordance with the findings of Prabhu (1998), Gogoi (1999), Vankar (2000) who also reported that the mordants at different percentages show better results with respect to colour fastness properties in dyeing.

Table 13 : Optimization of mordant concentration for rose flower.

Sr. No.	Mordant	Percent mordant conc.	Washing fastness grades	
			CC	CS
1.	Zinc chloride	3	3	4/5
		5	3/4	4/5
		7	3/4	4
2.	Copper sulphate	3	3	4/5
		5	3/4	4/5
		7	4	4/5

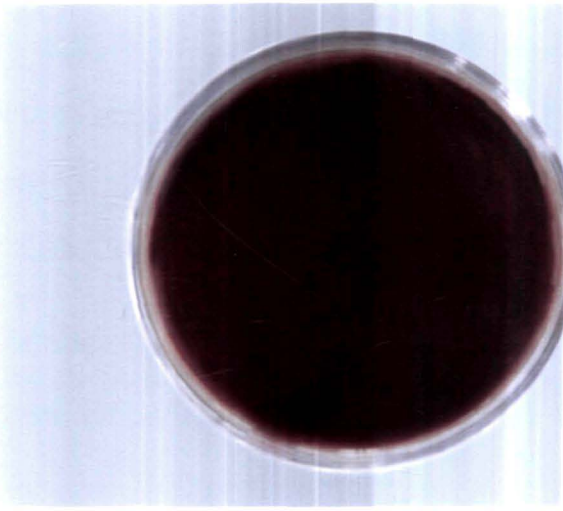
CC : Colour change; CS : Colour staining

Higher concentration of mordant were not selected due to their non-ecofriendly nature, however, further increase in mordant concentration added to brighter colour.

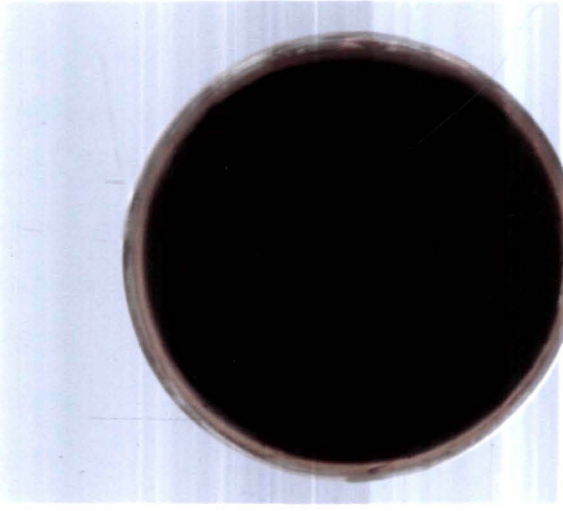
Optimum variable of printing paste for printing :

To optimize the printing paste (Plate 16) of marigold flower 6 gm dye concentration was extracted at pH 7 for 45 min, the dye solution obtained after extraction was concentrated to 7.5 ml to make the dye paste. Guar paste at pH 6 showed the best results in case of marigold.

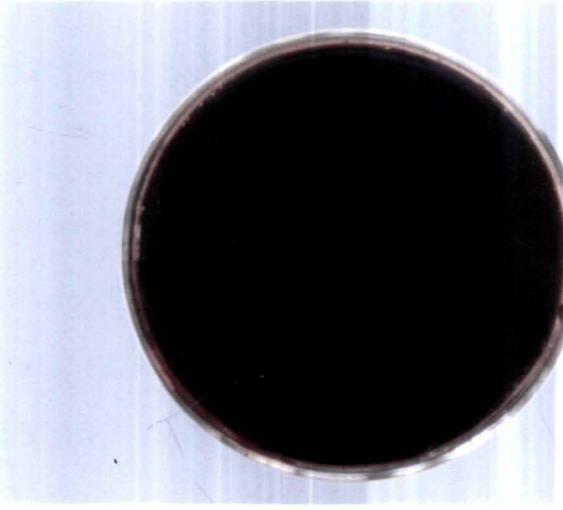
Optimum Printing Pastes of Marigold



Without Mordant



Copper Sulphate



Chrome

Plate 16

The dye paste and the guar paste was mixed in the ratio 1:5. The optimum per cent fixer (1.5%) was added to above paste on the basis of OWPP. Two mordants, chrome and copper sulphate were selected and their optimum concentration was 3 and 5 per cent respectively on the basis of OWPP.

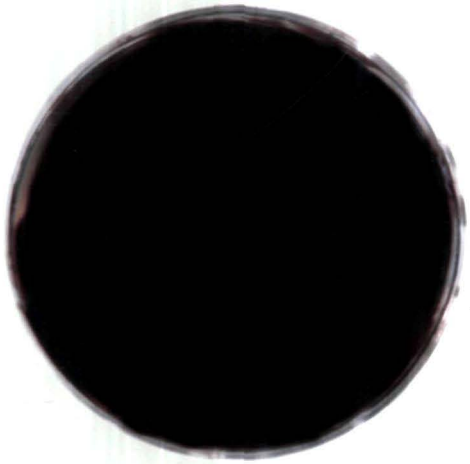
Table 14 : Optimum variables of printing paste.

Sr. No.	Optimum conditions	Marigold flower	Rose flower	Peepal bark
1.	Dye material concentration (%)	6	10	10
2.	Extration (min.)	45	15	30
3.	Extraction pH	7	8	8
4	Dye paste (ml)	7.5	10	5
5.	pH of guar paste	6	6	4
6.	Dye paste & guar paste ratio	1:5	1:5	1:5
7.	Fixer conc. (%)	1.5	1.5	1.5
8.	Mordants	Chrome	Chrome	Zinc chloride
		Copper sulphate	Ferrous sulphate	Copper sulphate
	Mordant Conc. (%)	3	5	7
		5	3	5

The printing paste of rose flower (Plate 17) was standardized using optimum dye concentration 10 g extracted at pH 8 for 15 minutes The dye solution obtained after extraction was reduced to 10 ml to make the dye paste. The best results in case of rose were obtained at pH 6 of guar paste. The dye paste and the guar paste was mixed in the ratio

Optimum Printing Pastes of Rose

Without Mordant

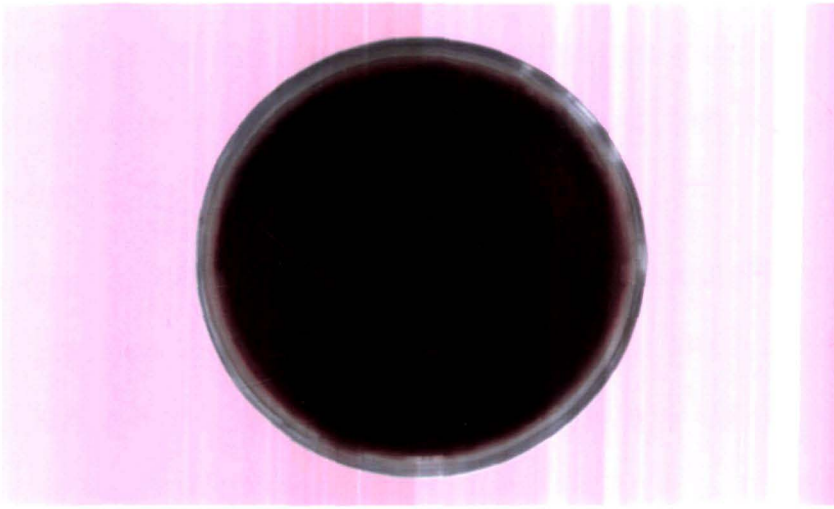


Chrome

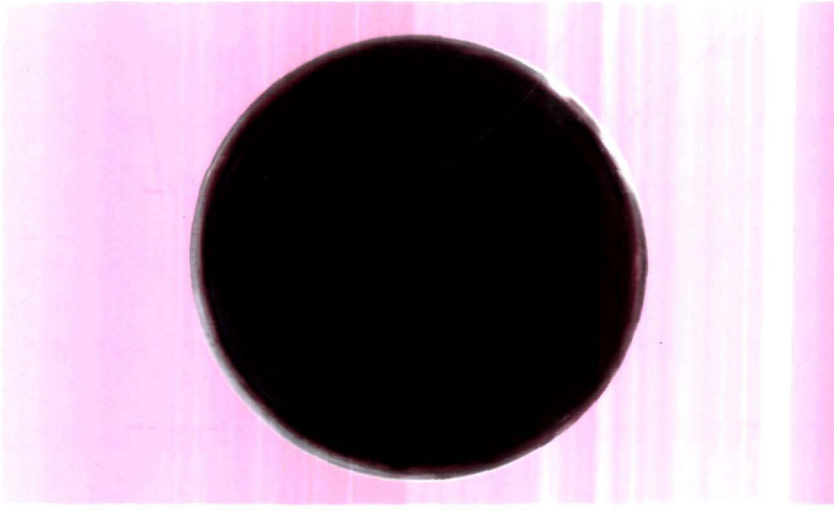
Ferrous Sulphate

Plate 17

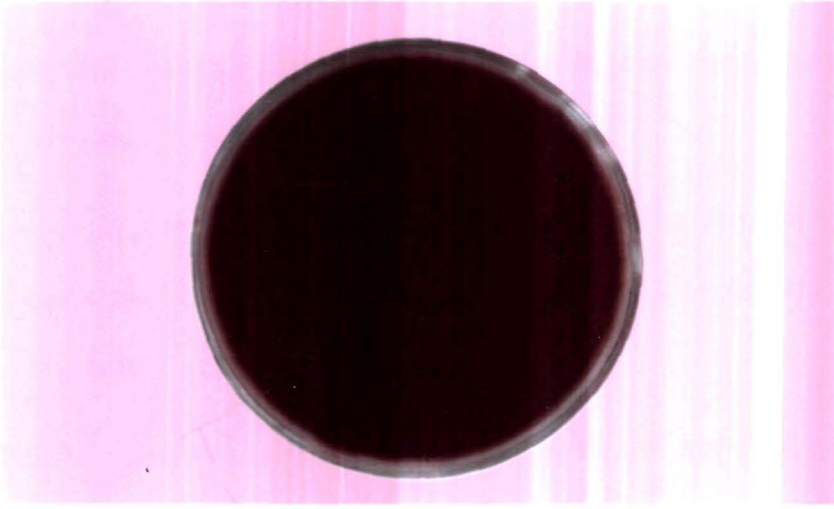
Optimum Printing Pastes of Peepal



Without Mordant



Copper Sulphate



Zinc Chloride

Plate 18

1:5. The optimum per cent fixer (1.5%) was added to the above paste on the basis of O.W.P.P. Two mordants, chrome and ferrous sulphate were selected and their optimum concentration was 5 and 3 per cent respectively on the basis of OWPP.

To optimize the printing paste of peepal bark (Plate 18), 10 gm dye concentration was extracted at pH 8 for 30 minute the dye solution obtained after extraction was concentrated to 5 ml to make the dye paste. Guar paste of pH 4 depicted showed the best results in case of peepal printed samples. The dye paste and the guar paste was mixed in the ratio 1:5. The optimum per cent fixer i.e. 1.5 per cent was added to above paste on the basis of O.W.P.P. Two mordants, copper sulphate and zinc chloride were selected and their optimum concentration was 5 and 7 per cent respectively on the basis of OWPP.

Colours obtained with selected vegetable dyes

Various colours were obtained with selected vegetable dyes when used with different mordants (Table 15) (Plate 19). Colours obtained with marigold flowers without mordant was silky Beige and when mordanted with copper sulphate and chrome produced were olive green and mustard colours resectively.

The rose flowers without mordant gave autumn gold colour. Colours obtained using chrome were coffee brown and black with furrous sulphate.

Shades obtained with peepal bark without mordant was tea rose when mordanted with copper sulphate deep rose and with zinc chloride golden brown colours were obtained .

Colors Obtained Using Optimum Printing Variables

Marigold	Rose	Peepal
		
Without Mordant	Without Mordant	Without Mordant
		
Chrome	Chrome	Zinc Chloride
		
Copper Sulphate	Ferrous Sulphate	Copper Sulphate

Plate No. 19

The results of the present study highlighted that there was change in colours of prints with the change of mordants. It has also been reported in the researches conducted by Goel and Chauhan (1996) Gupta and Goel (1997), Goel and Goel (1997).

Table 14 : Colours obtained with selected vegetable sources.

Sr. No.	Vegetable Dye	Mordant	Percent mordant concentration	Colours obtained
1.	Marigold flower	Without mordant	---	Silky Beige
		Chrome	3	Mustard
		Copper sulphate	5	Olive green
2.	Rose flower	Without mordant	---	Autumn gold
		Chrome	5	Coffee brown
		Ferrous sulphate	3	Black
3.	Peepal bark	Without mordant	---	Tea rose
		Zinc chloride	3	Golden brown
		Copper sulphae	7	Deep rose

Colour fastness grades of printed samples using optimum printing paste :

The ability of a dye in association with a given substrate to withstand the various agencies such as washing, sunlight and crocking, etc in processing or use is called its fastness properties. The Table

depicted the colour fastness properties of the samples printed with standardized printing paste of all the three dyes.

i) Washing fastness grades :

The colour fastness grades to washing of cotton printed samples of the selected dyes using different mordants are presented in Table 16. The results are being expressed in terms of colour change and staining of adjacent cotton and woollen material.

The result revealed that the washing fastness grades of marigold flowers for colour staining was 5 with both the mordants (Chrome and copper sulphate). Hence, it was depicts that there was no staining. The grades for colour change ranged from 4-4/5. But after washing the change was towards brighter side. Hence indicating good to very good washing fastness but brighter shades were obtained.

The washing fastness grades of rose petals for the colour staining ranged from 4-5. Hence, it is perceived that there was little or no staining. The grades for colour change ranged from 3/4 - 4/5 thus result throws light that the fastness against washing of rose dye was fairly good to very good and also when mordanted with chrome showed more brighter results after washing. Goel and Chauhan (1996) reported that mordanted printed samples after washing showed colours faster and brighter.

The results of peepal bark for colour staining in case of peepal was 4/5 indicating very little staining. The grades for colour change ranged from 3/4-4. Thus, it is inferred that the washing fastness for colour change was fairly good to good.

Sunlight fastness :

The results highlighted (Table 11) that the light fastness was good to excellent for all the three dyes as their score ranged from 4-5. Thus, it can be concluded that colours were fast to sunlight.

ii) Rubbing fastness :

The finding of colour fastness tests of cotton printed samples with selected dyes using different mordants to rubbing are given in Table 16.

a) Dry rubbing fastness :

It is evident that the dry rubbing fastness for colour change and colour staining ranged from 4/5-5 for marigold and rose printed samples, while for peepal printed sample the grades for colour change and colour staining were 5. Hence, the result elucidated that the dry rubbing fastness was very good to excellent for marigold and rose flowers and excellent for peepal bark.

Wet rubbing fastness :

The results of colour fastness of printed samples with selected dyes using different mordants to wet rubbing is depicted in Table 16.

Thus the data throws light that the wet rubbing fastness of marigold dye was good for colour change (4) while good to very good for colour staining (4-4/5). The colour change of rose printed samples were also fairly good to very good (3-4/5) while colour staining ranged from very good to excellent 4/5-5 and for peepal, good (4) in terms of colour change and very good to excellent (4/5-5) for colour staining.

Table 16 : Colour fastness grade of samples printed using optimum printing paste

Veg. dyes	Mordant	Per cent mordant conc.	Colour fastness grades						
			Wash		Light	Rubbing			
						Dry		Wet	
			CC	CS		CC	CS	CC	CS
Marigold	Without mordant		3	4/5	4/5	4	3/4	4/5	4/5
	Chrome	3	4/5*	5*	4/5	5	4/5	4	4/5
	Copper sulphate (5)	5	4*	5*	5	4/5	4	4	4
Rose	Without mordant		3	3/4	4	3/4	3	3	4
	Chrome	5	4/5*	5*	5	5	4/5	4/5	4/5
	Ferrous sulphate	3	3/4	4	4/5	4/5	3	3	5
Peepal	Without mordant		2/3	3/4	4	4	3	3	4/5
	Zinc chloride	5	3/4	4/5	5	5	4	4	5
	Copper sulphate	7	4	4/5	5	5	4	4	4/5

* Brighter than original
CC : Colour change; CS : Colour staining

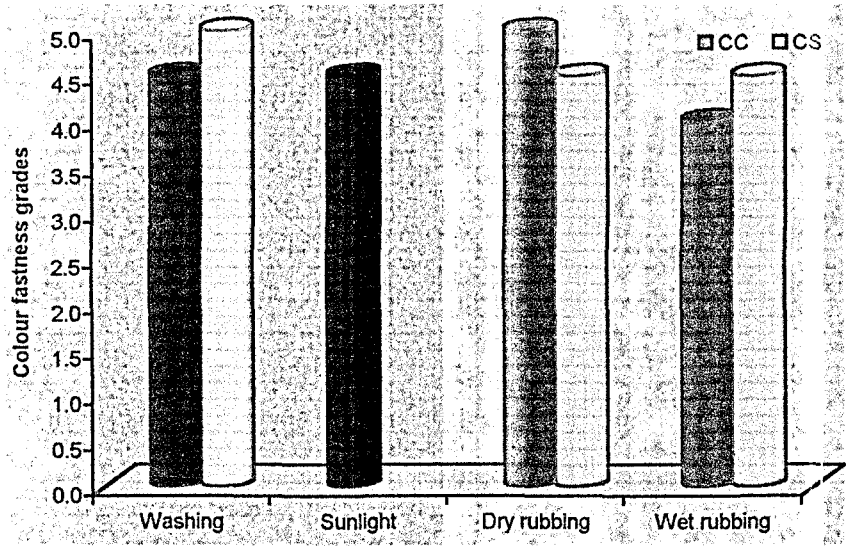


Fig. 1 : Colour fastness grades of chrome mordanted marigold printed samples

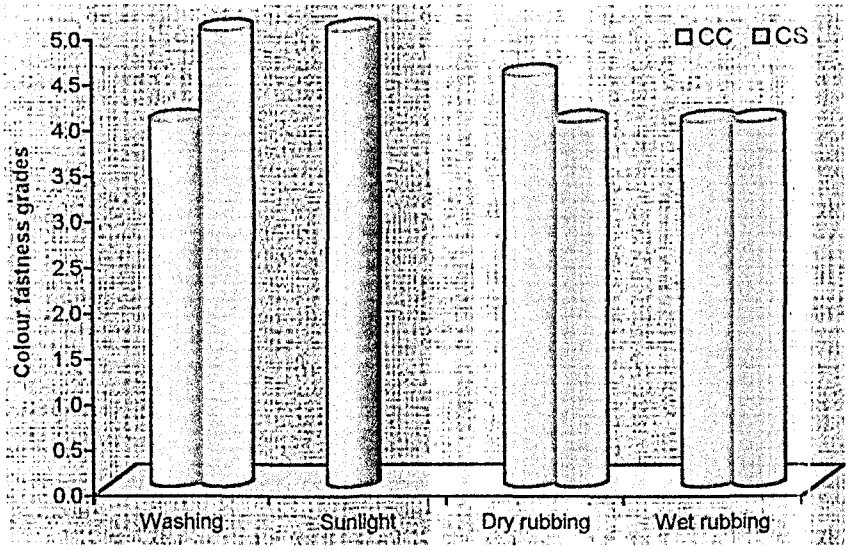


Fig. 2 : Colour fastness grades of copper sulphate mordanted marigold printed samples

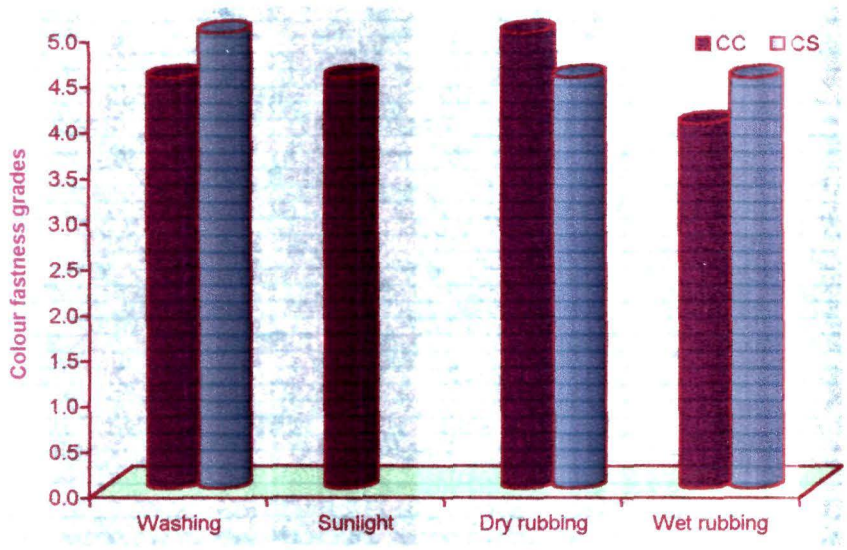


Fig. 1 : Colour fastness grades of chrome mordanted marigold printed samples

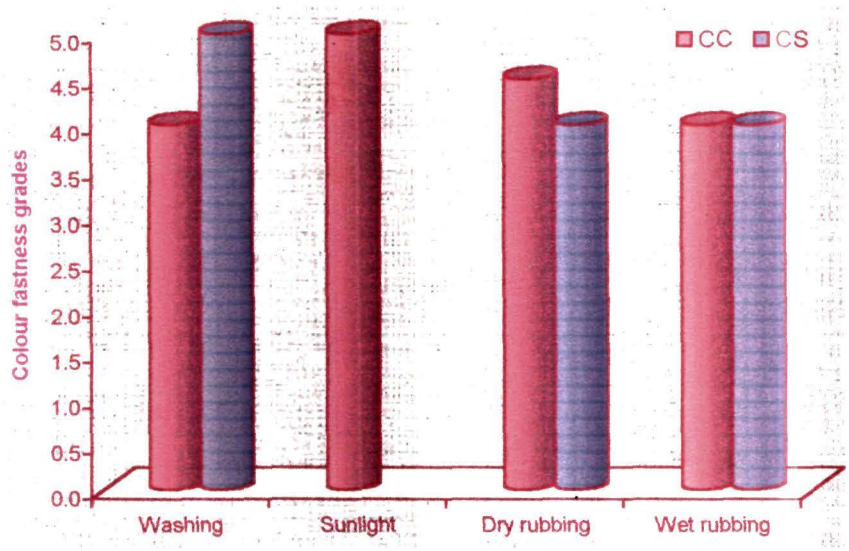


Fig. 2 : Colour fastness grades of copper sulphate mordanted marigold printed samples

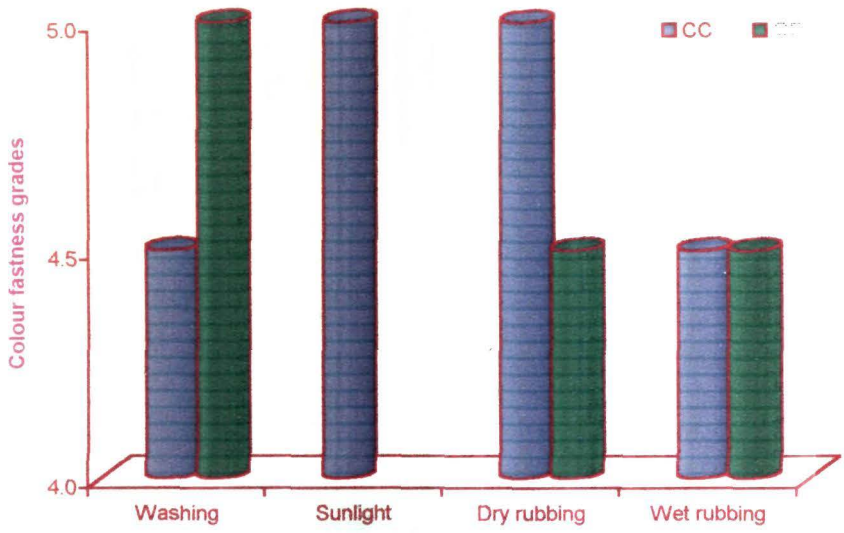


Fig. 3 : Colour fastness grades of chrome mordanted rose printed samples

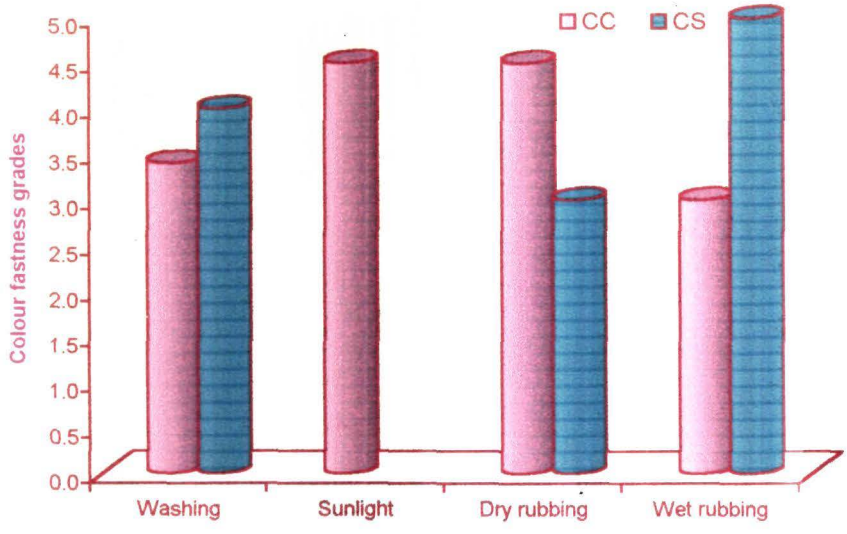


Fig. 4 : Colour fastness grades of ferrous sulphate mordanted rose printed samples

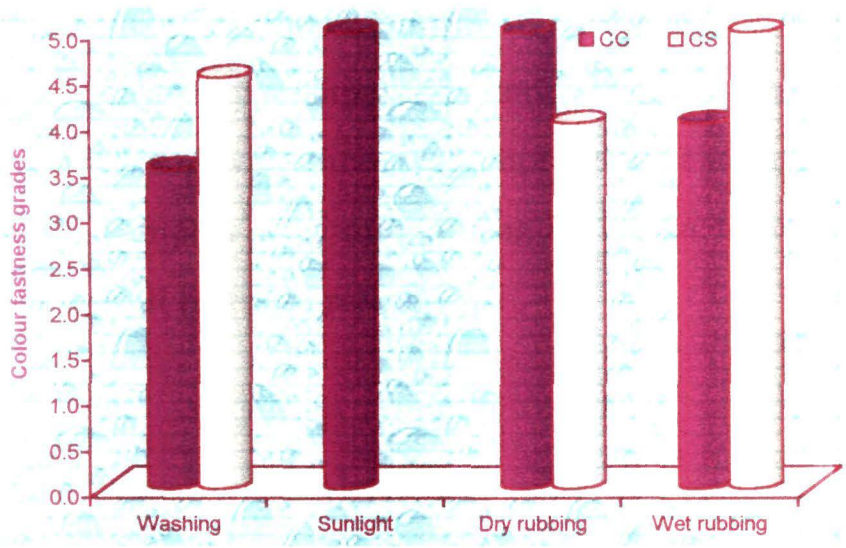


Fig. 5 : Colour fastness grades of zinc chloride mordanted peepal printed samples

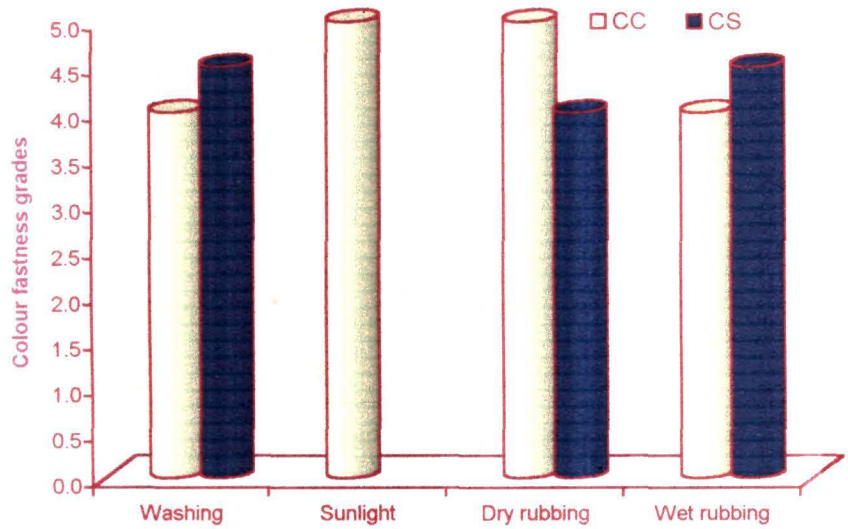
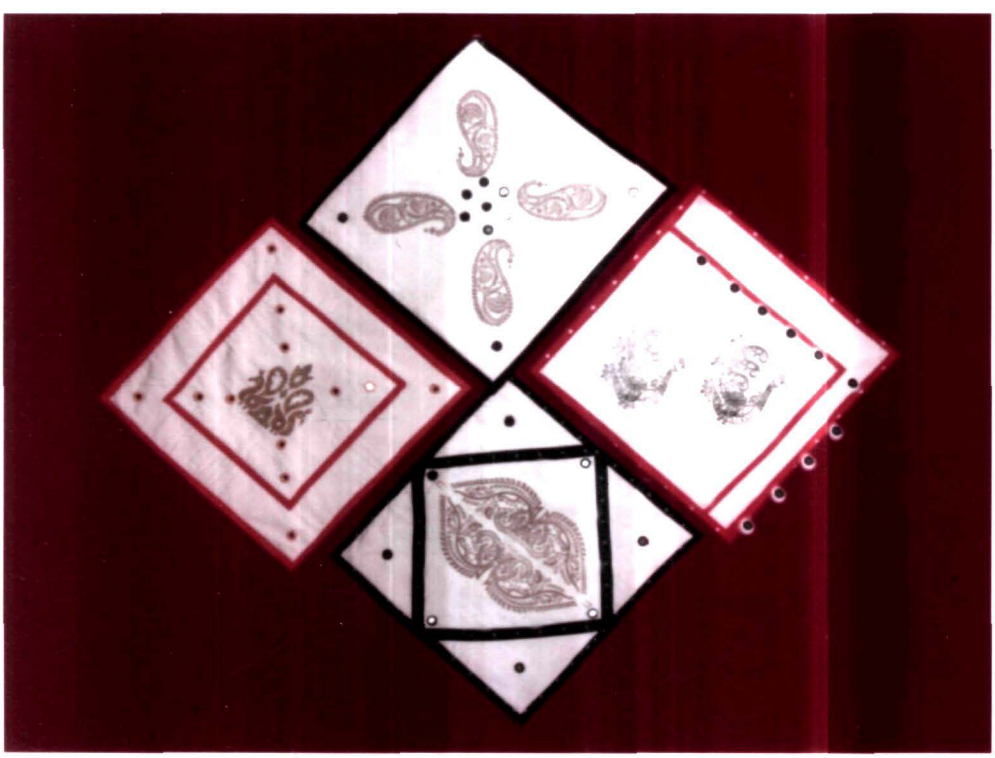


Fig. 6 : Colour fastness grades of copper sulphate mordanted peepal printed samples

Chindwade et al. (1997), Gogoi (1999) and Devi et al. (2000) reported that mordanted samples showed good fastness peroperties for all agencies when compared to control samples during dyeing with natural dyes. Likewise there was improvement in fastness properties of printed samples after mordanting as it can be concluded on the basis of overall fastness rating.

VALUE ADDED PRODUCTS

Kurta



Cushion Covers

Plate No.20

CHAPTER-V

SUMMARY AND CONCLUSION

Printing is a process of producing designs on textile fabrics using one or more dyestuffs. It is also known as localized dyeing. The art of printing with natural dyes has played an important role in beautifying the textiles, since time immemorial. But with the introduction of synthetic dyes the use of natural dyes gradually went out of existence because of the fact that brilliant colour and shades with good fastness properties could be produced with synthetic dyes. However the production of synthetic dyes involves carcinogenic chemicals which are highly hazardous. On the other hand natural dyes are nonhazardous, biodegradable and have better compatibility with environment. Hence natural dyes are back in vogue due to ecological consideration. In near future the demand for the fabrics printed with natural dyes in likely to increase not only due to safety and healthy environment but also for their beauty and novelty. A lot work has been done and still being carried out in different parts of country on exploring the sources for natural dyes, but their use in printing has not much

CHAPTER-V

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been explored. With this view the endeavour was made to apply the natural dyes for cotton printing with the following objective :

Objective of the study :

1. To standardize the recipe of printing paste for printing cotton fabric with selected natural dyes.
2. To study the effect of mordants on the prints obtained with selected natural dyes.
3. To analyze the colour fastness of printed fabric.

Three vegetable dyes namely, marigold, rose and peepal were selected for printing on the basis of printing ability and colours obtained. The printing paste was standardized by optimizing dye extraction pH, dye paste, pH of guar paste, dye paste and guar paste ratio, fixer concentration and mordant concentration. During process of standardization the sample were printed, steamed and rinsed in cold water and got evaluated for optimization of each variables. Visual assessment was got done from five clothing and textiles experts to optimize dye extraction pH, dye paste and pH of guar paste whereas washing fastness assessment was done to optimize dye paste and guar paste ratio and mordant concentration. Colour fastness of printed samples to washing, sunlight and crocking was also studied.

The optimum dye material concentration taken for all the three dyes i.e. marigold, rose and peepal were 6, 10 and 10 per cent respectively. The dye material was extracted at five different pH (5,6,7,8,9) and pH 7 for marigold pH 8 for rose and peepal was optimized

on the basis of colours obtained. The marigold dye was extracted for 45 min, rose dye was extracted for 15 min and peepal dye was extracted for 30 min. The dye solutions obtained after extraction was concentrated to make the dye paste at 3 different volumes viz. 10 ml (A), 7.5 ml (B) and 5 ml (C). Dye paste 'A' (10 ml) for marigold, dye paste 'B' (7.5 ml) for rose and dye paste 'C' (5 ml) for peepal were optimized. Another component required for printing paste was thickener i.e. guar gum. The pH of guar paste was tried out at 3 different pH value i.e. 4, 6, 8 and the best colour of marigold, rose and peepal dyes were assessed at pH 6, 6 and 4 respectively. To make the printing paste, dye paste and guar paste were mixed in three different ratios i.e. 1:4, 1:5 and 1:6 and the best colour and sharp out lines for all the three selected dyes were adjudged at 1:5 dye and guar paste ratio. For optimizing the fixer concentration, samples were printed with four concentration viz. 0.5, 1.0, 1.5 and 2.0 per cent whereas fifth sample was without fixer. At 1.5 per cent fixer concentration all the three selected dyes showed the best results.

Seven different mordants were tried and on the basis of different shades obtained and washing fastness, two mordants were selected for each selected vegetable dye. The mordants selected for marigold dye were chrome and copper sulphate, for rose dye were chrome and ferrous sulphate and for peepal dye were zinc chloride and copper sulphate. Three concentrations i.e. 3, 5 and 7 per cent on the basis of weight of printing paste of all the selected mordants for each dye were tried. The washing fastness was very good when marigold printed

samples were mordanted with 5 per cent chrome and 3 per cent copper sulphate. In case of rose printed samples when mordanted with 3 per cent chrome and 5 per cent ferrous sulphate indicated good to very good results. Peepal printed samples with 5 per cent zinc chloride and 7 per cent copper sulphate gave fairly good to good results. Colours obtained with three different selected dyes were silky beige, mustard, olive green, autumn gold, coffee brown, black, tea rose, golden brown and deep rose. Hence, in total 9 shades were obtained.

The samples printed with standardized recipe were subjected to washing, light and crocking fastness.

The colour staining of washed samples of all the three dyes was either negligible or very little staining. The colour change grades were good to very good for marigold dye, fairly good to very good for rose dye and fairly good to good for peepal dye.

The light fastness for all the three selected dyes used for printing ranged from very good to excellent.

The fastness to dry rubbing of samples printed with peepal dye was excellent in terms of colour change and colour staining but very good to excellent for marigold and rose dyes. The wet rubbing fastness to colour change ranged from good to very good for marigold and rose, dyes, but fairly good to good for peepal dyes. The wet rubbing fastness to colour staining were good for marigold and peepal dyes fair to very good for rose dyes.

CONCLUSIONS

- The optimum dye material concentration for marigold flower was 6%, for rose flower and peepal bark was 10% and extractionof dye pH was 7, 8 and 8 and dyeing extraction time was 45, 15 and 30 min respectively.
- The dye extracted under optimum conditions was concentrate to 7.5 ml for marigold dye, 10 ml for rose dye and 5 ml for peepal dye.
- The optimum pH of guar paste for all the selected dyes were acidic.
- The optimum dye paste and guar paste ratio for all selected dyes was 1 : 5.
- The optimum fixer concentration for all the three dyes was 1.5 per cent.
- Different mordant produced their own different colours, which modified the hues produced by different dyes, thus producing a variety of shades and tints from a single dye.
- All the mordants improved the fastness properties of printed samples with natural dyes.
- The suitability of mordants for printing cotton with natural varied from dye to dye. Two mordants for each dyes were selected i.e. for marigold : chrome and copper sulphates for rose : chrome and ferrous sulphate and for peepal : copper sulphate and zinc chloride.

- The optimum mordant concentration was 3% chrome and 5% copper sulphate for marigold dye, 5% chrome and 3% ferrous sulphate for rose dye and 7% copper sulphate and 5% zinc chloride for peepal dye.
- The colour fastness for all the selected dyes was very good to excellent.

RECOMMENDATIONS

- Marigold flower, rose flower and peepal bark being eco-friendly can be effectively used for cotton printing on commercial scale.
- A wide range of shades with good fastness properties were obtained hence, it is recommended for clothing and upholstery items.
- The waste of these dyes is biodegradable.
- The shades and hues of the colours obtained from the natural dyes vary with the species and age of the plant, water conditions and temperature fluctuations. Hence it is recommended of the atmospheric conditions of the area from where the dye material is to be collected should be considered.

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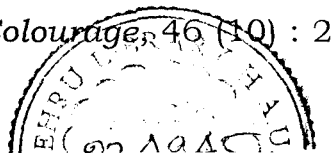

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APPENDIX-I

PLANT MATERIALS TRIED FOR THE STUDY

Sr. No.	English name	Common name	Botanical name	Part used
1.	Ashoka, mast tree	*Ashok*	Polyalthia longifolia	Bark
2.	Ashoka, mast tree	*Ashok*	Polyalthia longifolia	Leaves
3.	Banyan	*Bar, Bargad*	Ficus bengalensis	Bark
4.	Boungainvillaea	Boungainvillaea	Boungainvillaea sp.	Leaves
5.	Boungainvillaea	Boungainvillaea	Boungainvillaea sp.	Flowers
6.	Bathua	*Bathua*	Chenopodium album	Leaves
7.	Corriander	*Dhania*	Coriandrum sativum	Leaves
8.	Eucalyptas	*Sapheda*	Eucalyptus resinifera	Leaves
9.	Eucalyptas	*Sapheda*	Eucalyptus resinifera	Bark
10.	Heena	*Mahendi*	Lawsonia alba	Leaves
11.	Iron wood	*Kasud*	Cassia siamea	Leaves
12.	Jatropha	Jatropha	Jatropha indica	Flower
13.	Jatropha	Jatropha	Jatropha indica	Leaves
14.	Marigold	*Ganda*	Targetus erecta	Flowers
15.	Peacook flower flamboyant tree	*Gulmohar*	Delonix regia	Leaves
16.	Peacook flower flamboyant tree	*Gulmohar*	Delonix regia	Flowers
17.	Peepal	*Pipal*	Ficus religiosa	Bark
18.	Rose	*Gulab*	Rosa indica	Flower
19.	Yellow oleander	*Pili kaner*	Thevetia peruviana	Leaves
20.	Yellow oleander	*Pili kaner*	Thevetia peruviana	Flowers