

Project (Thesis): TE-407

"Study on effect on concentration of Soda on dyeing of woven cotton fabric with Reactive dye"

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Date of Submission: 24 July 2012

This report presented in partial fulfillment of the requirements for the Degree of Bachelor of science in Textile Engineering.

Abstract

Cotton is a natural fiber and it is a cellulosic fiber. Reactive dye is most commonly used for cotton dyeing. It is used to dye wide range of shades like light shades to dark shades of cotton fabric. The aim of our work is to observe the effect of dyed sample when we change the concentration of soda in the solution. Here we conceder three different concentration of soda 20 gm./l, 15 gm./l &10 gm./l. One thing we carefully considered that the entire three samples are similarly processed and the fastness properties like color fastness to wash; color fastness to rubbing is strictly controlled. Then we observed the depth of the shed with the help of data color software (CCMS).

ACKNOWLEDGMENT

At the very outset, we would like to express our deepest gratitude to almighty ALLAH for giving us the strength and opportunity to finish the report within schedule time.

Foremost, we would like to express our sincere gratitude to our advisor Md. Rabiul Islam Khan for the continuous support of our study and research, for his patience, motivation, enthusiasm and immense knowledge. His constructive suggestions and criticism helped us for the successful completion of the thesis work. His guidance helped s in all the time of research and writing of this thesis. We could not have imagined having a better advisor and mentor for our study.

We are very much grateful to Mr. Mahinur Rahman (General Manager) Hossain Dyeing & Printing Mills Ltd. to give us this opportunity to make our project work at his organization.

We are also grateful to all the staffs of Hossain Dyeing & Printing Mills Ltd. They are always cooperative to us during our project work at their organization.

We are also grateful to the department of Textile Engineering, DIU for its facilities, specially its lab facilities. We are also grateful to all the concerned people of textile engineering lab.

Last but not the least; we would like to thank our family, for their ever present support from thesis inception until the last sentence was penned.

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1. INTRODUCTION

The reactive dye is dissolved in water to which an electrolyte is added to exhaustion of the dye. Cotton fabric is then introduced to the dye liquor and the dye is exhausted on to the cotton fibers. For the dye exhaustion two types of electrolyte: Glauber salt (Na₂SO₄.10H₂O) and Sodium Sulphide (Na₂S) are used separately at various amount. Various color of Reactive dye are used as dye staff to produced various type of shade%. For the fixation and forming covalent bond alkali was added to the dye liquor. For the fixation two types of alkali: Soda ash and Caustic soda are used separately or mixture at various amounts. The covalent link is formed between the dye molecule and the hydroxyl group of cellulosic fiber. Reactive dyes have specific temperatures at which reaction between dye and fibers are optimum. During dyeing initial temperature was 45^oC and final temperature was 100^oC to get a good shade. To fulfill our thesis and also for the color fastness evaluation of the dyed fabric three types of colorfastness test were treated such as: Rubbing test, Abrasion test and Washing test.

2. REACTIVE DYE

2.1 Reactive Dye: Why So called:

A dye that, under suitable conditions, is capable of reaction chemically with a substrate to form a covalent dye – substrate linkage.

Reactive dye containes reactive group and this reactive group make covalent bonds with the fibers and becomes part of the fibers.

Reactive dyes react with the fibres. Mainly react with cellulosic fibres

e.g.cotton, jute, blast fibres, viscose, flax. It can also be applied to protein fibres. e.g. wool &silk.

Here the reactive dye present in the dye molecule act as an integral component of the ibre polymer after dyeing in a favorable condition. Mainly the reaction occurs in an alkaline condition ($P^{H} > 10$). The covalent bond is formed between the dye molecule and –OH groups of cellulose or –NH₂ (amino) group of polyamide or wool fibre.

The general formula of Reactive dye can written be as follows:

D-X-Y Here, D- >Chromophore of dye post X->Bridge Y- > Functional group

Bond formation between dyes &fibre: D-X-Y + Fiber = D-X-Y –Fibre (Covalent bond).

Reactive dyes are water soluble: D-F + Cell-OH--> Dye-F-O-cell (F=Functional group)

Reactive dyes are also react with water which is known as Hydrolysis:

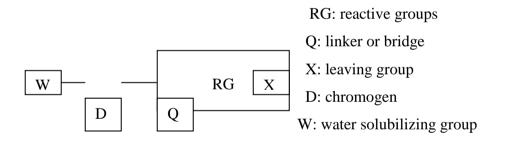
D-F + H-OH--> Dye-F-OH Dye + water = Hydrolyzed dyes.

The general reaction between dye molecules and the fibres are as below:

 $D-SO_2-CH_2-CH_2-OSO_3Na + OH-Cell \longrightarrow D-SO_2-CH_2-CH_2-O-Cell + NaHSO_3$ $D-SO_2-CH_2-CH_2-OSO_3Na + NH2-Wool \longrightarrow D-SO_2-CH_2-CH_2-NH-Wool + NaHSO_3$

2.2 General dye features:

Dyes contain anionic functional groups; structure of acid, direct or mordant dyes with reactive group that form covalent bond with fibre. The general structure A reactive dye is as below:



Simply

Dye- X

Where, the word 'Dye' includes all physical, structural components of water soluble cellulose reactive dye, except the leaving group X

Solubilizing group – NH₂, -NH-CH₂, -OH, NO₃, -N-(CH₃)₂, -COOH, -SO₃Na, -CH₃, -OCH₃-NO₂

History of Reactive dye:

On the occasion of 100 years celebration of synthetic dye manufacturing, two chemists of ICI Company (UK) name Stephen and Rattee tried to manufacture new dye stuff. Thus they succeed to invent a new dye in 1956 which was named RECATIVE DYE. This was manufactured for dyeing cellulosic fibres.

The 1st three reactive dyes were procion yellow R, procion Brilliant red 2B and procion Blue 3G.For this effort they were awarded Gold Medal of the Society of Dyes and Colorists for the year 1960.

These dyes came to our country in Mid 60's and became very popular during 80's

2.3 Properties of reactive dyes:

Physical / Chemical properties:

> Chemical structure:

Mostly triazinyl & vinyl sulfone derivatives small & simple molecular structures. Anionic in electrostatic nature.

> Physical state:

Used as solutions, depends on base compounds.

> Molecular weight:

Low generally ranges 69-21 gm/mole. The higher the MW the more costly the dyeing because more dye has to be used to produce the same shade.

> Water solubility:

Generally very good. Sometimes modified to lower solubility.

> Hydrolysis:

Dye fiber bond is hydrolysis by strong acids or bases. The more reactive dischloro & trichloro phrimidine are especially hydrolysis prone. During the application of conventional reactive dyes 15-40% of the dye is hydrolysis.

> Spectra:

Have very bright shades owing to their very narrow & hug absorption bands which result from their sample structures.

> Bonding types:

Covalent bond to fiber substrate. Vinyl sulfone form amino bonds with silk, wool & either bond to Cellulose. Acid chlorides form amide bonds with wool & either bonds with cellulosic.

Application Properties of reactive dyes:

Leveling	: Very good.
Exhaustion	: good.
Migration	: extremity good.
Acid fastness	: dye fire is hydrolyzed.
Alkali fastness	: fair of good Index 3-5.
Light fastness	: very good. Index 5-6.
Chlorine fastness	: limited.
Wash fastness	: very good. Index 4-5.
Perspiration fastness	: good. Index 4-5.
Useful color	: full range of colors from extremely right to heavy darks.
	No flukes. No fluorescent shades.
Rate of dyeing	: very rapid.
Dyeing process	: exhaust.
After treatment	: soaping & rinsing to remove hydrolyzed dyestuffs.

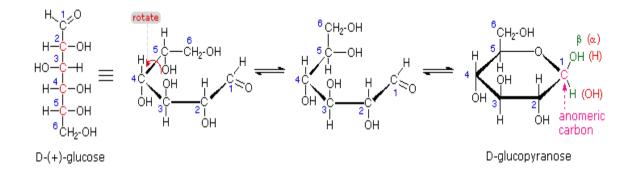
2.4 Importance of Reactive group present in Reactive dyes:

Reactive groups do not contribute color which is determined by chromogen group

- > The reactivity of vinyl sulphone group is less than halogen group.
- If no of reactive group increases, binding also increases depending on dye structure.
- \blacktriangleright Reactive dye absorb up to 90%.
- > If the molecular wt of reactive group increases, reactivity also increases.
- > Reactivity of vinyl sulphone group increases with increases of PH and temp.
- Sulphone group has mere solubility but it is not stable.
- > Chlorine imparts medium reactivity, but it is cheap.
- > Reactivity of Fluorine is the least and its rate of hydrolysis is also less.

2.5 Criteria of cellulose for attaching Reactive dye:

Chemical structure of cellulose molecule:





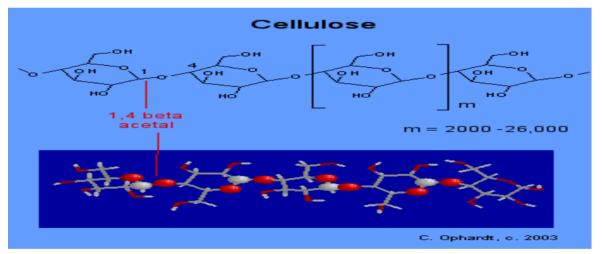


Figure no:

Cellulose, the polymer of cotton,

With a degree of polymerization of about 5000 cellubiose units (i.e. n = 5000)

Glucose unit:

* Each glucose unit contains one primary hydroxyl group (-CH2OH) and two secondary hydroxyl groups (>OH)

*Primary hydroxyl group ((-CH2OH) at C6 position is more reactive than the secondary hydroxyl groups at C2 and C3 position

*C2 hydroxyl group is supposed to be acidic than C3 hydroxyl group under suitable alkaline condition and more reactive.

*The hemiacetal group at C1 position is the most active.

*The reaction between reactive group and cellulose takes place predominantly with primary hydroxyl group.

*In case of monochloro triazinyl dyes, the reaction ratio of -CH2OH &-CHOH is 15:1 i.e. C6: c2 or C3 =15:1

*In case of dicholor trizinyl dyes, the reaction ratio of -CH2OH &-CHOH (3:1 to 7:1)

* Secondary hydroxyl group is the reactive while primary one is the most reactive **2.6 Classification of Reactive Dyes:**

A. On the Basis of Reactive group:

Two types

1) Halogenated heteroycles

Triazine group :





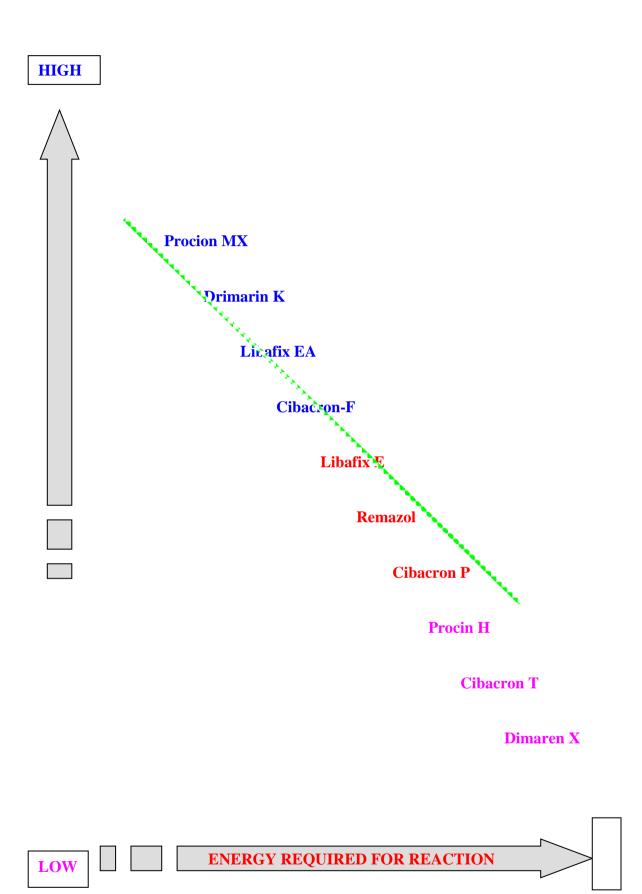


2). Activated Vinyl Compounds:

- a) Vinyl Sulphone (D-SO₂-CH₂-CH₂-) Ex: Ramazol.
- b) Vinyl Sulphonamide (D-SO₂-NH-CH₂-CH₂-) Ex: Levafix.
- c) Vinyl acrylamide (D-NH-CO-CH₂-CH₂-) Ex: Primazine.

B.On the Basis of reactivity:

- 1. Low reactivity: Ex:Drimarene X
- 2. Moderate reactivity: Ex: Remazol.
- 3. High reactivity : Ex: Procion MX
- **1.** Lower reactive dyes: Reactivity of these dyes is low. So highly alkaline environment required for the fixation of these dyes with substrate. Here P^H is maintained 12-12.5 by using NaOH in bath. Example: Drimarene X, Cibacron T.
- **2. Medium reactive dyes:** Reactivity of these dyes is medium. Here P^H is maintained 11-12 by using Na₂CO₃ in bath. Example: Levafix E, Remazol.
- Higher reactive dyes: These dyes are highly reactivity. So fixation of these dyes is easy & lower alkaline medium is kept. Here P^H is maintained 10-11 by using NaHCO₃ in bath. Example: Procion Mx, Drimarene KLP.



C. On the basis of dyeing temperature and method:

- **1. Cold brand:** These types of dyes are highly reactive as they possess highly reactive group in their constituent. So dyes of these dyes can be done in lower temperature i.e. 32-60^oc. Example: Procion Mx, Levafix E etc.
- 2. Medium brand: These types of dyes contain reactive group of moderate reactivity. So dyeing is done in higher temperature than that of cold brand. Here temperature is required for dyeing i.e. $60-70^{\circ}$ c.
- Hot brand: These types of dyes contain reactive of least reactivity. So high temperature is required for dyeing i.e. 72-93^oc temperature is kept for dyeing. Example: Procion H, Cibacron T etc.

Recent classification:

Classification of reactive dye types has been formulated recently as:

Group 1: Alkali controllable reactive dye:

These dyes have optimal temperature of fixation between 40° C & 60° C. They are characterized by relatively low exhaustion in natural salt solution before alkali is added. They have high reactivity & care in addition of alkali is necessary to achieve level dyeing, preferable at a controlled dosage rate. Typical example of dyes belonging to this group has dichlorotriazine, chlorodifluoropyrimidine, and dichloroquinoxaline or vinylsulphone reactive system.

Group 2: Salt controllable reactive dyes:

Dyes in group show optimal fixation at a temperature between $80^{\circ}C$ & the boil. Such dyes exhibit comparatively high exhaustion ay neutral P^H, so it is important to add salt carefully to ensure that dyeing are level. Electrolyte addition is often made portion wise or preferably at a controlled rate of dosage. Dyes with these properties typically have low reactivity systems such as trichloropyrimidine, aminochlorotriazine or bis (amionochlorotriazine). Amino fluorotriazine dyes in the Cibacron F (CGY) range have high substantively & should thus be regarded as salt controllable but they are sufficiently reactive for fixation at 60° C or even lower temperatures by batch wise application.

Group 3: Temperature controllable reactive dyes:

This group is represented by those dyes that react with cellulose at temperatures above the boil in the absence of alkali, although if desired they can be applied under the same condition as the salt controllable group with alkaline fixation at a temperature between 80°C & the boil. Dyes in this group have self leveling characteristics so there is no need to use auxiliary products to facilities level dyeing. Good results can be achieved by controlling the rate of temperature rise. At present only the kayacelon React (KYK) range of bis (amino nicotinotriazine) dyes belong to this group.

Chemical classification of reactive Dyes:

Reactive dyes can be classified chemically into three different groups:

1) <u>Chloro triazinyl reactive Dyes</u>

- (a) Monochloro dyes
- (b) Dichloro/ Bifunctional
- (c) Trichloro

Monochloro dyes

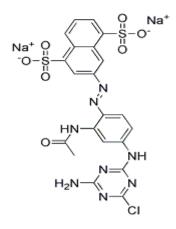
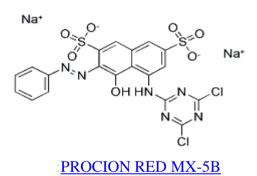


Fig: Cibacron Yellow R

(a) Dichloro/ Bifunctional





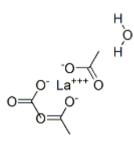


Fig: Reacton

(2) Vinyl sulphone Dyes:

Dye.SO₂CH= CH₂

(3) Heterocyclic halogen containing Reactive dye

I) Drimarine K

II) Levafix E

4. Primazine P Dyes

2.7 Trade Name of Reactive dyes:

Trade name	Company	Country	
Procion	ICI	UK	
Cibacron	Cibageigy	Switzerland	
Colvazol	Lonsen	China	
Dychufix	Dyesin	China	
Reactone	Cibageigy	Switzerland	
Levafix	Bayer	Germany	
Priazin	BASF	Germany	
Remazol	Hoechst	Germany	
Uhopid	People's china	China	
Mikacion	Mitshui chemical Co.Ltd.	Japan	
Cavalite	Dupont	USA	
Diamarene	Sandoz	Switzerland	

Table no. 01: Trade name of reactive dyes.

2.8 Hydrolysis of Reactive Dyes/ Technical difficiency of reactive Dyes:

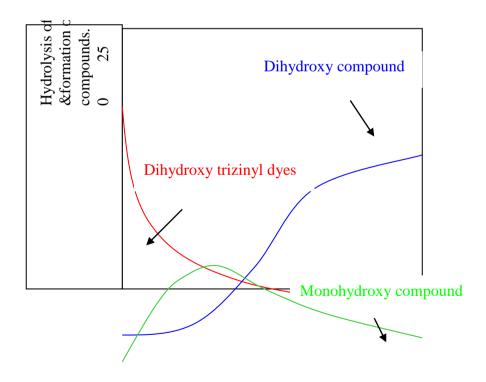
Under alkaline condition, Reactive dyes react with the terminal hydroxyl group of cellulose. But if the solution of the dye is kept for long time its concentration drops. Then the dye react with the hydroxyl group of water the reaction of dye with water is called Hydrolysis of reactive dye.

In case of Vinyl Sulphone Dyes:

a) Dye-SO2CH=CH2 + Ho- celluloso Dye - SO2CH2-CH₂O - cellulose

DYE + Cotton \rightarrow Dyed cotton

b) Dye-SO2CH= CH2 + HO- H \longrightarrow Dye-SO2 CH2 CH2 O-H Dye + Water \longrightarrow Mono Hydroxy compound.



15	30	45	60	75
Time of dyeing (min)				

Figure no. 02: Dyeing time Vs Hydrolysis of reactive

dyes.

Hydrolysis increase with:

- a) Higher temperature of dyeing.
- b) Higher P^H .
- c) Higher M: L ratio.
- d) Longer dyeing time.

2.9 REACTION MECHANISM OF REACTIVE DYES WITH CELLULOSE:

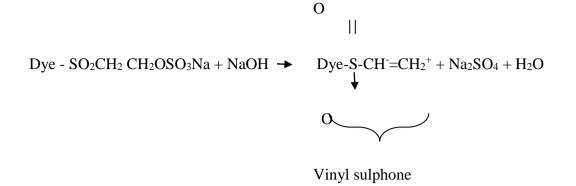
The reaction of reactive dyes with cellulose generally follows two major reaction viz (i) Nucleophilic Addition reaction & (ii) Nucleophilic substitution reaction. The former one occurs for heterocyclic aromatic ring having the reactive groups & latter one occur in case of vinyl sulphone reactive dye.

 Nucleophilic Addition reaction: This also called Michael reaction. The term Michael reaction specifically refers to the addition of carbon-ion to an activated alkene & Alkyne. Hetero-nucleophilic addition is called Michael dye reaction.

* This reaction has two phases-

- a. Elimination of masking or protecting group. (Dyeing process).
- b. Nucleophilic Addition.
- Elimination of masking group:

The vinyl sulphone group is present in the reactive dye in protected form. This group remains inactive in dyes as β -Sulphatethyl sulphone which has ability to react with the cellulose. But this group in presence of optimum alkali generated Vinyl sulphone group in situ process.



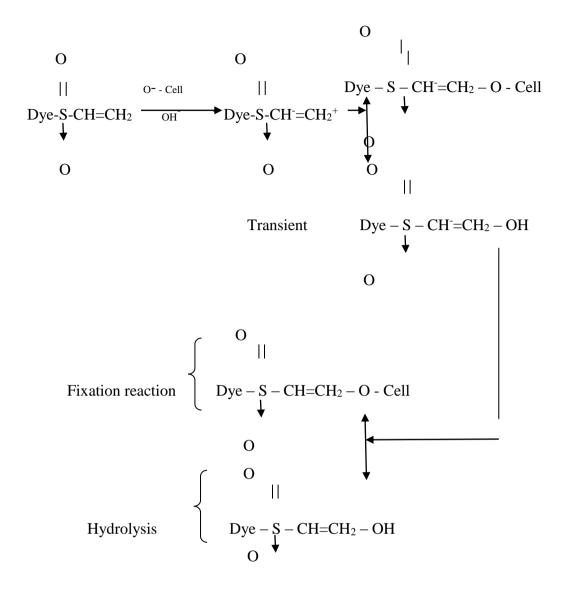
On the other hand the alkali is to causes the acidic dissociation of some hydroxyl groups in the cellulose. This produces the cellulosic ion (Cell-O⁻) which is liable to nucleophilic attack of the reactive group.

 $Cell - OH + NaOH \rightarrow Cell - O^{-} + H_2O + Na^{+}$

Note: Here Alkali used to produce for cellulosic ion

• Nucleophilic Addition reaction:

In the vinyl sulphone group generated in the dye bath or in situ by the carbon – carbin double bond (C=C) is polarized by the powerful electron atracting sulphone group. This polarization confers a positive character on the terminal carbon atom favoring nucleophilic addition either by cellulose (Cell – O^-) ion or by OH⁻ of the water.



2.Nucleophilic substitution reaction:

This compreses two steps viz-

1) Produce Nucleophilic species:

 $e^{P^{H_{9-11}}} Cell - O^{-} + H_2O$

2) Substitution Reaction or Nucleophilioc Attack:

2.10 DYEING MECHANISM OF REACTIVE DYES:

Dyeing with reactive dye involved three stages:

- 1. Exhaustion of dye.
- 2. Fixation of dye.
- 3. Soaping / Washing.

Exhaustion of dye:

Dyeing is started in neutral solution so that there is little like hood of the dye reacting with the cellulose. During this stage of dyeing, some reactive dye will be absorbed by the fibers, the amount depending upon its substantivity. Sodium chloride or sulfate is added to promote exhaustion of dye by neutralizing the negative charge formed in the fabric surface.

Fixation by alkali:

After the initial exhaustion phase, the P^H of the dye bath is increased by complete or gradual addition of the appropriate type and amount of alkali. The fixation process is as follow:

- a) Nucleophilic substitution.
- b) Nucleophilic addition:

DSO₂-CH₂-CH₂-OSO₃Na DSO₂-CH₂=CH₂ + Cell-OH SO₂-CH₂-CH₂-O-Cell

Soaping / Washing:

Soaping or washing is done to remove unreacted, hydrolyzed dye and residual of salt and alkali. The residual of salt and alkali are removed by successive rinsing in cold and then hot water. This also removes some unfixed dye. Soaping with detergent remove the remainder. The dyeing is then finally rinsed in warm water. Soaping repeated for deep dyeing.

FACTORS OF FIXATION OF DYEING WITH REACTIVE DYE:

The efficiency of fixation is:

1) The reactivity ratio, the ratio of rate constants for the fixation reaction and hydrolysis;

2) The substantivity ratio, the relative concentrations of dye absorbed into the substrate and remaining in the dye bath;

- 3) The diffusion coefficient of the dye in the substrate;
- 4) The liquor ratio and;
- 5) The surface area of the substrate available for absorption of dye.

2.11 ACTORS OF DYEING CYCLE / PHASE OF REACTIVE DYE:

- 1. Pretreatment of the substrate
- 2. P^H of the substrate prior to dyeing
- 3. P^H of the dye bath
- 4. Solubility of the dyestuff
- 5. Dyeing temperature
- 6. Electrolyte concentration

- 7. Dyeing time
- 8. Material and Liquor ratio (M:L)
- 9. Type of alkali
- 10. Washing off sequence
- 11. Quality of water and salt.

1. Pretreatment of the substrate

The process of removing natural impurities like oils, fats, waxes, pectin, protein, amino acid and hydrophobic impurities from a yarn or fabric is called "Scouring". Scouring is done to improve absorbency of the textile material by removing the oils and fat in the yarn or fabric by boiling.

The pretreatment includes the scouring and bleaching of the substrate prior to dyeing. The main object of scouring treatment is removing the major impurities from the cotton and improving the absorbency. The material shall be free from any contaminants and natural coloring matter in the scouring and bleaching treatment. The extant of the pretreatment such as ground whiteness depends upon the target shade, as brighter and whiter ground whiteness is required for light and bright shades and dark and dull shades can be dyed on scoured ground without any difficulty.

2. **P^H of the substrate**

 P^{H} of the substrate prior to dyeing must be controlled and it should be either neutral or slightly acidic because if the p^{H} is alkaline at the beginning, the dye molecule may form a permanent bond or premature fixation leading to unlevel dyeing.

3. P^H of the dye bath It should be weakly acidic to neutral in the dyeing bath in migration and exhaustion phase, before addition of alkali. It must be checked and regulated because presence of bicarbonates in the water may increase the P^H with increase in temperature, which results into partial fixation of the dye molecules resulting in unlevel dyeing. The P^H in the fixation stage must be kept at 10.0 – 11.0, which shall be achieved by either soda ash along or a mixture of soda ash and caustic soda.

4.Solubility of the dyestuff

It is better to consider the dye solubility chart of individual colors provided by the manufacturer, the dyes with higher solubility are more suitable for better shades and color fastness control.

5. Dyeing temperature

The temperature and rate of rise of the dye bath affects the affinity of the dye molecules towards fiber, rate of hydrolysis, migration and covalent formation. Therefore the dyeing temperature selected must be as per the dye sub class. Effect of temperature on the build up and fixation of individual dyes must be studied to form dyes groups having similar characteristics and then these groups must be selected for making combination shades.

6. Electrolyte concentration

The dyes must be exhausted by addition of salt or glauber salt before starting the fixation of color. For electrolyte concentration in the dye bath to be used please refer to tables provided by the dye manufacturers. The electrolyte used must be free from unwanted impurities such as metal salt (iron, copper, etc) calcium content, insoluble material, hardness creating salts.

7. Dyeing time

The dyeing time mist be selected based upon the depth of the shade. The timing must be optimized to get maximum exhaustion as well as maximum fixation of the color in the dye bath. Based on the exhaustion and fixation curves of the individual dyestuffs, an optimum tine can be selected. There is advantage of increasing the fixation time than desired because that will not help either in exhaustion or fixation. Generally dark shades need more time in fixation phase than the lighter shades.

8. Material and Liquor ratio (M:L)

The M:L of the dye bath affects the dye or shade performance to a large extent, as for as possible robust dye combination must be used, which are unaffected by the change in liquor ratio. The chemical concentrations must be changed with the change in liquor ratios.

If a dye house is having different capacity machines with different M:L ratio then it shall be taken into consideration from the lab recipe stage.

9. Type of alkali for fixation

There are different methods applied to achieve the right P^{H} for the fixation of reactive dyes. Normally soda ash alone is used for the fixation purpose; however a mixed alkali system of soda ash and caustic soda is also used, particularly in the case of dark shades. A gradual P^{H} change is preferred over the shock change for better dyeing results, therefore a dosing system is strongly recommended in the reactive dyeing to achieve consistent shade reproducibility.

10. Washing off sequence for reactive dyes

The hydrolyzed and unfixed dyes which are present in the dye bath as well as on the fiber after the completion of dyeing cycle, these hydrolyzed dyes have no affinity for the fiber but still they act as direct dyes and in the presence of electrolyte penetrates inside the fiber, with the rise in temperature of washing and soaping. If these are not removed before soaping and washing these can severely affect the color fastness properties. Such trouble can be reduced or eliminated by the following an optimized washing off sequence after dyeing.

The dyed goods must be free from any inorganic salt before going for a soaping treatment. Generally a non ionic soaping agent is used for soaping purpose to get good washing fastness. An organic acid such as acetic acid is must be used to neutralize the dye bath. The soaping treatment can be done up to boiling temperature to remove the unfixed and hydrolyzed dye effectively. A higher soaping temperature can be selected because unfixed dye has practically no affinity for the fiber and the loosely held dye rapidly diffuse out. The soaping treatment is recommended in a neutral bath because under alkali soaping conditions at higher temperature the dye fiber bond may break and result into loss of color value due to rupture of dye fiber bond. If the soaping treatment is carried out efficiently and carefully then there is no need of cationic dye fixation treatment at the end of the dyeing cycle.

2.12 Factors considered for Dye selection:

1. <u>Selection of dyeing method:</u>

- I. Discontinuous method.
- II. Semi-continuous method-(a) pad Batch method(b) pad Jig method
- III. Continuous method- (a) pad steam method
 - (b) pad dry method
 - (c) pad thermo fix method

Selection of method depends on:-

- Speed of diffusion on the fibre
- Affinity of dye to fibre
- Reactivity of dye stuff.

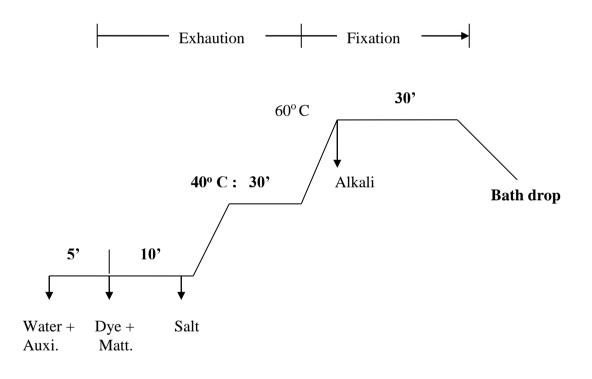
2. Selection of Brand

- Hot brand
- Medium brand
- Cold brand
- 3. Cost of dyes
- 4. Chemical groups.
- 5. Quality of products
- 6. Fastness required.
- 7. Quality of dyes.
- 8. Availability of dyes.
- 9. Reproducibility.

Dyeing process of cold Brand dyes:

Dyestuff- 3% Salt-60 gm/L Soda ash -15g/L M: L -1.10 Temp -60⁰C Time -1 hr

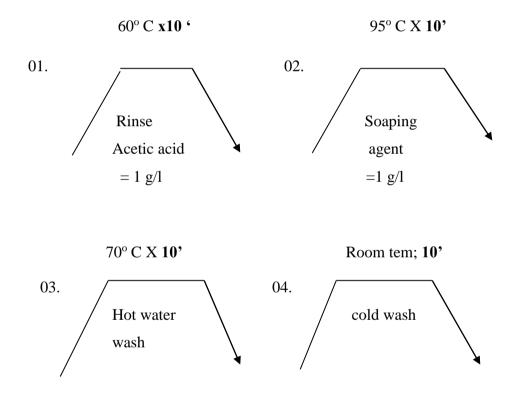
Dyeing curve:



Procedure:

According to dyeing curve at first auxiliaries and water are added in the dye bath. And it is kept for 5 minute. Then material dye is added respectively. Then after 10 minutes salt is added. After adding salt dye is heated to 40^{0} - 50^{0} C kept for 20-30 minutes. This is the exhaustion period of dyeing. Then alkali is added in the dye bath. After adding alkali the dye bath heated for 40-60 minute at 50^{0} - 60^{0} C. This is the fixation period.





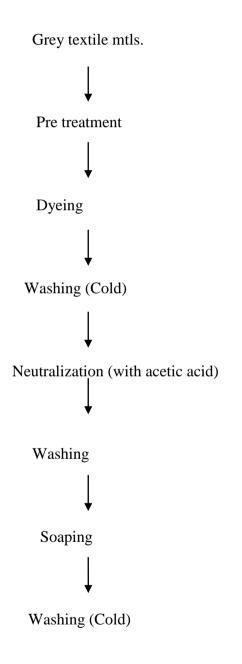
1) After dyeing operation is completed the dye material is put into a bath containing 1% stock solution of acetic acid. This operation is done neutralizing the fabric. This operation is performed at 60° C for minutes.

2) Secondly the material is treated with a 1g/L soap solution, which removes the unfixed dye from fabric surface and makes the surface clean.

3) Thirdly material is treated with a hot water bath.

- 4) Fourthly material is treated with a cold water bath.
- 5) Finally the material is dried in a drier.

Sequence of Dyeing:



Dyeing process with Hot Brand dyes:

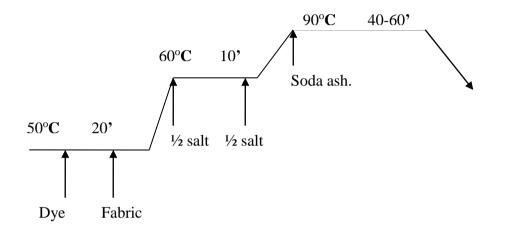
Recipe:

Dyestuff -- >4% Salt -- >80g/L Soda -- >20g/L M: L -- >1.15 Temperature -- >50-90⁰C Time -- >1-1/2 hrs

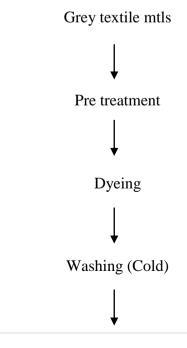
Dyeing Procedure:

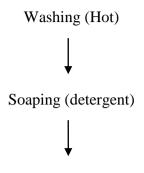
At first paste is prepared with cold water and dye .then prepare dye sol ⁿ with Hot water. Dye bath is adjusted to 50° C and dye sol ⁿ is added and Fabric is dyed about 20 min to the dye bath and temp is raised to 60° C continuously. Now ½ salts is added and dyed for 10 min and then rest of ½ salt is added to bath and dyed for 30 min. Then temp is raised to 90° C and Soda ash is added and dyeing for 40-60 min .Then washing with Cold water and washing with Hot water. Then soaping with 2g/L detergent and finally cold wash is performed.

Dyeing Curve:



Sequence of dyeing:





Washing (Cold)

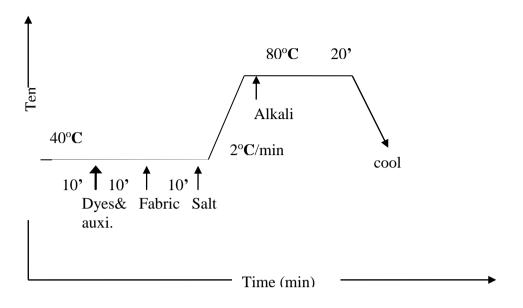
Application of Reactive Dyes:

Dyeing recipe:

Anti creasing agent	>0.3 g/L
Wetting agent	>0.5 g/L
Sequestering agent	>0.5 g/L
Reactive dyes	>0.5% owf
Soda ash	>5 g/L
Caustic	>1 g/L
Salt(Common/glubers)	>80 g/L
M: L Ratio	>1: 8g/L
Temp.	>60-80 ⁰ C
Time	>60-90 min

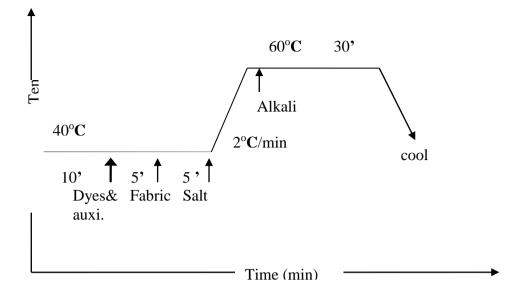
1. Monochloro triazine dyes (MCT):

Example: Procion Red H-3B Or C. I Reactive Red 3



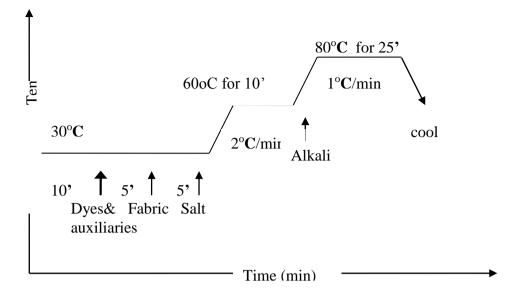
2. <u>Vinyl Sulphone Dyes (VS):</u>

Example: Reactive Black B Or C. I Reactive Black 5

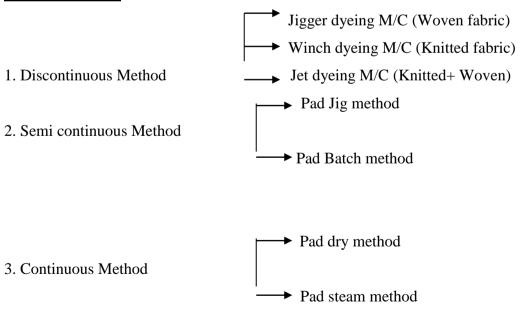


3. Mixed Bifunctional Dyes (MCT+VS):

Example: Reactive Black HFGR



Dyeing Methods:



Pad Batch method:

Recipe:

Dyestuff	>3%
Soda ash	>15g/L
Salt	>60g/L
Urea	>150g/L
Na- alginate	> little amount.

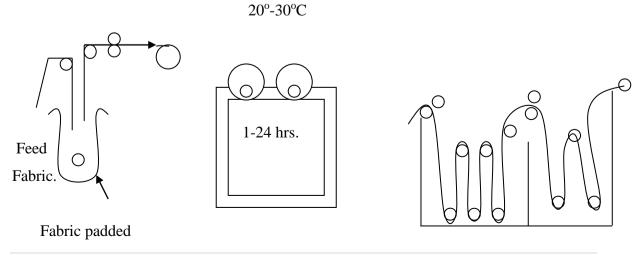
1) Pad Batch (Cold) process:

Procedure:

Fabric is padded in the bath dye, alkali, salt, at Room temp. The temp should be kept constant to avoid variation in shade.

After padding, the batches are wrapped in polyethylene sheet and stored at room temp for 1-24 hrs. During storage period the rolls may be kept slowly rotating to prevent seepage of dye liquor.

When the required batching time has passed, the cloth is washed in a open width washer.



In dye solution

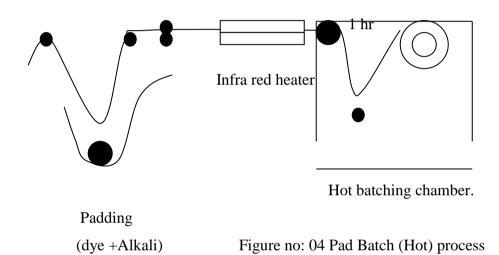
Batch Section.

Wash

Figure no. 03:Pad Batch (Cold) process

2) Pad Batch (Hot) process:

In this method, the milder alkali is used in the pad bath. In the pad batch method, the properly prepared fabric is padded with the dye and alkali, passed bet ⁿ infa red heaters to pre heat the padded fabric to 50 to 95^oC and batched on a large diameter roller in a heat chamber.



The batching is done under controlled conditions of temperature and humidity for a sufficient time to ensure diffusion and fixation of the dye in the fabre. During this period, the batch is kept slowly rotating to avoid seepage of dye liquor. The cloth is then remove for washing.

Continuous Method:

1) Pad steam Method:

In this process, the fabric is padded with the dye sol ⁿ with $60-80^{\circ}$ C and then fabric is passed through drying chamber. Then The fabric is padded with chemical (NaOH + Salt). Then The fabric is passed through a steam chamber at $100^{\circ}-102^{\circ}$ C for 30-60 sec. Then fabric is washed in open widbeth washer.

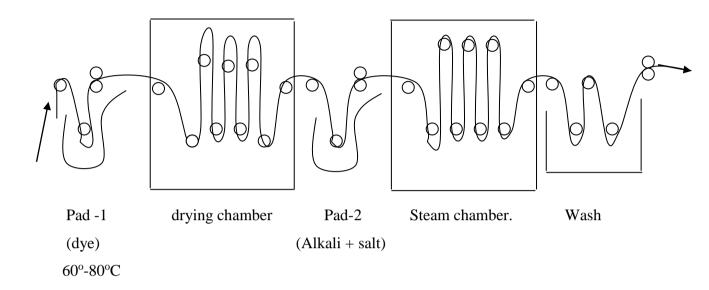


Figure no. 05: <u>Pad steam Method.</u>

2.13 Stripping:

Stripping becomes necessary when uneven dyeing occurs. By stripping azo groups (- N=N-) from the dye is removed.

1) Partial stripping method:

Partial stripping is obtained by treating the dyed fabric with dilute acetic acid or formic acid. The commanded conc. is bet ⁿ 5 to 10 parts glacial acid or 2.5-10 parts of formic acid per 1000 parts of water.

Recipe: Glacial acetic acid $= 5-10$ parts			
Water	>1000 parts		
OR			
Formic acid	>2.5 to 10 parts		
Water	>1000 parts		

Temp	>70-100 ⁰ C
Time	> Until the desired shade is obtained.

Process- The goods are entered and temp is raise to $70-100^{\circ}$ C and the treatment is continued until the shade has been reduced to the desired amount thoroughly. washing is then necessary to remove the products of hydrolysis.

2) <u>Full stripping:</u>

For complete stripping, the goods are firstly treated with $Na_2S_2O_4$ Sodium hydrosulphite at boil. Then at room temperature for 30 min.

2.14 PROBLEMS DYEING WITH REACTIVE DYE:

- 1. Lower exhaustion (For mono function reactive dye) which consequently causes the smaller fixation of dyes to fiber.
- 2. High yeild of colorants in dye effluent. (water organism and algi in water & other micro-organism requires photosynthesis with the help of sunlight and oxygen but colorants degrade the oxygen dissolved in the water as they requires oxidation, which destroy the organisms).
- 3. High concentration of electrolytes in effluent.
- NaCl is unique but it is responsible for machine corrosion.
- Cl & SO₄ increases BOD (Bio chemical oxygen demand) & COD (Chemical oxygen demand), decreasing dissolved oxygen and destruct ecobalance.
- 4. Strong alkalinity in the effluent.(More alkaline more COD required).

5. Hydrolysis of dyes is typically around 30 - 40% depending on the dyes structure.

(If reactive group hydrolyzed first then there is no possibility of reaction with cellulose. But if first reaction with cellulose occurs then possibility of hydrolyzed is less).

- 6. Lengthy series of wash off process engaged to achieving the good or optimum fastness.
- 7. Time consuming due to lengthy wash off process.

(Cationic fixer – unfixed dyes become hydrolyzed and form metal complex, only effective for first wash but for second wash it may break complex structure and unfixed dyes react again.

By using cationic fixer light fastness may decrease).

- 8. Other (especially color fastness to light) fastness is difficult to achieve in optimum level).
- Removal of color is difficult & has to engage very extensive chemical treatment and reactive dyes with Cu – phthalocyanine azo grouphave high risk to form metal complexes with metals in the water.

2.15 LABORATORY IR DYEING MACHINE - IR Dyer



Figure no: 06 Laboratory ir dyeing machine - ir dyer

IR COLOR is a compact laboratory IR type dyeing machine, providing a versatile and most importantly, environmentally clean method of sample dyeing, the dye pots are heated from the top by infrared radiators, the inclination angle of the dye pots to the disc is designed to get a good liquor circulation by the rotation of the disc.

Temperature range:20-160°C Heating capacity: 3/5 kw, Infra-red heating system Cooling.: Air circulation Revolution speed:Vari-speed control, 10-50 rpm, Size of dyepots:150, 300, 500, 1000, 6000 ml Machine dimension for 12 or 24 dye pots 150 ml Chermical Injection device optionnel The model featured here is known as the infrared sample dyeing machine

Some of the very striking features of this model are given here:

- State-of-the-art unique touch screen control.
- Easy availability of single, double or even triple bath versions.
- Data interpretation can be done graphically.
- Fully self-functional dye bath.
- Wide range of beaker option.

2.16 Computer color matching system

First in 1963, ICI dyestuff division (UK), established the CCMS for color match prediction is their lab but it was for their customers only. In 1973, ICS(International Color System)manufactured CMS commercially.

The basic things required to carry out the matching are:

- 1. Color measurement instrument(Spectrometer).
- 2. Reflectance from a mixture of dyes or pigments applied in a specific way.
- 3. Optical model of color vision to judge.

Function of CCMS:

Color match prediction

©Daffodil International University

- > Measurement of color difference
- Mesmerism (physical test in light)
- > Pass or Fail option
- Color fastness rating
- Cost comparison
- > Shade library
- ➢ Strength evaluation
- ➢ Whiteness evaluation
- ➢ Reflectance(R) &K/S measurement
- \succ Etc.

Figure no. 07: Computer Color Matching

System

Color matching:

The process of assuring that a color on one medium remains the same when converted to another medium. This is extremely difficult because different media use different color models. Color monitors, for example, use the RGB model, whereas process printing uses the CMYK model. As color desktop publishing matures, color matching is gaining more and more attention. The most recent Windows and Macintosh operating systems include a color management system (CMS) to assist in color matching.

Color management:

In digital imaging systems, color management is the controlled conversion between the color representations of various devices, such as image scanners, digital cameras, monitors, TV screens, film printers, computer printers, offset presses, and corresponding media.

The primary goal of color management is to obtain a good match across color devices; for example, a video which should appear the same color on a computer LCD monitor, a plasma TV screen, and on a printed frame of video. Color management helps to achieve the same appearance on all of these devices, provided the devices are capable of delivering the needed color intensities.

Color calibration

Calibration is like characterization, except that it can include the adjustment of the device, as opposed to just the measurement of the device. Color management is sometimes sidestepped by calibrating devices to a common standard color space such as <u>RGB</u>; when such calibration is done well enough, no color translations are needed to get all devices to handle colors consistently. This avoidance of the complexity of color management was one of the goals in the development of RGB.

Color translation

Color translation, or color space conversion, is the translation of the representation of a color from one <u>color space</u> to another. This calculation is required whenever data is exchanged inside a color-managed chain. Transforming profiled color information to different output devices is achieved by referencing the profile data into a standard color space. It is easy to convert colors from one device to a selected standard and from that color space to the colors of another device. By ensuring that the reference color space covers the many possible colors that humans can see, this concept allows one to exchange colors between many different color output devices.

The Mathematics of Computer Color Matching

For a computer formulation program to work effectively there must be a mathematical relationship between the concentrations of each dye used to produce a sample and the sample's color. It is not the purpose of this paper to discuss in complete detail the mathematics of color matching, but a brief description will follow. More detailed information may be obtained from a large assortment of textbooks and articles.

The mathematical basis for all color matching software is the Kubelka-Munk series of equations. These equations state that for opaque samples such as textile materials, the ratio of total light absorbed and scattered by a mixture of dyes is equal to the sum of the ratios of light absorbed and scattered by the dyes measured separately. Where absorption is defined as "K" and scattering is defined as "S", Kubelka-Munk states that _:

(K/S) mixture = (K/S) dye 1 + (K/S) dye 2 + (K/S) dye 3 + ...

K/S is not a readily measurable quantity, but it can be calculated from the reflectance of a sample -- "R" -- by the Kubelka-Munk equation that states _:

K/S = (1 - R) / 2R

As an example, if a sample has a reflectance of 20% at a wavelength of 500nm, then the K/S can be calculated as:

K/S = (1 - 0.2) / 2(0.2) = 1.6

If the K/S of a target color is measured at several wavelengths, the concentrations of each dye can be calculated by trial and error from primary dyeings to achieve the closest match. A computer color matching program is capable of performing hundreds of iterations in a short period of time to produce the initial dye concentrations.

Graphical Analysis of Primaries Using K/S:

K/S calculations are invaluable in evaluating the build characteristics of dyestuffs because there is a direct relationship between K/S and dye concentration. In Figure 1, the K/S values of several samples are plotted versus the sample concentrations.

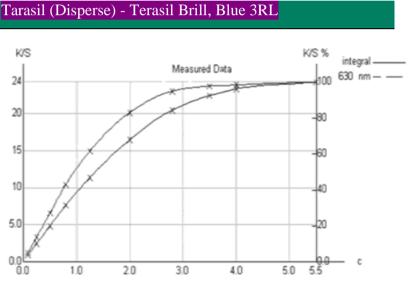


Figure : K/S vs. Dye Concentration

The uniformity of this curve will determine the software's ability to accurately generate new formulas,

Especially in the unknown areas between the known concentrations. Two curves are displayed, one based on K/S for each dye at the wavelength of maximum light absorbance and one based on K/S integrated over the range of 400-700nm. The shapes of these curves may differ due to hue changes as dye concentration increases.

Tarasil (Disperse) - Terasil Orange 2RL

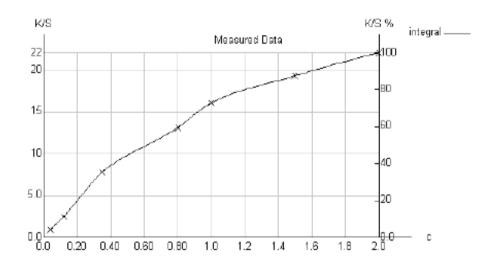


Figure : K/S vs. Dye Concentration - Measured Data

At first glance, the K/S curve for the black dye in Figure 5 appears to be acceptable. If the formulation program was asked to generate a concentration of this dye corresponding to a sample K/S value of 17, it would predict approximately 2.4%. Notice, however, the large gap in the concentration levels between 1% and 4%. If an additional point was dyed at 2.5% and added to the database, the computer's predicted concentration would change to approximately 1.6%.

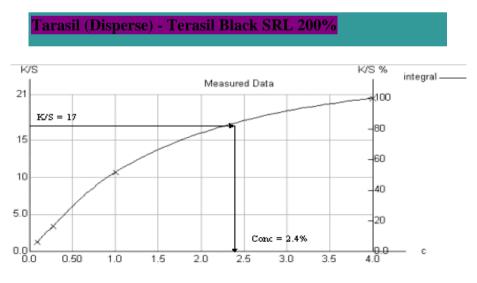


Figure : K/S vs. Dye Concentration for Black SRL.

Graphical Analysis of Primaries Using Reflectance

For most dyes, the percent reflectance's for the darker concentrations are so low relative to that of the light concentrations that it is difficult to determine from the %R graph if there are any problems with the darker dyeing. To make evaluation easier, a plot of K/S versus wavelength is used. In Figure 9, K/S versus wavelength is plotted for the violet dye seen previously in Figure 7.

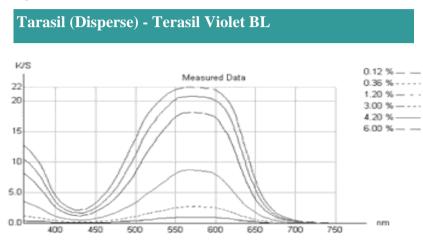


Figure 11: K/S vs. Wavelength

Confirmation of Primary Accuracy

K/S and reflectance graphs are essential tools in determining the quality of the primary data. After any questionable points have been deleted or replaced, the accuracy of the database should be confirmed by generating match predictions for a series of dyed "known's".

A "known" is simply a piece of material that has been dyed using the same techniques and substrate that were used to dye the primaries. When asked to predict a formula for a known sample, the computer formulation software should produce a formula reasonably close to the actual concentrations that were used to produce the sample. Small variations are common and are typically due to dye interaction that cannot be accounted for when the primaries are dyed separately.

Advantage of CCMS:

1. Customer gets the exact shade wanted with the knowledge of Mesmerism.

2. Customer often has a choice of 10-20 formulation that will match color. By, taking

costing availability of dyes and auxiliaries into account, he can choose a best one.

3. 30-300 times faster at achieving match with CCMS than by eye alone.

Most important theme to analyze the spectrophotometer report

 $\Delta L = Lightness$

 $\Delta C = Chroma$ (Unsaturated/saturated)

 $\Delta H = Hue angle (0^{\circ} \sim 360^{\circ})$

<u>Sign</u>	+(Positive)	-(Negative)	
Δa	Redder	Greener	
Δ b	Yellower	Bluer	
HUE	$\Delta \mathbf{H}$		
	+(Positive)	-(Negative)	
Red	BTH Yellower than STD	BTH Bluer than STD	
Yellow	BTH Greener than STD	BTH Redder than STD	
Green	BTH Bluer than STD	BTH Yellower than STD	
Blue	BTH Redder than STD	BTH Greener than STD	
Difference in	Sign	Addition	
Δa	+ve	Green dye	
Δa	-ve	Red dye	
Δb	+ve	Blue dye	
Δb	-ve	Yellow dye	

Table on: 02.Grey Scale for Assessing Change in Shade

EN ISO 105-A03 / IUF 132 / VESLIC C 1211

This Grey Scale is for assessing the degree of change in shade caused to a dyed Textile fabric / yarn in color fastness tests. For example, the change of shade of wool and cotton fabrics in the wash fastness, perspiration fastness, etc.

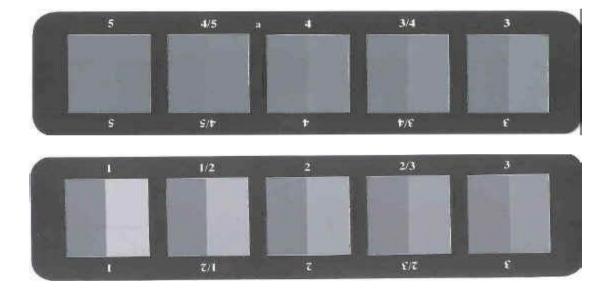
The scale consists of nine pairs of gray color chips each representing a visual difference and contrast.

The fastness rating goes step-wise from:

Note 5 = no visual change (best rating) to Note 1 = a large visual change (worst rating).

The gray scale has the 9 possible values:

5, 4-5, 4, 3-4, 3, 2-3, 2, 1-2, 1.





It is now quite common to measure the Grey Scale change in color instrumentally. This is made using a suitable reflectance spectrophotometer according to the test method procedure,

EN ISO 105-A05.

Grey Scale for Assessing Staining

EN ISO 105-A03 / IUF 132 / VESLIC C 1211

This Grey Scale is for assessing the degree of staining caused by a dyed Textile / yarn in color fastness tests. For example, the staining of wool and cotton fabrics in the wash fastness, perspiration fastness, etc.

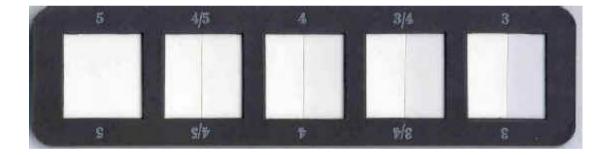
The scale consists of nine pairs of gray color chips each representing a visual difference and contrast.

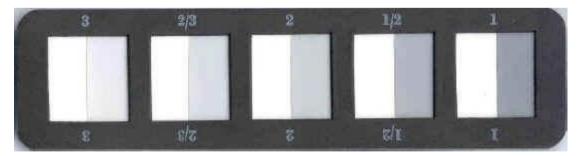
The fastness rating goes step-wise from:

Note 5 = no visual change (best rating) to Note 1 = a large visual change (worst rating).

The grey scale has the 9 possible values:

5, 4-5, 4, 3-4, 3, 2-3, 2, 1-2, 1.





It is now quite common to measure the Grey Scale for assessing staining instrumentally. This is made using a suitable reflectance spectrophotometer according to the test method procedure, EN ISO 105-A04.

3. Experimental techniques & Methodology:

Dyeing method: Exhaust dyeing.

Material	: Poplin fabric.
Apparatus	: Electric balance (to weight dyes &other chemicals) Beaker. Pipette Conical Flask. Lab dip dyeing machine &Etc.
Used dyes	: Reactive dyes.

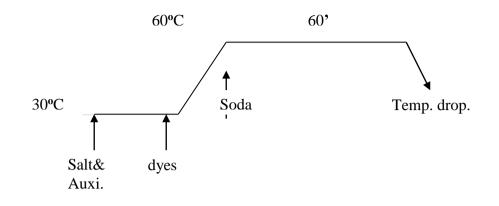
Used chemical &

Auxiliaries	:Glauber salt (Na ₂ SO4.10 H ₂ O),Leveling agent (Drimagen E-2R),
Anticreasing	agent (Imacol C-2F), Wetting detergent. Acetic acid, Soaping detergent.

Dyeing condition:

Time	: 60min.
Temperati	ure: 60°C
M: L	: 1:30

Dyeing curve:



After treatment:

- Hot Wash(at 80°C for 10 min.).
- Neutralization (1g/l acetic acid).
- Soaping(95°C x 10min).
- Cold wash.

3.2 Fastness measurement

3.3 Color fastness to wash: Color fastness to wash is very important for Lab-dip. There are varieties otesting procedure, because -

- Washing conditions may vary from one country to another.
- The methods depend on the use of dyed goods.
- ✤ To evaluate repeated washing accelerated test methods are used.

<u>The degree of fading and staining of dyed goods for washing depends</u> <u>upon the following factors:</u>

- Temperature range may be from 40° C to 95 °C.
- The type and amount of detergent added to the washing bath. In many testing procedures a standard detergent is used.
- The extent of mechanical action which can be varied by changing the agitation speed in a washing machine or by adding steel ball to revolving the bath.
- ✤ The washing liquor to goods ratio is 50:01 . e. The hardness of water.
- The rinsing, drying, or pressing methods used to restore the sample after the washing test.

Principle:

A specimen (Lab-dip) in contact with specified adjacent fabric or fabric is laundered, rinsed and dried. The specimen/composite sample is treated under appropriate condition in a chemical bath for short time. The abrasive action is accomplished by the use of a liquor ratio and an appropriate number of steel balls. The change in color of the specimen (dyed sample) and the staining of the adjacent fabric is assessed by recommended Grey Scales (1-5).

Apparatus and Materials:

- Wash-wheel with a thermostatically controlled water bath and rating speed of (40 ± 2) rpm,
- Stainless steel container (capacity 55±50 ml),
- Stainless steel ball (dia = 0.6 cm, weight = 1 gm),
- SDC, Multifibre fabric (Acetate / Cotton / Nylon /Polyester / Acrylic / Wool),
- ✤ Thermometer,
- ✤ Sewing machine,
- Dryer,
- Color matching cabinet and
- ✤ ISO Scales.

Reagents:

- i. Reference detergent (ECE detergent 77)
- ii. Sodium Carbonate / Soda Ash
- iii. Distilled water (Grade 3) and
- Iv. Etc.

Test specimen:

Cut a sample of dyed goods 10 cm x 4 cm and sew it with same size multifibre. This is the composite test sample.

Test procedure:

(ISO recommendation No. 1-5).

Conditions for washing:

Test	Temperature	Time(Minute)	Steel Ball	Chemicals
ISO-105-	40	30	00	Soap (5g/l)
C01				
ISO-105-	50	45	00	Soap (5g/l)
C02				
ISO-105-	60	30	00	Soap (5g/l)+Soda2g/l
C03				
ISO-105-	95	30	10	Soap (5g/l)+Soda2g/l
C04				
ISO-105-	95	240	10	Soap (5g/l)+Soda2g/l
C05				

Table no:03

Evaluation: Compare the contrast between the treated and untreated sample with Grey Scales for changing color of dyed sample & staining of adjacent fabric in a color matching cabinet.

Numerical rating for color changing is the shade and staining to adjacent fabric. Number of method used.

We have followed ISO-105-CO1 to measure the color fastness to wash where the Composite sample is treated in a wash wheel for 30 minutes at $(40\pm2)^{\circ}$ C with 5 g/1 standard soap.

3.5 Color fastness to Rubbing



Principle:

Figure No. 13 Rubbing tester

This test is designed to determine the degree of color which may be transferred from the surface of a colored fabric to a specific test cloth for rubbing (dry + wet).

Equipment:

- Crock meter
- Cotton rubbing cotton
- ✤ Grey Scale
- Stop watch and
- ✤ Color matching cabinet.

Size of fabric:

14 cm x 5 cm three pieces of sample

Test procedure:

- \clubsuit Lock the test specimen onto the base of the crock meter.
- Using the spinal spring clip, set 5 cm x 5 cm of the white cotton fabric to the finger of the crock meter.
- ✤ Lower the covered finger on the test sample.
- Turn hand crank at the rate of one turn per second ($10 \times 10 \text{ sec}$).
- Remove the white rubbing test cloth and evaluate with grey scale.

Result and discussion

3.1 Dyeing recipe with reactive dyes

Dyeing Recipe:	
Blue	0.25%
Red	3.5%
Yellow	0.25%
Salt	80g/l
Soda	20g/l
M:L	1:30
Time:	30min
Temperature:	60° C

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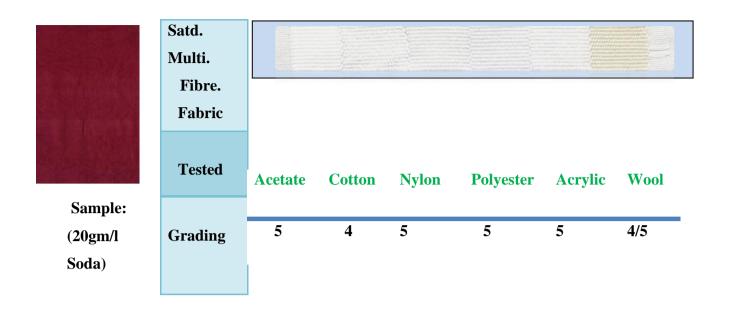
Dyeing Recipe:	
Blue	0.25%
Red	3.5%
Yellow	0.25%
Salt	80g/l
Soda	15g/l
M:L	1:30
Time:	30min
Temperature:	60° C



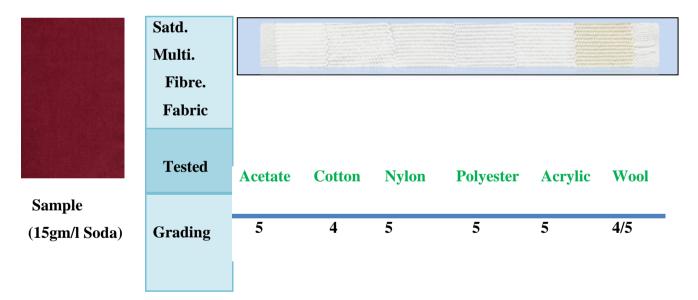
Dyeing Recipe:		
Blue	0.25%	
Red	3.5%	
Yellow	0.25%	
Salt	80g/1	
Soda	10g/l	
M:L	1:30	
Time:	30min	
Temperature:	60° C	



The fastness grading for sample A (20gm/l Soda)

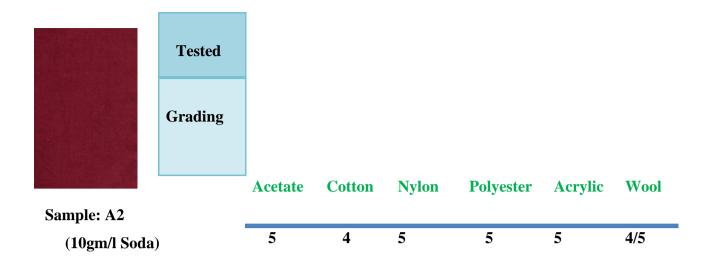


The fastness grading for sample A3 (15gm/l Soda)



The fastness grading for sample A2 (10gm/l Soda)





Comments:

In this test, we see that the color staining is same for all three cases. The color staining properties is very fine. The color fastness properties of soda ash is use are equally. So the deep shade of Red color we can use different concentration of Soda ash.

Colour fastness to Rubbing

Rubbing fastness grading for sample A1 for Soda (20gm/l)

Sample A	Dry rub fastness	Wet rub fastness		
	4/5	3		
			D	
			Dry	Wet

Rubbing fastness grading for sample Soda (10gm/l)

Sample A	Dry rub fastness	Wet rub fastness		Carp and
	4/5	3		0
L			Dry V	Wet

Rubbing fastness grading for sample

Soda (15gm/l)

Sample A	Dry rub fastness	Wet rub fastness	
	4/5	3	
			Dry Wet

Comments:

In this test we see that the color rubbing is same for all three cases. The color rubbing properties is very fine. The color fastness of rubbing properties we use different concentration of Soda.

<u>Measurement of the depth of the shade with data colour</u> (Spectrophotometer):

LAB

10/07/2012 01:52 PM

Colorspace : CIELAB		Observer : 10 Degree	10 Degree Mode : Reflectance		Spectro : COLOR GUIDE	
<u>Standard</u> soda (15gm/l)	<u>Illum</u> D65 TL84 CWF	<u>L</u> * 68.556 63.872 64.610	<u>a*</u> -30.503 -18.717 -20.182	<u>b*</u> -20.804 -33.007 -30.479	<u>C*</u> 36.922 37.945 36.555	H* 214.281 240.420 236.466

COLOR DIFFERENCE REPORT

Comments:

As a standard batch soda (15gm/l) was taken, similarly Batch 1 and Batch 2 was taken for soda (10gm/l) and Soda (20gm/l) respectively. We see in this comparison Batch 1 is lighter than Standard batch. Batch 2 is deeper than Batch 1. From the above observation we can easily conclude that shad of batch 2 (20 gm/l) soda is deeper than other two samples.

5. Conclusion

Cotton fabric is dyeing by the using of reactive dyes. Our project work we used Reactive Dyes, because it's fastness properties is good. This dye is mostly used in Textile Industry. We had done our project work on Reactive dyeing of cotton woven fabric with the change of alkali and showing the variation of shade (color difference) and fastness properties.

At last, we have completed our project work successfully with the help of **Md**. **Mahinur Rahman(General Manager)** Hossain Dyeing & Printing Mills Ltd. HE helped us gratefully.

Finally, we are deeply indebted to our supervisor **Md. Rabiul Islam Khan Lecturer** Department of Textile Engineering, Daffodil International University, whose help, suggestions and encouragement helped us to complete our project work. We would like to thank our respected teacher, Prof. Dr. Mahbubul Haque, Head of the Department of Textile Engineering,, Prof. Dr. S. M. Mahbub-Ul Haque Majumder, Dean, Department of Textile Engineering , for his inspiration, prudent advice, affectionate guidance to make this project work.

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Industry:

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