CHAPTER 3
MATERIALS AND METHODS

The present study entitled '**Studies on computer aided color measurement, matching and compatibility of dyes for compound shades production on textiles using natural dyes'** was undertaken in the following steps. The experimental design is depicted using a schematic diagram in Figure 3.1.

3.1 Materials Used

The materials used for the study include fabric, chemicals, dyes, apparatus, and instruments. All the details for these are mentioned in this section.

3.1.1 Cotton Fabric

Ready for dyeing (R.F.D.) bleached 100% cotton cambric fabric was selected for this study. The fabric was procured from R.M.P. Fab Sourcing Pvt. Ltd., Faridabad, Haryana, India. The following tests were conducted for the fabric received.

Determination of Yarn Count and Construction

English count for the warp and weft was calculated using Beeseley balance. The sample was taken randomly, and the count was calculated. The warp count obtained was 62Ne, and the weft count was 58Ne.

The ends per inch and picks per inch were calculated using pick glass. The sample was taken randomly, and the reading was calculated. The result obtained was EPI * PPI – 102*82.

Determination of fabric GSM, Weave and Width

The gram per square meter (GSM) of the cotton fabric was calculated using the GSM cutter instrument. The samples of a diameter of approximately 113 mm and an area of 100 cm^2 were cut and weighted. The sample weight in grams was multiplied by 100 to get the GSM of the fabric. Three random samples were cut, and an average was calculated. The average GSM found was 78.

The weave of the sample was checked using a magnifying glass, and it was found that the fabric was made using plain weave. The width of the fabric was 52".

Fiber Identification, Absorbance and Whiteness Index

Fiber identification was done using appearance, burning, and chemical tests. The standard test method for all identification methods was used, and the material was found to be 100% cotton

The absorbance test of the fabric was done manually using the standard drop test method, and the material was found to be a good absorbent.

The Whiteness Index of RFD fabric was calculated using a spectrophotometer. The value of the whiteness index was found to be 77.67 in CIE-76/ 10 degrees in D65 illuminant.

Tensile Strength and Elongation Test

Tensile strength was measured using the ASTM D 5034 one-inch grab method, and it was found to be warp -23.5 kg and weft – 16.4 kg. The elongation at break as warp-8.9% and weft – 21.9%

3.1.2 Chemicals, Dyes and Auxiliaries

Citric Acid $C_6H_8O_7$ (anhydrous), Sodium Carbonate Na_2CO_3, Alum (Aluminium Potassium Sulphate) $KAl(SO_4)_2 \cdot 12H_2O$, and non-ionic detergent were used in this present work. All these were laboratory-grade chemicals from Fisher Scientific and Loba Chemie Pvt. Ltd. The details of dye materials used were as per Table 3.1 (A & B) and were procured from Jaipur.

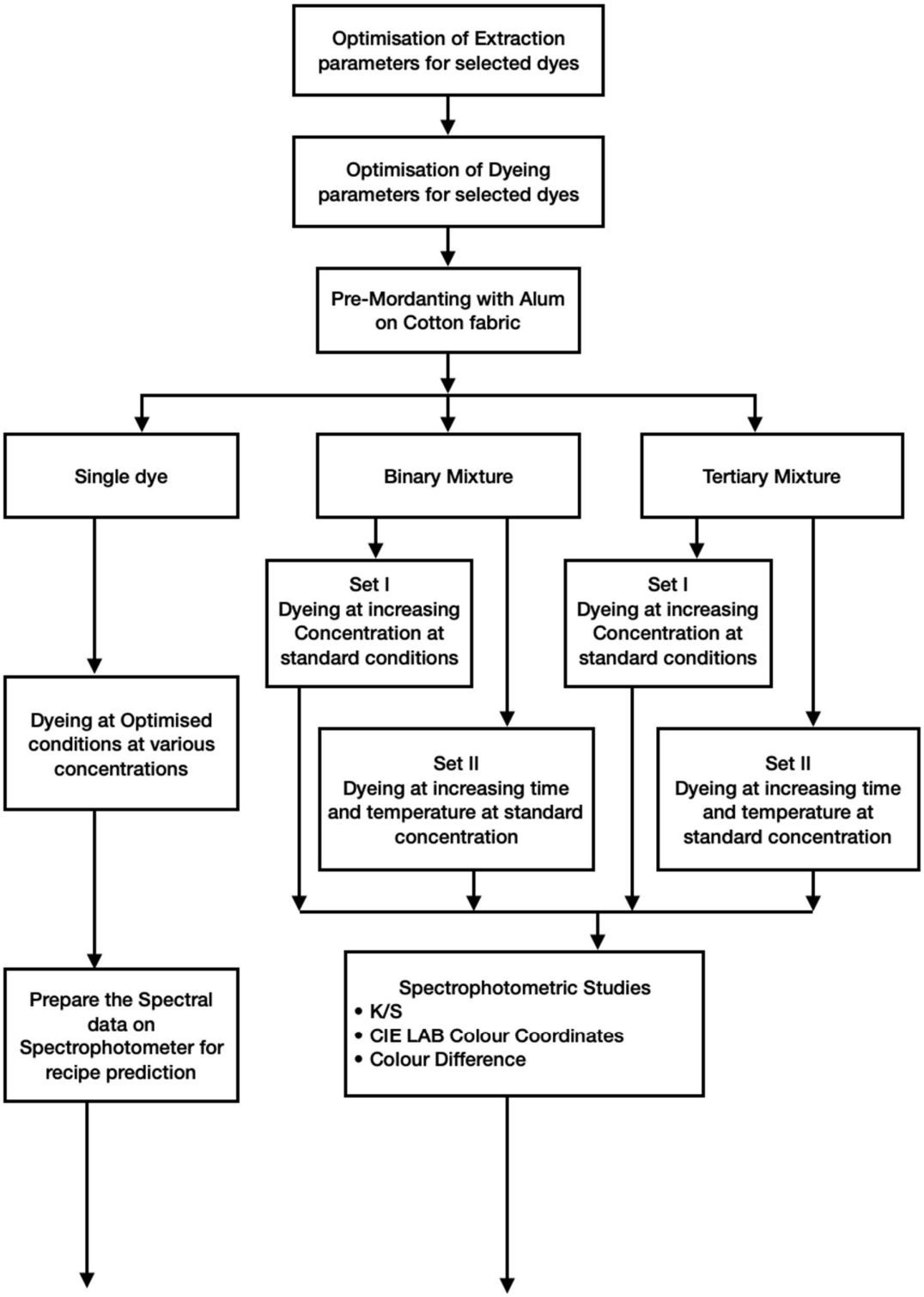
Table 3.1 (A) : Details of dyes used

S. No.	Common name	Hindi Name	Botanical name	Color used	Code Given
1	Madder	Manjistha	*Rubia tinctorum*	Red	D
2	Sappanwood	Pathangi	*Caesalpinia sappan*	Pink	S
3	Annatto	Sinduri	*Bixa orellana*	Orange	A
4	Marigold	Genda	*Tagetes erecta*	Yellow	M
5	Pomegranate	Anar	*Punica granatum L.*	Yellow	P
6	Catechu (called as Katha in this study)	Katha	*Acacia catechu*	Brown	K
7	Bark of Acacia (called as Babool in this study)	Babool	*Acacia nitotica*	Brown	B
8	Himalayan Rhubarb	Dolu	*Rheum emodi*	Brown	R

Table 3.1 (B): Details of dyes used

S. No.	Common name	Dye Classification based on application	Structure type	Main colouring Pigment	Some other compounds
1	Madder	Mordant / Disperse	Anthraquinone dyes	Alizarin	purpurin, xanthopurpurin, pseudopurpurin, rubiadin
2	Sappanwood	Mordant / Disperse	Anthocynin, homoisoflavonoid	Brazilin	Brazilein (oxidative form of braziline)
3	Annatto	Direct	Carotenoids	Bixin	Tannins
4	Marigold	Mordant	Carotenoid	Lutein	Flavnoid
5	Pomegranate	Direct	Anthocynins (Flavanoids)	Tannins (Punicagalin, Punicalin)	polyphenols, flavonoids, hydrolyzable tannins
6	Katha	Mordant / Disperse	Tannins	Catechin	ascorbic acid, riboflavin, thiamine, niacin, and carotenoids
7	Babool	Direct	Tannins /Flavonoid	gallic acid, ellagic acid	quercetin, isoquercetin, catechin, epicatechin
8	Himalayan Rhubarb	Mordant	Anthraquinone derivatives	Chrysophanic Acid	Rhein, emodin, aloe-emodin and chrysophanol

Schematic Diagram of Work Plan



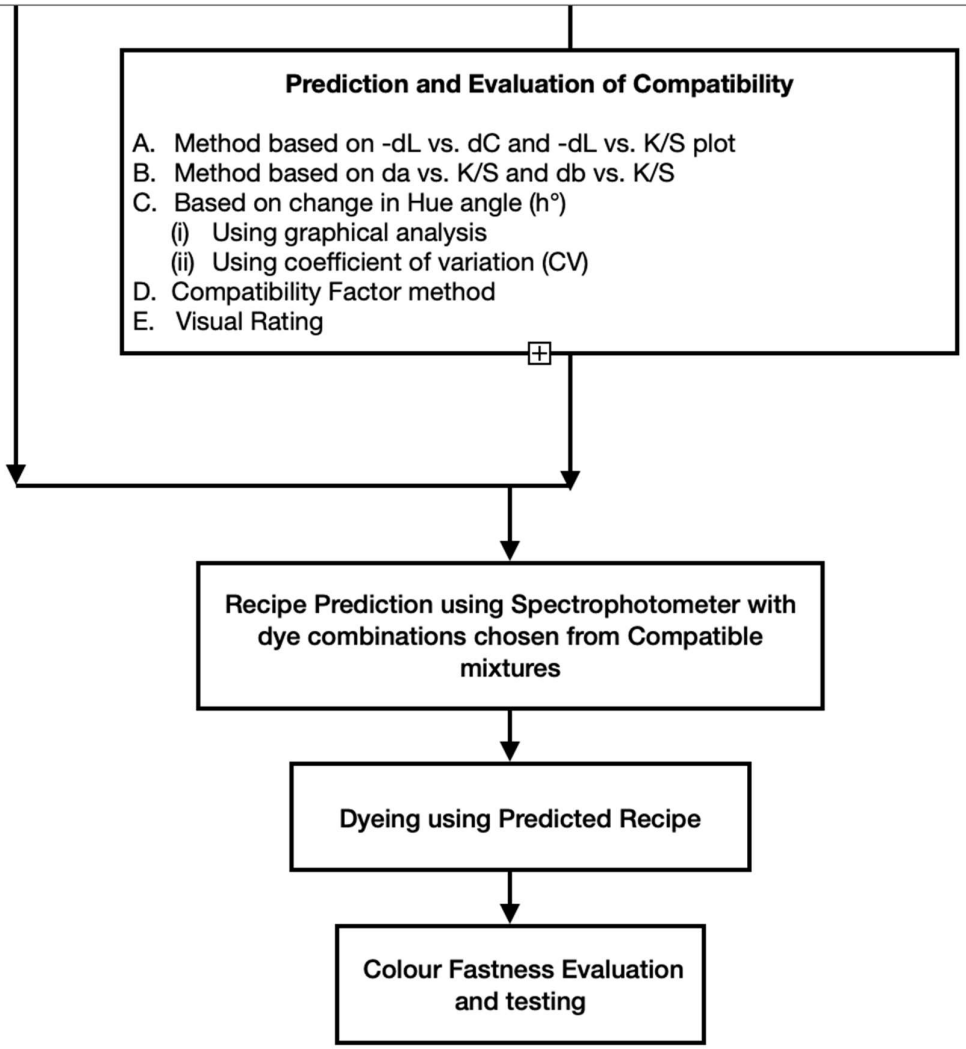


Figure 3.1: Schematic Diagram of Work Plan

3.1.3. Instruments and Apparatus

Electronic pH meter, Electronic Weighing Scale, HTHP Beaker Dyeing Machine; Make- R. B. Electronic & Engineering Pvt Ltd., Laundrometer and Crock meter; Make-Ramp Impex Pvt Ltd., Light fastness tester; Make - Innolab, Shimadzu Spectrophotometer for optical density, Konica-Minolta Spectrophotometer, model-3600d for dyed samples were used in this work.

3.2 Methods

This part includes a pilot run and an experimental study. The pilot run acted as the basis of experimental work, including optimizing the extraction and dyeing process. The experimental work includes the dyeing in mixture and recipe prediction work. All the details of the methods used are mentioned in this section.

3.2.1 Pilot Study

The pilot study was done to find the optimum extraction and optimum dyeing conditions. The optimum extraction conditions were used to get the maximum color extraction for all samples dyed in the present study. The second part of the pilot-run study was to find the optimum dyeing conditions for each dye. The optimum dyeing conditions were then used for experimentation. This section explains both the optimization processes.

Optimization of Extraction Parameters for all the selected dyes

Dye material for Marigold, Pomegranate, Madder, Annatto, Babool (*Acacia nilotica*), Sappanwood, Rhubarb, and Katha (Catechu) dye was taken for extraction optimization. The optimization was done using a range of temperature, time, and MLR as per Table 3.2.

Table 3.2: Experimental setup for Extraction Optimization

Parameter	Trail	Control Condition
MLR	1:10	Temperature -80°C, Time-60 mins
	1:20	
	1:30	
Temperature (°C)	60	MLR-1:20, Time - 60 mins
	80	
	100	
Time (min)	30	MLR-1:20, Temperature -80°C
	60	
	90	

The extracted solution was measured using a spectrophotometer to obtain the absorbance value / optical density. Optical density was used to find the maximum color extract from each dye; hence, the optimized parameters for extraction were found. The optimized parameters for respective dyes were then used to extract and further dye cotton fabric.

Optimisation of Dyeing Parameters

Box Behnken's design of experiment was used for the experimental setup as indicated in Table 3.3. 17 samples were dyed for each dye, with a total of 136 samples for all 8 dyes to find the optimum dyeing condition for each dye.

Dye extraction was done as per requirement at optimum extraction conditions. Ready for dyeing (R.F.D.), bleached cotton cambric fabric samples were mordanted using alum (5% owf) with M.L.R. – 1:60, temperature – 90°C for 60 mins. After the complete process, the samples were rinsed twice to remove the excess alum particles on the surface to prepare them for dyeing.

Table 3.3: Experimental Setup for Optimization of Dyeing Parameters

Control Factors	
Parameter	**Values**
pH	3,5,7
Temperature (°C)	65, 80, 95
Time (min)	30, 60, 90
Response Variable: Surface Color Strength (K/S)	

The dyeing of pre-mordanted samples was done at M.L.R. 1:90 with 30% owf extracted dye material at a range of pH, time, and temperature. The dyeing conditions for all 17 samples are mentioned in Table 3.4. The dye solution was prepared as per the recipe, and the mordanted sample was dipped into it. The pH of the solution was adjusted by using citric acid and sodium carbonate using an electronic pH meter. The HTHP dyeing machine was

prepared, and dyeing started at 40°C. The rate of temperature increase was kept at 1°C/min for uniform dyeing. The dyeing continued as per the required conditions of temperature and time. The bath was then cooled down, and all the dyed samples were repeatedly washed with hot and cold water. Then, the samples were subjected to soaping using 1gm/liter non-ionic detergent at 60°C for 15 minutes, followed by washing and drying.

Table 3.4: Dyeing Conditions for selected dyes as per Box Behnken Design of Experiment

Madder (D)	Sappanwood (S)	Annatto (A)	Marigold (M)	Pomegranate (P)	Katha, (K)	Babool, (B)	Rhubarb (R)	pH	Temperature (°C)	Time (min)
D1	S1	A1	M1	P1	K1	B1	R1	5	65	30
D2	S2	A2	M2	P2	K2	B2	R2	3	65	60
D3	S3	A3	M3	P3	K3	B3	R3	7	65	60
D4	S4	A4	M4	P4	K4	B4	R4	5	65	90
D5	S5	A5	M5	P5	K5	B5	R5	7	80	30
D6	S6	A6	M6	P6	K6	B6	R6	3	80	30
D7	S7	A7	M7	P7	K7	B7	R7	5	80	60
D8	S8	A8	M8	P8	K8	B8	R8	5	80	60
D9	S9	A9	M9	P9	K9	B9	R9	5	80	60
D10	S10	A10	M10	P10	K10	B10	R10	5	80	60
D11	S11	A11	M11	P11	K11	B11	R11	5	80	60
D12	S12	A12	M12	P12	K12	B12	R12	3	80	90
D13	S13	A13	M13	P13	K13	B13	R13	7	80	90
D14	S14	A14	M14	P14	K14	B14	R14	5	95	30
D15	S15	A15	M15	P15	K15	B15	R15	7	95	60
D16	S16	A16	M16	P16	K16	B16	R16	3	95	60
D17	S17	A17	M17	P17	K17	B17	R17	5	95	90

The optimized parameters were then found based on the maximum K/S obtained for each dye. Using the obtained value of K/S, the best 6 dyes were obtained to complete a set of

two yellow, two red/orange, and two brown dyes. The common dyeing conditions for all the dyes had to be found to dye the binary and tertiary mixtures. The common dyeing conditions were found using the design expert software to maximize K/S for each.

3.2.2. Experimental Study

The common dyeing condition for all the dyes obtained from the pilot study was used for the experimentation. As per the objectives of the study, the following work was done in this part of the study:

- Dyeing using binary and tertiary dye mixtures
- Dyeing of primary dyes at various percentages for spectral data
- Prediction of recipe and dyeing of samples as per standard

Dyeing using Binary and Tertiary dye mixtures

As per the study's objective, the binary and tertiary dye mixtures were used to dye the samples. Hoffman (1988) and McLaren (1976) worked on the compatibility check using plot ΔL versus K/S or Δ C versus K/S. It is generally used for compatibility analysis of two dyes for two sets of the progressive depth of shade developed for a binary mixture of dyes. It is done by varying the temperature and time of dyeing for one set and varying total dye concentrations of the binary mixtures of dyes in another set to judge whether the two sets of curves for shade buildup run alike or not. In the present study, this approach is adopted for the compatibility assessment.

In the case of binary mixtures for 6 dyes, a total of 12 mixtures were possible, and in the case of tertiary mixtures, a total of 8 mixtures were possible. For each mixture, two sets of samples were prepared, *viz.* set I and set II. For each set I, six samples were dyed at an equal increment of color percentage up to the total depth of shade of dye mixture with an equal concentration of the component dyes. The dyeing of all the samples in the set I was done at the common dyeing condition found in pilot study. For set II, six samples were dyed with specific time period intervals with a corresponding increase in temperature, keeping the constant concentration (with an equal concentration of component dyes).

A total of 144 samples were dyed for 12 binary mixtures, and 96 samples were dyed for 8 tertiary mixtures. The details of the mixtures are mentioned in Table 3.5 and Table 3.6.

Table 3.5: Details of Binary Mixture

S. No.	Dye - I	Dye - II	CODE
1	Pomegranate	Babool	PB
2	Pomegranate	Annatto	PA
3	Pomegranate	Katha	PK
4	Pomegranate	Madder	PD
5	Marigold	Madder	MD
6	Marigold	Annatto	MA
7	Marigold	Babool	MB
8	Marigold	Katha	MK
9	Babool	Annatto	BA
10	Babool	Madder	BD
11	Katha	Madder	KD
12	Katha	Annatto	KA

Table 3.6: Details of Tertiary Mixture

S. No.	Dye - I	Dye - II	Dye - III	CODE
1	Pomegranate	Babool	Annatto	PBA
2	Pomegranate	Babool	Madder	PBD
3	Pomegranate	Katha	Annatto	PKA
4	Pomegranate	Katha	Madder	PKD
5	Marigold	Babool	Annatto	MBA
6	Marigold	Babool	Madder	MBD
7	Marigold	Katha	Annatto	MKA
8	Marigold	Katha	Madder	MKD

The following dyeing process and conditions were used for the dyeing of 6 samples (sample 1 to sample 6) in set I in binary and tertiary mixtures.

Dye extraction was done as per requirement at optimum extraction conditions for the respective dyes in the mixture. The dye solution concentration was calculated as the sum of all dyes in mixtures with equal concentration of each dye. The mordanting process was same as done in pilot study. The dye solution was prepared as per the recipe mentioned in Table 3.7. The incremental dye material concentration (%owf) was taken with an MLR of 1:90. The

dyeing of the mordanted sample was started at 40°C on the HTHP dyeing machine. The rate of temperature increase was kept at 1°C/min for the dyeing process. The dyeing continued for 90 minutes at 85°C. The bath was then cooled down, and all the dyed samples were repeatedly washed with hot and cold water. Then, the samples were subjected to soaping with 1gm/liter non-ionic detergent at 60°C for 15 minutes, followed by washing and drying.

Table 3.7: Dyeing conditions of Set I for Binary and Tertiary mixtures

Sample Number	pH	Dye material concentration (%owf)	Temperature (°C)	Time (min)
Sample 1	7	5	85	90
Sample 2	7	10	85	90
Sample 3	7	15	85	90
Sample 4	7	20	85	90
Sample 5	7	25	85	90
Sample 6	7	30	85	90

The following dyeing process and conditions were used for the dyeing of 6 samples (sample 7 to sample 12) in set II in binary and tertiary mixtures:

Dye extraction was done as per requirement at optimum extraction conditions for the respective dyes in the mixture. The dye solution concentration was calculated as the sum of all dyes in mixtures with equal concentration of each dye. The mordanting process was same as done in pilot study. 30% owf (sum of all dyes in mixture with an equal concentration of each dye) extracted dye solution was taken as per the required MLR of 1:90. Dye solution was prepared, and the mordanted sample was dipped into it. The HTHP dyeing machine was kept ready, and dyeing started at 40°C. The rate of temperature increase was kept at 1°C/min for the dyeing process. As per the compatibility check method, the samples were drawn at increasing temperatures and time conditions as per Table 3.8 The last sample was drawn after 90 minutes at 85°C. All the samples were cooled down and repeatedly washed with hot and cold water. Then, the samples were subjected to soaping with 1gm/liter non-ionic detergent at 60°C for 15 minutes, followed by washing and drying.

Table 3.8: Dyeing Conditions of Set II for binary and tertiary mixtures

Sample Number	pH	Dye material concentration (%owf)	Temperature (°C)	Time (min)
Sample 7	7	30	55	15 min after starting at 40°C
Sample 8	7	30	70	30 min after starting at 40°C
Sample 9	7	30	85	45 min after starting at 40°C
Sample 10	7	30	85	30 min after 85°C
Sample 11	7	30	85	60 min after 85°C
Sample 12	7	30	85	90 min after 85°C

The compatibility analysis was done using various methods mentioned in the upcoming section.

Dyeing of Primary Dyes at various percentages for Spectral data

Dyeing of each of the 6 selected dyes was done at varied percentages to prepare the spectral data using a spectrophotometer for recipe prediction. 10 samples were dyed for each dye starting from the minimum to maximum possible concentrations. The dye material concentrations (% owf) used were as per Table 3.9.

Table 3.9: Concentration of dye material for primary dyes samples

Sample Number	Concentration (%) for Annatto, Madder, Marigold, Pomegranate, Katha, and Babool dye material
Sample 1	1
Sample 2	2
Sample 3	4
Sample 4	8
Sample 5	12
Sample 6	20
Sample 7	24
Sample 8	32
Sample 9	40
Sample 10	44

The following recipe and dyeing process used for the dyeing of these samples:

Extracted dye material (% owf) – 1%, 2%, 4%, 8%, 12%, 20%, 24%, 32%, 40%, and 44%.

MLR – 1: 90

Temperature - 85°C

pH – 7

Time – 90 mins

Dye extraction was done as per requirement at optimum extraction conditions for the respective dyes. The mordanting process was same as done in pilot study. The dye solution was prepared as per the recipe, and the mordanted sample was dipped into it. The HTHP dyeing machine was kept ready, and dyeing started at 40°C. The rate of temperature increase was kept at 1°C/min for the dyeing process. The dyeing continued for 90 minutes at 85°C. The bath was then cooled down, and all the dyed samples were repeatedly washed with hot and cold water. Then, the samples were subjected to soaping with 1gm/liter non-ionic detergent at 60°C for 15 minutes, followed by washing and drying.

10 samples were dyed for each dye. A total of 60 samples were dyed for 6 dyes. These samples were scanned using a spectrophotometer and served as spectral data to predict the recipe.

Prediction of Recipe and Dyeing of samples as per standard

As per the study's objective, the recipe was predicted for several chosen standards using computer color-matching (CCM) equipment. The predicted recipe for each standard was used to dye the samples. The suitable standards from the PANTONE – Cotton planner were selected as per Table 3.10.

The recipe was predicted using a spectrophotometer, and some recipe options were obtained. A compatible, suitable recipe option was selected from the obtained options. It has been observed in practical work with CCM that the sample dyed using the predicted recipe does not match entirely with the standard, and hence, the recipe was optimized to match visually.

Table 3.10: List of Pantone standards for recipe prediction

S. No.	PANTONE-TCX number
1	TCX 15-1327
2	TCX 16-1317
3	TCX 15-1317
4	TCX 14-1122
5	TCX 16-1220
6	TCX 15-1213
7	TCX 15-1415
8	TCX 14-1316

The following recipe and dyeing process were used for the dyeing of these samples:

Extracted dye material (% owf) – As per the recipe given by CCM

MLR – 1: 90

Temperature - 85°C

pH - 7

Time – 90 mins

The mordanting process was same as done in pilot study. The dye solution was prepared as per the recipe, and the mordanted sample was dipped into it. The HTHP dyeing machine was kept ready, and dyeing started at 40°C. The rate of temperature increase was kept at 1°C/min for the dyeing process. The dyeing continued for 90 minutes at 85°C. The bath was then cooled down, and all the dyed samples were repeatedly washed with hot and cold water. Then, the samples were subjected to soaping with 1gm/liter non-ionic detergent at 60°C for 15 minutes, followed by washing and drying.

Colorimetric and color difference parameters were recorded for the standard and dyed samples using two CIE standard illuminants, D65 and A. The L, A, B, C, and H values were obtained for standard and sample in both light sources. The color difference parameters dE, dL, dC, dH, and metamerism index were also calculated.

3.3 Testing and Evaluation

3.3.1 Optical Density measurement

Dye powders for various materials, namely Marigold, Sappanwood, Pomegranate, Madder, Annatto, Babool, Himalyan Rhubarb, and Katha, were taken for extraction optimization. The optical density (absorbance) was measured for the extracted dyed solution using a spectrophotometer (Make-Shimadzu) in the visible region of spectra. Optical density value was used to find the parameters for maximum color extraction.

3.3.2 Computer Color Measurement using Spectrophotometer

The color parameters were measured using Konica-Minolta Spectrophotometer, model-3600d. The measurement was done in a commonly used Daylight (D-65) illuminant with a 10° standard observer over the 400-700 nm range. CIELAB coordinates L*, a*, b*, C*, h*, and K/S values were obtained with the help of relevant software for all the dyed samples.

K/S measures the surface color strength of dyed samples, and CIE L*, a*,b* values signify the lightness/darkness, redder/greener tone, and yellower/bluer tone, respectively. The R.F.D. fabric was used as standard, and the color difference values dL*, da*, db* and dC^*_{ab} were also determined. The CIE color coordinate values were used to find the optimization of dyeing parameters, compatibility in the dye mixture and shade matching.

3.3.3 Compatibility Evaluation

Set I & II dyed samples were obtained by binary and tertiary dyes mixture. Both sets have 6 samples each for the respective mixture. The compatibility was evaluated using the methods as below:

A. Method based on -dL vs. dC and -dL vs. K/S plot
B. Method based on da vs. K/S and db vs. K/S plot
C. Based on change in Hue angle (h°)
    (i) Using graphical analysis
    (ii) Using coefficient of variation (CV)
D. Compatibility factor method
E. Visual rating

A. Method based on -dL vs. dC and -dL vs. K/S Plot

Samanta et al., (2015) worked on compatibility check by plotting ΔL versus K/S or ΔC versus K/S. It is generally used for compatibility assessment of two dyes for two sets of the progressive depth of shade developed for a binary mixture of dyes. It is done by varying the temperature and time of dyeing for one set and varying the total dye concentrations of the binary mixtures of dyes in another set to judge whether the two sets of curves for shade buildup run alike or not. A plot of ΔL versus ΔC and ΔL versus K/S gives better results than other methods because they assume that there is no interaction between dyes and no change in the rate of dyeing in the presence of another dyestuff, which is not true. A plot of ΔL versus ΔC and ΔL versus K/S would require a precise temperature-controlled machine for progressive shade buildup. This method is time-consuming and subjective.

CIELAB coordinates L*, a*, b*, C*, and h* values were obtained for all the dyed samples using spectrophotometer. The undyed white fabric was used as standard, and the color difference values were also determined. For a binary or tertiary mixture, plots of –dL vs. dC were made for both set I and set II. The compatibility analysis was done based on the closeness of curves for set I and set II. In the same way, the plots of –dL vs K/S were made for both set I and set II. The compatibility analysis was done based on the closeness of curves for set I and set II (Samanta et al., 2015)

B. Method based on da vs. K/S and db vs. K/S Plot

Shukla & Dhuri, (1993) used the plots of da vs. K/S and db vs. K/S to check the compatibility of dyes. It was desired that the redness or yellowness buildup should be in accordance with K/S for both sets. In the case of compatible mixtures, the curves for set I and II in plots of da vs. K/S and db vs. K/S should coincide. The compatibility is assessed by observing the curves for both sets.

C. Based on change in Hue angle (h°)

(i) Using graphical analysis

If the dyes exhaust simultaneously i.e. as the time of dyeing increases the dyed material shows an increase in depth but no change in hue; the dyes are termed compatible in that particular combination, under the dyeing conditions. Beckmann *et al* (1972); Beckmann and Hoffmann, (1983).

Mclaren, (1976) devised an objective method with a view to answer the doubts and disputes caused by the earlier methods of assessing compatibility because of its subjective nature. With the introduction of color measurement systems, a series of compatibility tests can be assessed by measuring, e.g., the hue angle.

The hue angle (h°) values indicate the hue depend on the a* and b* in CIELAB coordinates. The plot of hue (h°) values against the samples in set I and set II shows the change in hue with time, temperature, and concentration. The assessment of mixture compatibility was conducted based on the observed nature of hue build-up or hue alteration. The compatibility of the dyes in the mixture was determined by observing whether there was any alteration in color between the samples dyed at different time, temperature, and concentrations.

(ii) Using coefficient of variation (CV)

In continuation of the change in hue angle (h°), it was observed that the change in hue angle can be quantified by measuring the C.V.%. In this concern, the value of C.V. for set I and set II was measured for all the combinations. It was also attempted to get a single value of C.V. for each combination using the sum of C.V.s of sets I and II.

The coefficient of variation was calculated to check the hue angle (h°) variation in both the mixture sets. The coefficient of variation was also used to give the compatibility rating for variation in hue angle (h°) values among all the 12 binary mixtures. The same rating method was used for 8 tertiary mixtures.

D. Compatibility Factor method

The K/S values are directly proportional to the colorant concentration on the fiber. To determine the K/S value of a mixture consisting of two or more colorants, K/S values can be added for the individual colorants that were dyed to the same depth as the mixture and measured at their respective λmax values. The ratio between the estimated and observed K/S values for the mixture at the respective λmax of component dyes should be as close as possible for compatibility.

The reported K/S ratio of the estimated mixture to the observed mixture is denoted as the compatibility factor (C.F.).

$$C.F. = \frac{(K/S) \text{ mix calculated}}{(K/S) \text{ mix observed}}$$

The compatibility factor (CF) should ideally be '1'. Such conditions are uncommon in actual dye combinations; hence, compatibility factor values always depart from "1" due to the fact that component dyes also contribute to the K/S values of the mixture at each λmax. Compatibility is assessed from the closeness of the C.F. values of both the component dyes calculated at their respective λmax.

The C.F. was obtained in the present study using the K/S data of mixture and component dyes. For binary mixtures, spectral data of sample number 5 from set I was used for all the combinations. The dye concentration for sample 5 was 25% owf. Primary dyes dyed samples were used for this purpose. The K/S data for component dyes was predicted at a dye concentration of 12.5% owf using design expert software.

E. Visual Rating

All binary and tertiary mixtures samples were visually assessed for the compatibility check. The visual check was done in a standard color-matching light box in a D-65 light source at a 45° angle. The compatibility was checked for sets I and II in terms of depth, hue, and chroma.

Two observers were asked to rate the 12 binary and 8 tertiary mixture samples for compatibility. They were asked to rate the samples out of 30 points for binary and out of 20 points in case of tertiary mixtures. The average rating of both observers was used to give a rating for the samples. The rating was converted into ranks to calculate Spearman's rank correlation coefficient. The value of the coefficient indicated the correlation between the two observers.

3.3.4 Color Fastness Evaluation

Color fastness properties were evaluated for selected samples. In colorfastness tests, color fastness to washing, color fastness to light, and color fastness to rubbing were done. The samples were chosen from primary dyes, binary, and tertiary combinations. A sample of dye concentration of 24% owf was tested for every primary dye. Sample number 5 from set I was chosen for each combination for binary and tertiary combinations. A total of 26 samples were tested for color fastness.

A. Colorfastness to Washing

A color fastness test was done for 26 samples using the AATCC test method 61-2009 (1B). The test was conducted on laundr-o-meter, make-Ramp Impex Pvt Ltd.

The test sample of size 15cm x 5cm was stitched with a multi-fiber strip of size 5cm x 5 cm. The AATCC multi-fiber strip contains acetate, cotton, nylon, silk, viscose, and wool fibers. The AATCC detergent was used as 0.37% powder detergent of total volume with a total liquor volume of 150 ml. The test sample with multi-fiber fabric was immersed in liquor with 10 rubber balls in the container. A 20-minute wash cycle was executed. The samples were then taken out, rinsed, and dried.

Utilizing the geometric greyscale, the multi-fiber strip's staining and the dyed samples' color change were visually evaluated. The color difference between stained and white fabric was compared and quantified using a standardized scale. The evaluation of color change entailed quantifying the difference in color between the tested sample and the original fabric using a scale.

B. Colorfastness to Light

The dyed samples were tested for their fastness to light. A fade-o-meter was used to carry out the testing using the test method ISO-105-B02. The testing was carried out by exposing the dyed samples for 20 hours and fading under light emitted from a calibrated carbon-arc lamp. The geometric grey scale was used for visual assessment to evaluate the degree of fading of dyed samples.

C. Colorfastness to Rubbing

The rubbing fastness of the dyed samples, in both dry and wet conditions, was assessed using the Crock-o-meter following the test method IS 766: 1988, which is based on ISO 105/X-1984.

Samples with dimensions of 14 cm x 5 cm were prepared and securely attached to the crock meter apparatus to conduct the necessary tests. The experimental procedure consisted of conducting a dry rubbing test on a scoured white cotton cloth. This required a force of 9N and executing 10 cycles. In the wet crocking fastness test, a scoured white cotton cloth was fully submerged in distilled water, resulting in a wet pickup of 100%. Efforts were made to preserve the moisture content of the cloth with regard to its wet rub fastness. The dry cotton fabric of a white color was originally affixed to one of the fingers of the rubbing arm on the crock-o-meter. This was thereafter followed by affixing the wet white fabric. Following that,

the test strokes were carried out. Subsequently, the samples were scrutinized for the presence of any staining on the white textile material and discoloration of the dyed sample.

3.3.5 Anti-Microbial Testing

Anti-microbial testing was carried out by using AATCC 100:- 2019 (Using Nutrient Agar) test method.

Below mentioned test condition were used:

Test Bacteria:

1. *Staphylococcus aureus*, American Type Culture Collection No. 6538. Gram-positive organism
2. *Klebsiella pneumonia* (ATCC 4352) = American Type Culture Collection No. 4352.Gram negative organism, 1-2 $x10^5$

- Sample Size: Swathes of 4.8 cm diameter for each bacterium
- Media Used: Nutrient Agar
- Dilution Media: Nutrient broth
- Method of plating: Pour plate method
- Inoculum/ Plate: 0.1 ml
- Incubation conditions: 37± 2°C for 24 hr

The test results were assessed as the percentage of bacterial reduction for both the bacteria.